Accurate Determination of Dielectric Properties in Small, High-Permittivity Dielectric Cylinders

N. Tagdulang*#1, P. Krkotić*, A. Diez*, M. Pont#, J.M. O'Callaghan*2
#ALBA-CELLS, E-08290 Cerdanyola del Vallés, Barcelona, Spain
*CommSensLab, Universitat Politècnica de Catalunya (UPC)
[1]ntagdulang@cells.es [2]joan.ocallaghan@upc.edu

***Abstract*— We describe a method for performing cryogenic permittivity and loss tangent measurements of small (few mm), high-permittivity dielectric cylinders used in surface impedance measurement of superconductors. We combine the use of sapphire and PTFE supports to hold the dielectric under test and provide good thermal conductivity to the cold head. Additionally, the sapphire support is used to facilitate the measurement of the cavity's metal loss and the assessment of its contribution to the overall resonator loss.**

***Keywords*— cavity resonators, dielectric losses, permittivity.**

I. INTRODUCTION

Hakki-Coleman shielded dielectric resonators have been commonly used to measure the surface impedance of small superconducting samples. Nowadays, they are key in the determination of microwave properties in High-Temperature Superconductor-Coated Conductors (HTS-CC), which are being considered as possible substitutes of copper in the beam screen of the Future Circular Collider (FCC) (hadron collider) [1], [2], [3]. HTS-CC are currently available in tapes having several mm in width (typically 12 mm) and over hundredths of meters lengths. The 12 mm size limitation favors the use of TiO_2 (rutile) cylinders as dielectrics in the Hakki-Coleman resonators used to test HTS-CC. Rutile's high permittivity keeps the electromagnetic fields away from the metallic walls of the cavity housing, minimizing their contribution to the resonator loss. However, rutile's dielectric loss is comparable to that of the superconducting samples under measurement. Therefore, accurate determination of HTS-CC surface resistance requires an accurate determination of the loss tangent of the rutile dielectric cylinder that these resonators have. This characterization has to be done for each dielectric cylinder used, since dielectric properties (particularly loss tangent) can change significantly from sample to sample.

In this work we present a measurement procedure to determine permittivity and loss tangent of high-permittivity dielectric cylinders, such as rutile crystals, as a function of temperature (30 to 100 K). The procedure is an adaptation of the techniques described in [4], [5] to the needs of HTS-CC characterization. It considers the need for characterizing small dielectric pieces (compared to those in [4]) and improves the assessment of the residual loss due to the cavity copper walls made in [5].

To test the procedure, we have used a single crystal c-oriented rutile cylinder of 4.12 mm in diameter and 3.02 mm in height.

II. MEASUREMENT SETUP

The dielectric resonator used (Fig. 1) is mounted on the cold head of a Gifford–McMahon closed-cycle cryocooler to perform measurements at variable temperature. A Lakeshore 321 temperature controller is connected to a DT-470 silicon diode temperature sensor and heater in the cold head. A thermocouple is attached at the top of the cavity to monitor thermal gradients. Differences in readings between this thermocouple and the sensor in the cold head where kept below 0.8 K. Semi-rigid cables terminated with 3.5 mm connectors are used between the cryostat feedthroughs and the resonator. An Agilent Twistorr 74FS turbomolecular pump with a Scroll IDP15 primary pump and a Leybold Thermovac TTR 911 N vacuum gauge are used to evacuate the cryostat and to monitor pressure. A Rhode-Schwartz ZNA vector network analyzer is used to measure S-parameters over a span centered about the resonance frequency of the TE_{011} mode using 401 points per sweep. The frequency span is close to ten times the 3 dB bandwidth (see details in [6]). To ensure a temperature stable measurement, the evolution of the frequency and unloaded quality factor is monitored as the resonator is cooled down and stabilized to the minimum temperature. Complete cool-down at a stable temperature is detected when there is no time-dependent trend in successive measurements of resonance frequency and quality factor. Once this is achieved, a slow, temperature-controlled ramp (0.5 K/minute ramp with 1 minute stabilization at every K) is applied to obtain the temperature-dependent dielectric properties.

Fig. 1 shows the shielded dielectric resonator used for dielectric characterization. The resonator is optimized to ensure coupling to the TE_{011} mode and to fulfill the following requirements:

- supports provide mechanical stability of the dielectric samples
- supports provide good thermal conductivity from the dielectric under test to the metal housing
- supports have low dielectric loss and low permittivity
- low loss contribution of copper enclosure
- TE_{011} mode relatively far from other modes

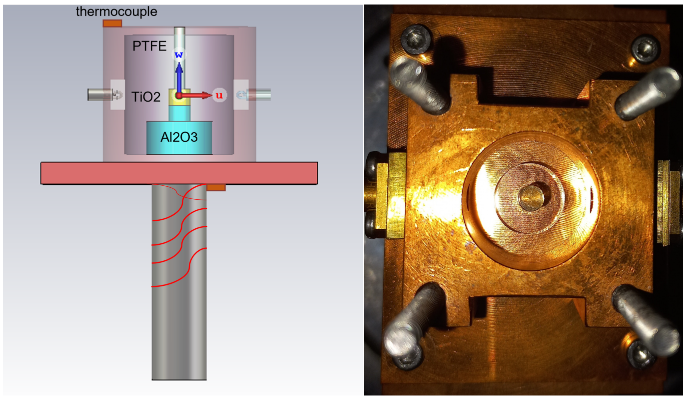

Fig. 1. Schematic of the measurement setup (including resonator) for measuring permittivity and loss tangent at variable temperatures (30-100 K) (left). It includes two temperature sensors to monitor thermal gradients. A photograph of the overhead view of the mounted sample is shown on the right.

Note that the resonator design includes two sapphire supports below the dielectric under test. The big bottom sapphire support has a nominal diameter of 12.00 mm and 6.02 mm nominal height. The small sapphire on top of it has a nominal diameter and height of 4.04 mm and 3.10 mm, respectively. This is unlike the design in [4], which uses quartz as a bottom support. Sapphire has a much lower dielectric loss tangent than quartz, a better thermal conductivity in the temperature range of interest (30 to 100 K) and its permittivity is much lower than that of rutile, so the electromagnetic fields in the resonator will be confined to the surroundings of the rutile cylinder, maximizing the contribution of the dielectric under test to the resonator's overall loss.

Transverse electric quasi- $\mathrm{TE}_{01\delta}$ mode where $0 < \delta < 1$ is excited through coupling loops in this measurement set-up. For simplicity, throughout this article, we shall refer to it as TE_{011}.

The design is optimized using CST [7] to ensure that the loss is predominantly due to the dielectric sample under test. In our design, at 50 K, about 75% of loss is due to the rutile and the rest is mainly due to loss in the metallic cavity walls. The dielectric loss due to the supports is negligible (about 0.02%). Even though the maximum loss contribution is due to the rutile, the contribution of the copper enclosure is not negligible and has to be measured separately.

III. MEASUREMENT PRINCIPLE

A. *Quality Factor and Resonance Frequency*

The resonator's unloaded quality factor and resonance frequency are obtained by processing its measured S-parameters with ARPE, an open-source software [6] available online at [8]. The algorithm used in ARPE discards outlier points in the frequency response due to the effect of nearby modes or distortion caused by the fixture, instrument, or measurement setup. The outlier removal procedure is recursive, with the points with the highest fitting error being removed first, and the resonance frequency and quality factor are re-calculated at each iteration step.

B. *Permittivity vs. Temperature*

To ensure that there are no significant thermal gradients between the dielectric under test and the cavity housing, we have compared permittivity measurements of a rutile cylinder placed into two different resonators: the one in Fig. 1 and a standard Hakki-Coleman resonator [9] using copper metal endplates. In the latter, the thermal conductivity of the metals between the dielectric and the thermocouple is high and the difference between the thermocouple readings and the actual temperature of the dielectric can be assumed to be low. To relate resonance frequency to permittivity, we used the equations given in [10] for the Hakki-Coleman resonator and CST simulations for the resonator in Fig. 1. The maximum relative difference between the two permittivities is 0.4% from 30 to 100 K, indicating that thermal gradients do not introduce significant errors in the measurements.

Fig. 2 shows the measured relative permittivity using the two resonators compared to the measurements in [4]. Error bars in the figure indicate the uncertainty due to dimensional tolerances in the measurement of the height and diameter of the dielectric cylinder. The uncertainties in the figure (0.6%) correspond to 20 μm tolerances.

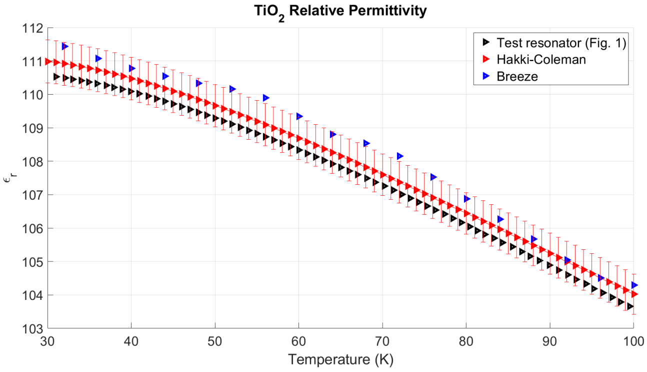

Fig. 2. Dielectric permittivity of a c-oriented rutile cylinder measured using the resonator in Fig. 1 and a Hakki-Coleman resonator with copper endplates, compared to that in [4]. Error bars are the result of 20 μm dimensional uncertainty in dielectric height and diameter.

C. *Copper Surface Resistance*

The contribution of the metal loss to the overall loss in the resonator in Fig. 1 is not negligible, hence accurate measurement of its surface resistance is necessary. Surface resistance is highly dependent on metal impurities, particularly in copper at low temperatures [11], so measuring the surface resistance of the actual copper in the cavity removes the uncertainty related to its composition. This can be done by measuring the empty cavity and scaling the resulting surface resistance to the frequency of the TE_{011} mode [4]. In our case, we can use the large sapphire crystal used as support in Fig. 1 to lower the resonance frequency and facilitate the measurement of the metal loss. By using the sapphire crystal with a PTFE cylinder to pressure it down and hold it (as shown in Fig. 3), we lower the resonance frequency from 19.49 GHz (empty cavity) to 9.72 GHz (TE_{011} mode in Fig. 3), making measurements simpler.

Surface resistance is related to the unloaded quality factor (Q_0) and dielectric loss ($\tan\delta$) of the resonator in Fig. 3 through the following expression:

$$\frac{1}{Q_0} = \frac{R_s}{G_s} + p_s \tan\delta_s + p_t \tan\delta_t \tag{1}$$

where R_s, G_s are the cavity wall surface resistance and geometric factor, p_s, $\tan\delta_s$, p_t, and $\tan\delta_t$ are the filling factors and dielectric loss tangents of the sapphire and PTFE supports, respectively. Their contribution to the sum in the right hand side of the equation above is negligible. Neglecting these terms, the perturbation analysis of the equation 1 indicates that the relative uncertainty in R_s is equal to that in Q_0:

$$\frac{\Delta R_s}{R_s} \approx \frac{\Delta Q_0}{Q_0} \tag{2}$$

which, in this cavity, is about 5%, as estimated by repeated measurements after assembling and disassembling the cavity.

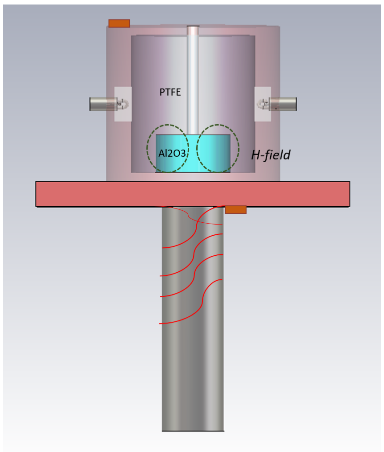

Fig. 3. Set-up for Cu surface resistance measurement at 9.72 GHz. As in Fig. 1, the TE_{011} mode is used.

D. Loss Tangent vs. Temperature

For the fully assembled dielectric resonator including the dielectric under test (Fig. 1) we can expand (1) to:

$$\frac{1}{Q_0} = \frac{R_s}{G_s} + p_d \tan\delta_d + p_s \tan\delta_s + p_t \tan\delta_t \tag{3}$$

where the terms p_d, $\tan\delta_d$ are the filling factor and loss tangent of the dielectric under test.

A perturbation analysis on the equation above reveals that the uncertainty due to the sapphire and PTFE supports is negligible. The analysis assumes a maximum uncertainty in loss tangent of 10^{-5} for the dielectric sample and supports [4], and 10^{-3} for the filling factors. The relative uncertainties in filling factors have been estimated through CST simulations. Under these conditions, and taking into account that $p_d \approx 1$:

$$\Delta \tan\delta_d \approx \sqrt{(\frac{\Delta Q_0}{Q_0^2})^2 + (\frac{\Delta R_s}{G_s})^2}. \tag{4}$$

Therefore, the uncertainty in loss tangent is due to the uncertainty in unloaded quality factor and surface resistance. To estimate $\Delta\tan\delta_d$ at 50 K, we use (4) with $Q_0 \approx 169000$ and an absolute uncertainty in Q_0 of 500 (which, as previously, is assessed by repeatedly re-assembling and re-measuring the cavity). The uncertainty in R_s is 5×10^{-4} Ω and the surface geometrical factor G_s is 16700 Ω. This makes $\Delta\tan\delta_d \approx 3 \times 10^{-8}$ at 50 K and 6.57 GHz.

IV. RESULTS AND DISCUSSION

A. Resistivity of the Cu Enclosure

The resistivity of Cu depends strongly on its impurities, and it is quanitified by its residual resistivity ratio (RRR) [11]. Fig. 4 shows the resistivity calculated from the Cu surface resistance measurements at 9.72 GHz, which corresponds to the RRR of ordinary copper. In performing the calculation, we assumed that the copper is in the anomalous skin effect regime [12]. We have estimated a 10.7% uncertainty in resistivity at 50 K due to the uncertainty in R_s described in Sect. III-C and the small uncertainity in resonance frequency ($\Delta f = 460$ kHz) estimated through repeated resonator measurements performed after assembling and disassembling the cavity.

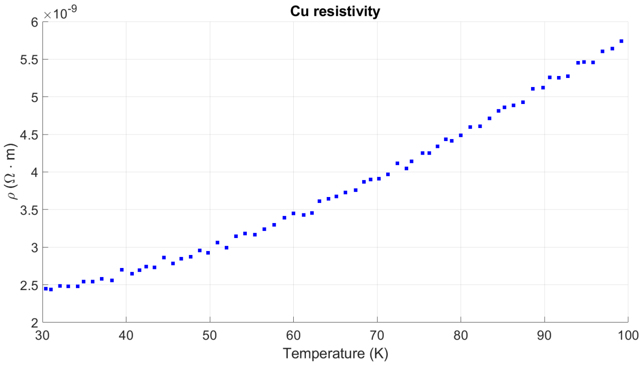

Fig. 4. Cu resistivity calculated from R_s measurements at 9.72 GHz.

B. Loss Tangent of TiO_2

We use (3) to determine loss tangent ($\tan\delta_d$) from Q_0 and R_s. The value of R_s at the resonance frequency of the resonator in Fig. 1 (6.57 GHz) is calculated using equations for the anomalous skin effect [12] from the resistivity data in Fig. 4. The contribution of the PTFE and sapphire supports is computed from their nominal loss tangent specifications ($\tan\delta_d$, $\tan\delta_t$), although it is negligible compared to other terms in (3). The resulting $\tan\delta_d$ is shown in Fig. 5.

Our results can be compared to those in [5] and [4] by assuming that $\tan\delta_d$ is proportional to frequency [4]. With that assumption, at 50 K, our value for $\tan\delta_d$ is about twice that in [4] which is, in turn, about twice that in [5]. The varying concentration of metal impurities in the rutile crystals mentioned in [5] may explain these differences.

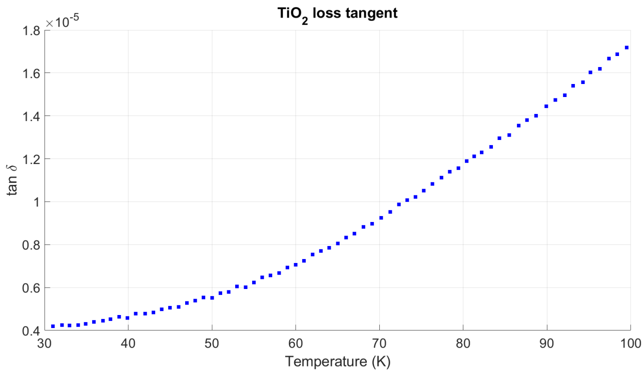

Fig. 5. Loss tangent in c-oriented single crystal rutile cylinder with azimuthal electric field at 6.57 GHz

V. CONCLUSION

We have shown a measurement setup to perform cryogenic characterization of the dielectric properties of small dielectric cylinders. The thermal conductivity of the sapphire supports minimizes thermal gradients, so the strong temperature dependence of the dielectric properties of rutile is compatible with our system based on conduction cooling. We show that in high-permittivity, small (few mm) dielectric cylinders, dimensional tolerances are critical. For rutile, 20 μm tolerances are needed to have 0.6% uncertainty in permittivity. Our permittivity results are consistent with those in previous publications. With proper frequency scaling, our loss tangent results are within the same order of magnitude than those in [4], [5], but they are about twice those in [4] and about four times those in [5]. Varying concentrations of nickel and other metal impurities [5] among the rutile samples used may be the reason for the disagreement. The high variability in loss tangent makes this an essential technique to be included in measurement procedures for accurate characterization of surface resistance in superconductive materials using rutile-loaded Hakki-Coleman resonators.

ACKNOWLEDGMENT

Authors wish to acknowledge the support provided by CERN under Grants FCC-GOV-CC-0072/KE3358 and FCC-GOV-CC-0210/KE4945. NT acknowledges MSCA-COFUND-2016-754397 for the PhD grant.

REFERENCES

[1] T. Puig, P. Krkotić, A. Romanov, J. O'Callaghan, D. A. Zanin, H. Neupert, P. C. Pinto, P. Demolon, A. G. Costa, M. Taborelli, F. Perez, M. Pont, J. Gutierrez, and S. Calatroni, "Coated conductor technology for the beamscreen chamber of future high energy circular colliders," *Supercond. Sci. Technol.*, vol. 32, p. 094006, July 2019.

[2] A. Romanov, P. Krkotić, G. Telles, J. O'Callaghan, M. Pont, F. Perez, X. Granados, S. Calatroni, T. Puig, and J. Gutierrez, "High frequency response of thick REBCO coated conductors in the framework of the FCC study," *Sci. Rep.*, vol. 10, p. 12325, July 2020.

[3] P. Krkotić, A. Romanov, N. Tagdulang, G. Telles, T. Puig, J. Gutierrez, X. Granados, S. Calatroni, F. Perez, M. Pont, and J. O'Callaghan, "Evaluation of the nonlinear surface resistance of REBCO coated conductors for their use in the FCC-hh beam screen," *Supercond. Sci. Technol.*, vol. 35, p. 025015, Jan. 2022.

[4] J. Breeze, *Temperature and Frequency Dependence of Complex Permittivity in Metal Oxide Dielectrics: Theory, Modelling and Measurement*. Springer, 2016.

[5] N. Klein, C. Zuccaro, U. Dähne, H. Schulz, N. Tellmann, R. Kutzner, A. G. Zaitsev, and R. Wördenweber, "Dielectric properties of rutile and its use in high temperature superconducting resonators," *J. Appl. Phys.*, vol. 78, p. 6683, Aug. 1995.

[6] P. Krkotić, Q. Gallardo, N. Tagdulang, M. Pont, and J. O'Callaghan, "Algorithm for resonator parameter extraction from symmetrical and asymmetrical transmission responses," *IEEE Trans. Microw. Theory Tech.*, vol. 69, pp. 3917–3926, June 2021.

[7] D. Systems. (2018) CST - Computer Simulation Technology. [Online]. Available: https://www.cst.com/

[8] (2021). [Online]. Available: www.arpe.upc.edu

[9] P. Krkotić, A. Aguasca, and J. O'Callaghan, "Small footprint evaluation of metal coatings for additive manufacturing," in *48th European Conf. (EuMC)*, 2018, pp. 882–885.

[10] J. Mazierska and R. Grabovickic, "Circulating power, rf magnetic field, and rf current density of shielded dielectric resonators for power handling analysis of high-temperature superconducting thin films of arbitrary thickness," *IEEE Trans. Microw. Appl. Supercond.*, vol. 8, pp. 178–187, Dec. 1998.

[11] R. Matucala, "Electrical resistivity of copper, gold, palladium, and silver," *J. Phys. Chem. Ref. Data*, vol. 8, pp. 1147–1298, 1979.

[12] R. G. Chambers, "Anomalous skin effect in metals," *Nature*, vol. 165, pp. 239–240, Feb. 1950.