Low temperature microwave characterisation of lithium fluoride at different frequencies

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Low temperature microwave characterisation of lithium fluoride at different frequencies

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Abstract

Precise knowledge of dielectric properties of materials is required to implement the material in high frequency devices and circuits. At microwave frequencies complex permittivity (dielectric constant and loss tangent) are the two mandatory parameters prior to any design. We have identified Lithium Fluoride as a potential candidate, which can be used in conjunction with superconducting and non-superconducting parts of several microwave communication devices. Even though dielectric constant of LiF is known at room temperature there only limited data presented at cryogenic temperatures. We have used a dielectric post resonator for the microwave characterisation of the rod shaped LiF crystal. In this paper, we have reported the dielectric constant (perpendicular component of the real part of complex permittivity) and loss tangent of two LiF crystals as a function of temperature (15–290 K) at frequencies of 8 and 16.5 GHz. We have also studied and reported the temperature coefficient of frequency and permittivity. The concept of using temperature coefficient of frequency as a standard is proved to be wrong in this paper. Microwave properties of other Fluorides are also compared with the LiF crystal.

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Keywords: Dielectric properties; Permittivity; Loss tangent; Lithium fluoride

1. Introduction

Properties of Lithium Fluoride (LiF) have been studied at optical frequencies [1–3]. LiF has the lowest index of refraction of all the common infrared materials [1]. Lithium fluoride is the material with the most extreme UV transmission of all and is used for special UV optics [2]. It transmits well into the UUV region at the hydrogen Lyman-alpha line (121 nm) and beyond. Lithium Fluoride is used for optical windows, prisms, and lenses in the UV to infrared where desired transmission is in the range 0.104–7 μm [3]. It is also used for X-ray monochromator plates where its lattice spacing makes it the most useful analysis crystal [2]. LiF is slightly plastic and has a relatively high thermal expansion coefficient. LiF is sensitive to thermal shock. The material can be cleaved. LiF is attacked by atmospheric moisture at 400 °C and softens at 600 °C.

Modest precautions should be taken against moisture and high-energy radiation damage.

Some of the physical properties of LiF are given in Table 1 [1–4]. Table 2 shows the comparison of dielectric constant of commonly used Fluorides. LiF is characterised at cryogenic temperatures by three different groups [4–6]. One of the advantageous of LiF crystal over fluorides is its higher permittivity. Also the permittivity of LiF is close to the permittivity values of Sapphire and Magnesium Oxide, which are the popular substrates used for the fabrication of superconducting thin films.

The moderately high dielectric constant of LiF could make LiF as a possible candidate in many microwave applications. It is essential for the design Engineers to know the microwave characteristics of LiF to design and fabricate devices and circuits using LiF. In order to use LiF in conjunction with superconducting or low temperature operating circuits it is vital to comprehend the dielectric behaviour of LiF as a function of temperature at microwave frequency. In this paper we have reported the complex permittivity of LiF at frequencies of 8 and 16.5 GHz and temperatures from 15 to 290 K. The variation of \( Q_0 \times f_0 \), temperature coefficient of permittivity and frequency with temperatures are also discussed.
2. Experimental techniques

The dielectric resonator technique is typically used for the microwave characterisation of dielectric materials of uniform shapes. Among the different types of dielectric resonators, Hakki–Coleman (HC) dielectric resonator is commonly used to characterise rod (or cylindrical) shaped dielectric materials at microwave frequencies [7–13]. The accuracy of measurements using HC dielectric resonator is high but the main difficulty is to machine samples precisely to realize the same height as that of the copper cavity. Also the linear thermal expansion coefficient of the metallic wall is different from that of the dielectric material. This constitutes a problem especially during low temperature measurement. Therefore we have used a TE\textsubscript{01}\text{\textsuperscript{d}} mode post dielectric resonator [4,14,15] as shown in Fig. 1. The dielectric under test is placed over a low loss support material.

The experimental system used for the microwave characterisation is shown in Fig. 2, which consists of Network Analyser (HP 8722C), closed cycle refrigerator (APD DE-204), temperature controller (LTC-10), vacuum Dewar, a PC and the TE\textsubscript{01}\text{\textsuperscript{d}} mode dielectric resonator. The resonator containing the LiF sample was cooled from room temperature to approximately 13 K. The TE\textsubscript{01}\text{\textsuperscript{d}} mode resonance is identified around 8 and 16.4 GHz for the sample of sizes 15 × 7.6 mm and 5 × 3.12 mm, respectively. The S\textsubscript{21}, S\textsubscript{11} and S\textsubscript{22} parameters data around the resonance were measured at the lowest temperature. TMQF technique [16] has been used to eliminate all kinds of parasitic losses and to precisely compute coupling coefficients \(k_1\) and \(k_2\). The unloaded \(Q\)-factor is calculated using [17]:

\[
Q_0(T) = \frac{Q_L(T)}{1 + k_1(T) + k_2(T)}
\]

where \(Q_L\) is the loaded \(Q\)-factor.

The \(S_{21}\) parameter is measured as a function of temperature from 20 to 290 K. The coupling coefficient for each measurement temperature is calculated using a simplified TMQF [18] and hence the unloaded \(Q\)-factor. The perpendicular component of the real part of relative permittivity and loss tangent (\(\tan\delta\)) of LiF was computed from the measured resonant frequency and unloaded \(Q\)-factor respectively using numerical methods.

3. Results and discussion

We have characterised two Lithium Fluoride crystals at frequencies 8 and 16.4 GHz, respectively. Each LiF crystal under test forms as a dielectric post resonator. Fig. 3 shows the measured unloaded \(Q\)-factor of both the LiF resonators. It is interesting to note that the \(Q\)-factor is higher at higher frequency. This attributes to the low energy-filling factor of the smaller sample. The loss tangent of the LiF crystal is calculated from the unloaded \(Q\)-factor and is shown in Fig. 4 at frequencies 8 and 16.5 GHz. In the calculation of loss tangent we have accounted for the losses in the copper cavity walls. The loss tangent is higher at higher frequency as expected. The \(\tan\delta\) shows a linear variation with respect to frequency throughout the temperature.

The \(\tan\delta\) of different Fluoride crystals such as Calcium Fluoride, Magnesium Fluoride and Barium Fluoride are compared with LiF and shown in Fig. 5 at a frequency of 16.5 GHz [7–9]. The losses of LiF at microwave frequencies are higher than its counterparts.

Fig. 6 shows the variation of resonant frequency of the resonator with temperature. The resonant frequency shifted by around 250 MHz (1.6%) at 16.5 GHz frequency region and 260 MHz (3.3%) at 8 GHz frequency region when the temperature decreases from 290 to 15 K.

The real part of relative permittivity is calculated from the frequency data shown in Fig. 6 using numerical techniques and the variation is illustrated in Fig. 7. The difference between the permittivities of the two samples is between 1.2 and 0.7%. This discrepancy appears due to the non-uniformity of the sample surface. The height of the smaller sample is approximately
uniform but the non-uniformity in height of the bigger sample is around 0.5%. Typically the error in permittivity is twice the error in dimensions and hence a maximum error of 1% is expected. The coefficient of linear thermal expansion data is not available at low temperatures and hence the dimensions are assumed as a constant at varying temperatures. When the temperature increases from 15 to 290 K the permittivity increases by 6% in both cases. It is estimated that the real shift in permittivity will be around 5% if the change in dimensions due to the temperature variation is accounted in the permittivity computation.

Fig. 8 illustrates the permittivity variation of different Fluoride crystals. The variation of permittivity of LiF is the highest among other Fluorides however the relatively high permittivity of LiF will assist the miniaturized circuit and device fabrication for communication applications.

4. Calculation of parameters—$Q_0 \times f_0$, temperature coefficient of $f_0$ and permittivity

In this section the variations of $Q_0 \times f_0$, temperature coefficient of frequency and permittivity with temperature is studied for LiF. Microwave Engineers quite often use the value of $Q_0 \times f_0$ as a constant for a given material. This may not be necessarily true in all cases. As it is shown in Fig. 3 the $Q_0$-factor at frequency of 16.5 GHz is higher than the $Q_0$ at 8 GHz. Therefore, in this particular case of measurement $Q_0 \times f_0$ will be higher at higher frequencies as shown in Fig. 9. $Q_0 \times f_0$ is not a constant as expected due to the difference in the energy filling
factors of the resonator loaded with dielectric sample of different dimensions.

The shift in resonant frequency of a dielectric resonator depends on the coefficient of linear thermal expansion and the temperature coefficient of permittivity of the dielectric material used [19]. The temperature coefficient of resonant frequency ($\tau_f$) of the dielectric resonator, temperature coefficient of permittivity ($\tau_\varepsilon$) and the linear expansion coefficient ($\alpha$) of the dielectric material can be related as follows [20,21]:

$$\tau_f = A_3 \tau_\varepsilon + A_d \alpha + \tau C_x$$

(2)

where: $A_3 = (\varepsilon_0 f_0 \Delta f_0 / \Delta \varepsilon_0)$ and $A_d = (D f_0 / \Delta D) + (L / f_0) (\Delta D / \Delta L)$ and $\tau C_x$ describes temperature properties of the cavity and can be considered 0 if dimensions of the cavity remains constant with changes in temperature. From theoretical analysis of the resonant structure one can estimate the values of $A_3$ and $A_d$ and their values are approximately $-0.5$ for $A_3$ and 1.0 for $A_d$ [20].

The variation of resonant frequency with temperature is crucial in many microwave applications. The temperature coefficient of frequency, $\tau_f$, of a material gives an indication of the frequency stability of circuits or devices. The $\tau_f$ can be defined as [21]:

$$\tau_f = \frac{f_{0R} - f_{0T}}{f_{0R}} \frac{10^6}{T_R - T}$$

(3)

where $f_{0R}$ and $f_{0T}$ are the resonance frequency at room temperature and at any temperature, $T$. Fig. 10 shows the temperature coefficient of frequency of LiF. As discussed in the previous section, the shift in resonant frequency is same in both cases. Therefore the $\tau_f$ will not be a constant. We have observed a 100% difference in the value of $\tau_f$ between the two cases. A similar study was carried out using a Calcium Fluoride Hakki–Coleman type resonator and a CaF$_2$ dielectric post resonator [22]. It was remarkable to note that $\tau_f$ calculated using both techniques differs significantly as in this case. This establishes that the temperature coefficient of frequency is not a material property as it is contemplated.
The temperature coefficient of permittivity can be calculated using the equation:

\[
\tau_f = \frac{\varepsilon_{0R} - \varepsilon_{0T}}{\varepsilon_{0R}} \left( \frac{T}{T_R} \right) \times 10^6
\]

where \(\varepsilon_{0R}\) and \(\varepsilon_{0T}\) are the permittivity at room temperature and at temperature \(T\).

Fig. 11 shows the calculated \(\tau_f\) of the LiF crystal from the permittivity data shown in Fig. 7. The maximum discrepancy between the two data is around 6%. This discrepancy could be able to reduce further with highly polished samples and by accounting the coefficient of linear thermal expansion. Therefore, temperature coefficient of permittivity is a material property and will be constant for a given material irrespective of the measurement techniques at a given temperature.

5. Conclusions

The complex permittivity of LiF is measured as a function of temperature from 15 to 290 K using a dielectric post resonator at frequencies 8 and 16.5 GHz. Table 3 shows the perpendicular component of the real part of relative permittivity, loss tangent, \(Q_0\times f_0\), temperature coefficient of frequency and temperature coefficient of permittivity at temperatures 20 and 290 K at frequencies 8 and 16.5 GHz. The relatively high permittivity and low loss formulates LiF as a suitable entrant in many microwave circuits. The systematic research work reported in this paper look into the details of different material parameters that can be used as a standard. It has been proved that \(Q_0\times f_0\) and \(\tau_f\) are not constant for a given material and hence cannot be used as a material property. The \(Q_0\times f_0\) and \(\tau_f\) depends on the type of measurement technique used and other parameters of the resonators such as energy filling factor and geometrical factors. However, the temperature coefficient of permittivity is a constant for a given material at a particular temperature.

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