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Microwave characterisation of CaF_2 at cryogenic temperatures using a dielectric resonator technique

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Abstract

Properties of calcium fluoride (CaF_2) have been well researched at UV, visible and IR range of frequencies but not at microwave frequencies. In this work we report the loss tangent and the real part of relative permittivity ε_r of CaF_2 measured in the temperature range 15–81 K and at frequency 29.25 GHz. The $\tan\delta$ and ε_r were determined by measurements of the resonant frequency and the Q_0-factor of a TE_{011} mode cylindrical copper cavity with superconducting plates containing the sample under test. The measured ε_r of CaF_2 was found to change from 6.484 to 6.505, and the $\tan\delta$ from 3.1×10^{-6} to 22.7×10^{-6} when temperature was varied from 15 to 81 K. Due to the low losses CaF_2 can be useful in construction of high Q-factor microwave circuits and devices operating at cryogenic temperatures.

Keywords: CaF_2; Microwave ceramics; Resonators; Dielectric properties

1. Introduction

Calcium fluoride crystals are used in many optical applications, including mirror substrates for UV laser systems, windows, lenses and prisms for ultraviolet, visible and infrared frequencies. CaF_2 is grown by the Stockbarger technique or the Brigdman method in diameter up to about 200 mm. Calcium Fluoride (VUV grade) crystals have the transmission range from 0.19 to 7.2 μm and low refractive index from about 1.35 to 1.51 through this range.[1] IR grade Calcium Fluoride is transparent up to 12 μm. Degradation due to moisture in the atmosphere is minimal, and polished surfaces may be expected to withstand several years exposure to normal atmospheric conditions. Due to its low refractive index, Calcium Fluoride can be used without an anti-reflective coating. The maximum temperature CaF_2 can tolerate is 800 °C in dry atmosphere. Low solubility and wide transmission makes this material useful for many applications, including mirror substrates for UV laser systems, windows, lenses and prisms for UV and IR applications.[1]

Due to the low relative permittivity and low losses, calcium fluoride can find applications in microwave planar circuits as a substrate material. Another possibility could be a hybrid high temperature superconductor (HTS)—silicon technology for microwave circuits as investigated in Refs. 2 and 3. As silicon atoms diffuse into HTS films during annealing at elevated temperatures resulting in deteriorating superconducting properties, CaF_2 was investigated for its usefulness to overcome this difficulty due to its chemical stability and structural and thermal compatibility with Si and GaAs.

Contrary to extensive data available for CaF_2 at optical frequencies, there is little data on microwave properties of this material, especially at cryogenic temperatures. In this paper we present results of precise measurements of the permittivity and loss tangent of CaF_2 at cryogenic temperatures from 15 to 81 K using the dielectric resonator technique. We have used the multifrequency Transmission Mode Q-Factor (TMQF) technique[4,5] for data processing to ensure high accuracy of calculated values of ε_r and $\tan\delta$. Also the thermal expansion phenomenon of the material was taken into account in the calculations.

2. Dielectric resonator measurement method

The superconducting dielectric resonator technique is a modification of the metallic dielectric resonator[6,7] and has recently been used to characterise various low loss single crystal and polycrystalline dielectric materials at microwave frequencies.[8–10] The Hakki-Coleman version of the dielectric resonator we used for the measurements

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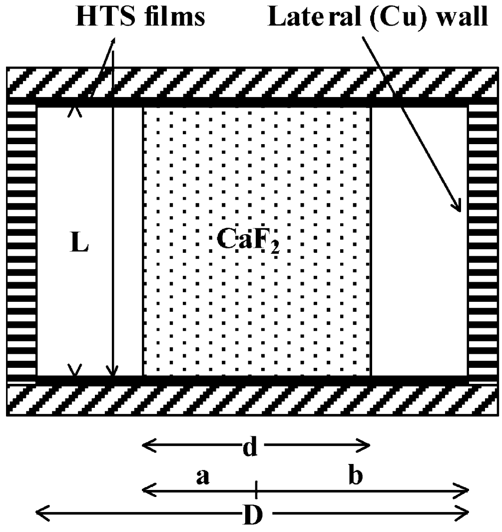

Fig. 1. Schematic of a TE$_{011}$ mode dielectric resonator.

of CaF$_2$ is shown in Fig. 1. The resonator consisted of the copper cavity of diameter of 9.5 and 3 mm height with superconducting endplates. The CaF$_2$ sample was machined into a cylinder with the aspect ratio (diameter to height) equal to 1.67, with 3.1 mm height and 5.0 mm diameter.

The real part of relative permittivity ε_r were determined from measurements of the resonant frequency of the resonator with the CaF$_2$ sample as the first root of the following transcendental equation[11] using software SUP12:[12]

$$k_{\rho 1}J_0(k_{\rho 1}b)F_1(b)+k_{\rho 2}J_1(k_{\rho 1}b)F_0(b)=0 \tag{1}$$

where:

$$F_0(\rho)=I_0(k_{\rho 2}\rho)+K_0(k_{\rho 2}\rho)\frac{I_1(k_{\rho 2}a)}{K_1(k_{\rho 2}a)}$$

$$F_1(\rho)=-I_1(k_{\rho 2}\rho)+K_1(k_{\rho 2}\rho)\frac{I_1(k_{\rho 2}a)}{K_1(k_{\rho 2}a)}$$

$$k_{\rho 1}^2=\frac{\omega^2\varepsilon_r}{c^2}-k_z^2,\quad k_{\rho 2}^2=k_z^2-\frac{\omega^2}{c^2},\quad k_z=\pi/L$$

and ω is the angular frequency (2πf), c is velocity of light, ε_o is free space permeability, ε_r is real relative permittivity of the sample and J_0, J_1, I_0, I_1, K_0, K_1, denote corresponding Bessel and Hankel functions.

The loss tangent tanδ of CaF$_2$ was computed from the measured Q_0-factor of the resonator on the basis of the well known loss equation,[11] namely:

$$\tan\delta=\frac{1}{p_e}\left[\frac{1}{Q_0}-\frac{R_{SS}}{A_S}-\frac{R_{SM}}{A_M}\right] \tag{2}$$

where Q_0 is the unloaded Q-factor of the entire resonant structure, R_{SS} and R_{SM} are the surface resistance of the superconducting and the metallic parts of the cavity respectively, A_S and A_M are the geometric factors of the superconducting part and metallic parts of the cavity and ρ_e is the electric energy filling factor.

Geometric factors A_S, A_M, and ρ_e to be used in (2) were computed using incremental frequency rules as follows:[11]

$$A_S=\frac{\omega^2\mu_0}{4}/\frac{\partial\omega}{\partial L} \tag{3}$$

$$A_M=\frac{\omega^2\mu_0}{2}/\frac{\partial\omega}{\partial a} \tag{4}$$

$$p_e=2\left|\frac{\partial\omega}{\partial\varepsilon_r}\right|\frac{\varepsilon_r}{\omega} \tag{5}$$

Computed values of the geometrical factors and the energy filling factors are given in Table 1.

Values of the surface resistance HTS endplates (R_{SS}) and copper walls (R_{SM}), necessary for Eq. (2) were measured in the same copper cavity but with the sapphire rod and results are given in Section 3.

3. Measurements of microwave properties of CaF$_2$

The measurement system we used for microwave characterisation of the calcium fluoride sample is shown in Ref. 10. The system consisted of Network Analyser (HP 8722C), closed cycle refrigerator (APD DE-204), temperature controller (LTC-10), vacuum Dewar, a PC and the Hakki-Coleman dielectric resonator in transmission mode. The CaF$_2$ sample was grown with the Brigdman method by Ref. 13.

3.1. *Measurements of* R_{SS} *and* R_{SM} *of the Hakki-Coleman cavity*

As mentioned in Section 2 the surface resistances R_{SM} and R_{SS} of the cavity needed to be measured first. To obtain precise values of R_{SM} we have measured S-parameters (S_{21}, S_{11}, and S_{22}) around the resonance of the Hakki-Coleman resonator with the sapphire rod and copper cavity. The measured data sets were processed

Table 1
The geometrical factors and the energy filling factor of the Sapphire and CaF$_2$ resonators

Dielectric rod	CaF$_2$	Sapphire
Frequency	29.3 GHz	24.65 GHz
A_M	22,029	22,319
A_S	329.8	280.6
p_e	0.96	0.97

with the Transmission Mode Q-Factor Technique[3,4] to obtain the loaded Q_L-factor and coupling coefficients as mentioned in Section 1. The TMQF method accounts for noise, delay due to uncalibrated transmission lines and its frequency dependence, and crosstalk in measurement data and hence provides accurate values of Q_L and the coupling coefficients β_1 and β_2. The unloaded Q_0-factor was subsequently calculated using the exact equation,[14]

$$Q_0 = Q_L(1 + \beta_1 + \beta_2) \tag{6}$$

Assuming loss tangent of the sapphire rod as 10^{-7}, the surface resistance of copper R_{SM} was calculated with Ref. 11 based on:

$$R_{SM} = A_M\left[\frac{1}{Q_0} - p_e \tan\delta\right] \tag{7}$$

where A_M is the metallic geometric factor for the copper cavity.

The surface resistance R_{SS} was measured with the sapphire rod in the copper cavity with end walls comprising of a pair of high quality $YBa_2Cu_3O_7$ thin films and calculated with Ref. 12 based on:[11]

$$R_{SS} = A_S\left[\frac{1}{Q_0} - \frac{R_{SM}}{A_M} - p_e \tan\delta\right] \tag{8}$$

Measured dependence of surface resistances, R_{SS} and R_{SM}, with temperature at frequency of 24.6 GHz are presented in Fig. 2.

As measurements of calcium fluoride were performed at a frequency of approximately 29.25 GHz, not at 24.6 GHz, the measured values of R_{SM} and R_{SS} were scaled assuming the square root frequency dependence for R_{SM} of copper and the frequency square law for R_{SS} of superconducting endplates.

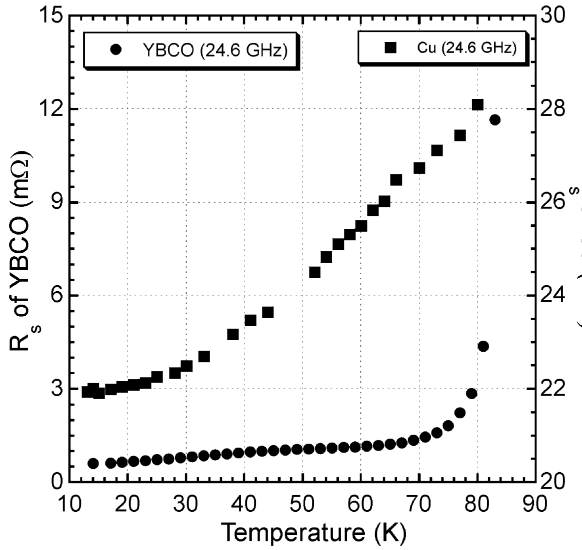

Fig. 2. Measured surface resistances of $YBa_2Cu_3O_{7-\delta}$ films and copper versus temperature.

3.2. *Measurements of microwave properties of calcium fluoride*

The Hakki-Coleman resonator with HTS endplates containing the CaF_2 sample was cooled from room temperature to approximately 12 K, and the resonant frequency of 29.25 GHz was obtained. The S_{21}, S_{11} and S_{22} parameters data sets around the resonance were measured as a function of increasing temperature from 13 to 81 K, and the Q_0-factor and f_{res} were calculated using the TMQF technique and Eq. (6) as before. The real relative permittivity ε_r of the calcium fluoride sample was calculated from the measured resonant frequency using Eq. (1). Variation of dimensions of the CaF_2 sample with temperature as shown in Fig. 3 were taken into consideration in the computations of ε_r using the temperature dependence of the linear thermal expansion coefficient after Ref. 15 as given in Fig. 4.

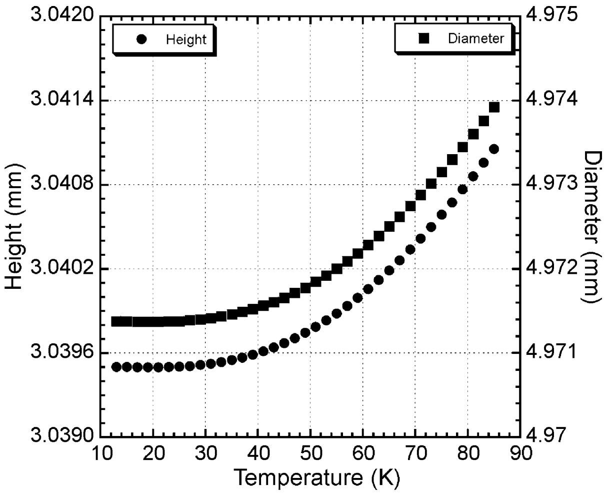

Fig. 3. Dimensions of the CaF_2 sample at cryogenic temperatures.

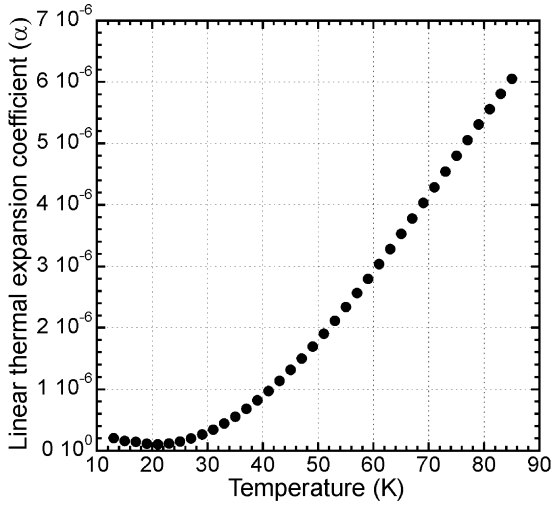

Fig. 4. Temperature coefficient of CaF_2 based on Ref. 15.

The real part of relative permittivity ε_r of the CaF_2 sample measured at temperatures from 15 to 81 K is shown in Fig. 5. The ε_r exhibited the magnitude of approximately 6.5 and increased with the temperature by approximately 0.33%; from 6.483 to 6.505.

The loss tangent of the CaF_2 sample was calculated using (2) from the measured unloaded Q_0-factor. The measured temperature dependence of $\tan\delta$ is shown in Fig. 6. The loss tangent showed an increase of 86% in the temperature range from 15 to 81 K; at temperatures of 15 and 81 K, the measured $\tan\delta$ of CaF_2 was 3.1×10^{-6} and 2.27×10^{-5}, respectively. Using a linear scaling, the calculated loss tangent of CaF_2 at temperature of 15 K and frequency of 10 GHz is only 1.05×10^{-6}. Our results show that CaF_2 exhibits very low losses at microwave frequencies and cryogenic temperatures, comparable to losses in Teflon.[9]

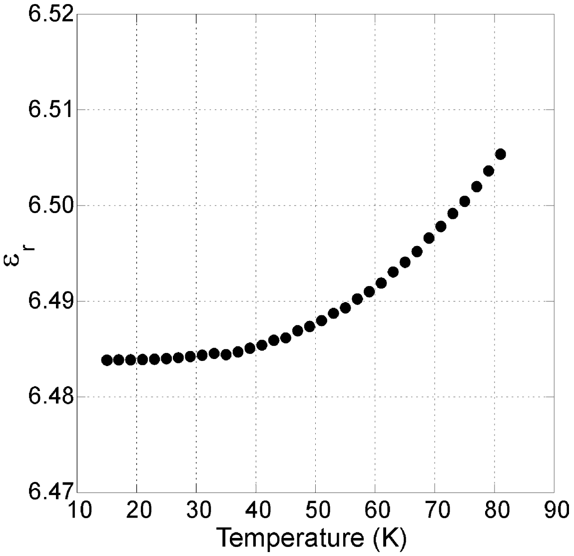

Fig. 5. Measured real part of permittivity of CaF_2 as a function of temperature at 29.25 GHz.

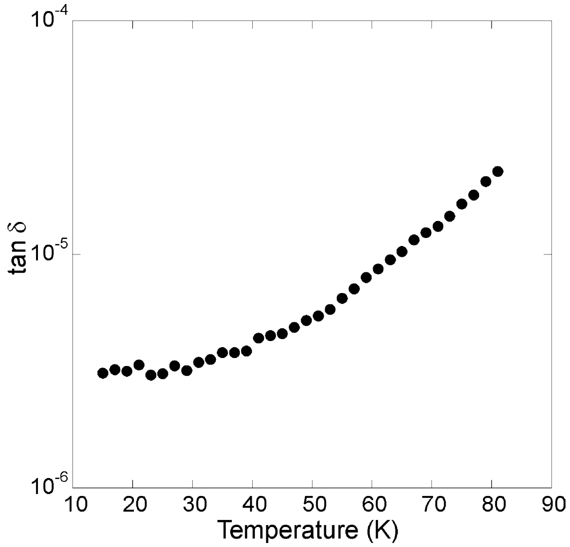

Fig. 6. Measured loss tangents of CaF_2 as a function of temperature at 29.25 GHz.

4. Error analysis of measured parameters ε_r and $\tan\delta$ of CaF_2

The accuracy of the measurement of the real part of permittivity using the dielectric resonator depends on the precision of the measurements of the resonant frequency and uncertainty in dimensions of the dielectric sample. We measured f_{res} with a resolution of 1 Hz using the Network Analyser HP 8722C. Hence to assess uncertainty in ε_r measurements the error analysis was performed assuming the uncertainty in the dimensions of the sample of 0.2 and 0.5% using the software SUP12.[11] Results of the error analysis are presented in Fig. 7 and show that the relative error $\Delta_r\varepsilon_r$ is approximately twice the uncertainty in dimensions.

The uncertainty in the loss tangent measurements is caused by the uncertainty in the measured unloaded Q_0-factor values, R_{SS}, R_{SM} and geometrical factors. The Most Probable Error (MPE) in $\tan\delta$ of CaF_2 can be expressed after Ref. 10 as:

$$\nabla_r \tan\delta = \left[\left|\frac{\Delta\rho_e}{\rho_e}\right|^2 + \left|\left(\frac{-1}{Q_0\rho_e\tan\delta}\right)\frac{\Delta Q_0}{Q_0}\right|^2 + \left(\frac{R_{SS}}{A_S\rho_e\tan\delta}\left(\left|\frac{\Delta R_{SS}}{R_{SS}}\right| + \left|\frac{\Delta A_S}{A_S}\right|\right)\right)^2 + \left(\frac{R_{SM}}{A_M\rho_e\tan\delta}\left(\left|\frac{\Delta R_{SM}}{R_{SM}}\right| + \left|\frac{\Delta A_M}{A_M}\right|\right)\right)^2 \right]^{1/2} \tag{9}$$

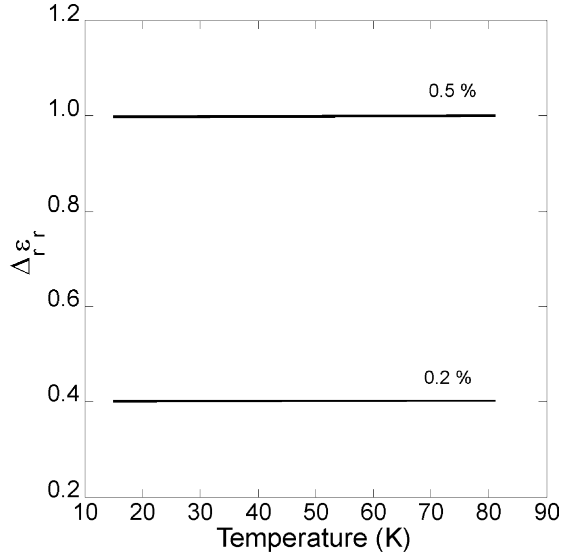

Fig. 7. Most probable error in ε_r versus temperature for 0.2 and 0.5% uncertainty in samples' dimensions.

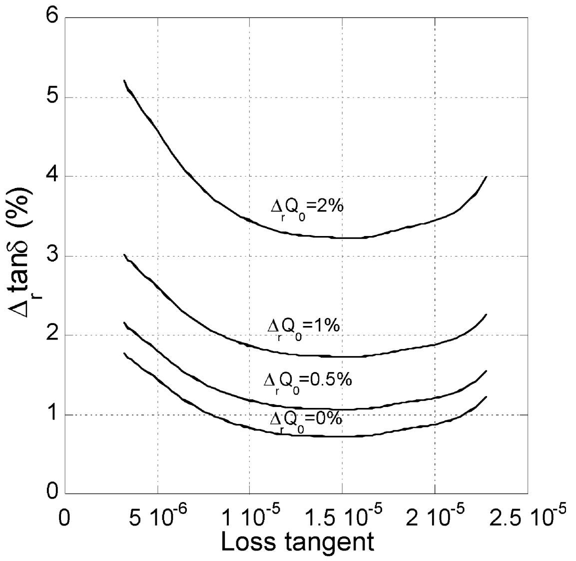

Fig. 8. Most probable error in tanδ of CaF_2 versus temperature for varying uncertainty in Q_o.

Table 2
Dimensions and permittivity and loss tangent of CaF_2 at 15 and 81 k

	Temp. (K)	CaF_2
Height (mm)	15	3.039 ± 0.006
	81	3.040 ± 0.006
Diameter (mm)	15	4.971 ± 0.009
	81	4.973 ± 0.009
Permittivity	15	6.483 ± 0.026
	81	6.505 ± 0.026
Tanδ ($\times 10^{-6}$)	15	3.1 ± 0.09
	81	22.7 ± 0.68
Frequency (GHz)		29.25

To assess errors in our measurements of tanδ we assumed uncertainties in R_{SS} and R_{SM} of 2%, and 0.5% for uncertainties in A_S, A_M and ρ_e. Calculated errors in measured loss tangent values of CeF_2 for assumed uncertainties in the Q_0-factor measurements of 0, 0.5, 1 and 2% are presented in Fig. 8. The calculated MPE in tanδ for perfect Q_0-factor measurements is approximately 1%. We assess the uncertainty in the Q_0-factor of our measurement system as 1%. Hence the most probable error in tanδ of CaF_2 (which varied from 3.1×10^{-6} to 22.7×10^{-6}) is between 1.7 and 3%.

5. Conclusions

The real relative permittivity and loss tangent of CaF_2 have been measured at frequency of 29.25 GHz at cryogenic temperatures using the Hakki-Coleman dielectric resonator with superconducting endplates. The recently developed Transmission Mode Q-Factor technique was used for data processing to remove noise, crosstalk and delay due to un-calibrated cable and connectors and to ensure high precision of measurements. Calcium fluoride was found to exhibit ε_r varying from 6.483 to 6.505, and tanδ from 3.1×10^{-6} to 22.7×10^{-6} in the temperature range from 15 to 81 K as given in Table 2. On the basis of performed error analysis we assessed the uncertainty in the measurements of ε_r and tanδ to be below 0.4 and 3%, respectively in the temperature range from 13 to 81 K. Our measurements have shown that CaF_2 is a very low loss material at cryogenic temperatures at frequency of 29 GHz. Hence, apart from optical applications, calcium fluoride can be useful in cryogenic microwave circuits where very low tanδ and resistance to atmospheric conditions are needed and the real relative permittivity of approximately 6.5 is adequate.

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