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Accurate Characterization of the Temperature Coefficient of Permittivity of Sapphire Utilizing the Dual-Mode Frequency Locked Technique

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***Abstract*- In this work we present a new method to obtain accurate characterization of the Temperature Coefficient of Permittivity (TCP) of an anisotropic crystal, which we name the Dual-Mode Frequency Locked (DMFL) technique. The technique requires precise simultaneous frequency measurements of two orthogonally polarized modes as a function of temperature. This was achieved by configuring a sapphire resonator as a Pound Locked Dual-Mode oscillator and measuring the frequencies with a frequency counter, while simultaneously recording the temperature with a platinum thermometer. From the two data files of frequency versus temperature, and the knowledge of electric and dimensional filling factors (obtained from Finite Element analysis), it was possible to calculate the integrals of the TCP without resorting to polynomial fits. This resulted in a precise model of the components of the TCP between 50 to 77 K for sapphire. The model is accurate enough to predict within a few degrees K, the frequency turnover temperatures of the difference frequency of a Dual-Mode oscillator.**

I. INTRODUCTION

Microwave resonator-oscillators with the best-recorded short-term frequency stability are based on sapphire crystal resonators excited in Whispering Gallery (WG) modes. The frequency stability of a resonator-oscillator is based primarily on two things: the Q-factor of the resonator, and temperature stability. Sapphire WG resonators have extremely high Q-factors, however they also have a large Temperature Coefficient of Permittivity (TCP), which means that temperature fluctuations will inevitably affect the resonance frequency. Previously we proposed a new method to design temperature compensated oscillators from the difference frequency of orthogonally polarized WG modes (Patent Pending) [1, 2]. Although the principle was verified experimentally, we showed that previously measured values of the TCP were not accurate enough to predict the right temperature of compensation [2].

The fractional temperature coefficient of permittivity (in units K^{-1}) in sapphire is anisotropic. If the dielectric resonator is excited with WGH (quasi-Transverse Magnetic) and WGE (quasi-Transverse Electric) modes simultaneously, it is possible to design the resonator so that the absolute Temperature Coefficient of Frequency (TCF in units Hz/K) of the two modes will be the same at some temperature. Thus the TCF of the beat frequency will be annulled [1]. This is impossible to achieve in an isotropic resonator, as the fractional TCF is approximately half the fractional TCP for all modes as well as the difference between modes. This new technique requires no paramagnetic [3], mechanical [4], or dielectric compensation techniques [5].

The DMFL technique has been successfully implemented between solid and liquid nitrogen temperatures. An accurate model of the components of the TCP for anisotropic sapphire has been a result of these measurements, which has enabled the predictions of compensation temperatures to within a few K. Thus, this work contains the necessary information to design a compensated resonator oscillator by simply choosing the modes and dimensions of the resonator. Plans are already underway to build an oscillator based on a measured compensation temperature at 50.6K (see Hartnett et. al. in these proceedings).

II. TEMPERATURE DEPENDENCE OF WHISPERING GALLERY MODES IN SAPPHIRE

Whispering Gallery (WG) modes in dielectric resonators are denoted as $WGH_{m,n,p}$ and $WGE_{m,n,p}$, where *m* is the number of azimuthal variations, *n* is the number of radial nodes, and *p* is the number of axial nodes. The fundamental mode families are $WGE_{m,0,0,}$ with dominant field components of E_r and H_z, and $WGH_{m,0,0}$, with dominant field components of H_r and E_z In this work we study WG modes in uniaxially anisotropic single crystal sapphire. In order to obtain a good Q-factor it is important that the axis of anisotropy is parallel to the cylinder axis. A schematic of the sapphire resonator is shown in Fig. 1. To excite the WGH mode an E_z field is excited, with a straight antenna probe from the top. To excite the WGE mode an E_r field is excited, with a straight antenna probe from the side.

All WG modes are hybrid and have a small amount of the non-dominant field component. The WGH modes are those with dominant axial electric field components, whereas the WGE modes are those with dominant axial magnetic field components. These mode identities are confirmed by calculating the electric energy filling factors in the perpendicular and parallel directions. These were computed

using finite element analysis, according to the following definitions:

$$p_{e_\perp}{}^H = 2\left|\frac{\partial f^H}{\partial \varepsilon_\perp}\right|\frac{\varepsilon_\perp}{f^H} \qquad p_{e_{//}}{}^H = 2\left|\frac{\partial f^H}{\partial \varepsilon_{//}}\right|\frac{\varepsilon_{//}}{f^H} \tag{1}$$

$$p_{e_\perp}{}^E = 2\left|\frac{\partial f^E}{\partial \varepsilon_\perp}\right|\frac{\varepsilon_\perp}{f^E} \qquad p_{e_{//}}{}^E = 2\left|\frac{\partial f^E}{\partial \varepsilon_{//}}\right|\frac{\varepsilon_{//}}{f^E}$$

The electric energy filling factors range between zero and one. They are a measure of the total fraction of electric field energy, in the dielectric perpendicular or parallel to the crystal axis. Values for the fundamental WGE and WGH mode families are shown in Fig. 2. Note that for WG modes $p_{e_\perp} + p_{e_{//}} \approx 1$.

Whispering Gallery modes mainly depend on the expansion and contraction in the radial direction and very weakly in the axial direction, since the modes have small field variations in the axial plane. As azimuthal mode number increases, the dependence of mode frequency on the diameter of the dielectric resonator also increases and the dependence on length decreases. This dependence is expressed as the dimensional filling factors, given by;

$$p_D = \frac{\partial f}{\partial D}\frac{D}{f} \qquad p_L = \frac{\partial f}{\partial L}\frac{L}{f} \tag{2}$$

Here p_D is the fraction of energy that propagates in the radial and azimuthal directions, and p_L is the fraction of energy that propagates in the axial direction. These values were calculated by utilising the finite element analysis [6] and are plotted in Fig. 3.

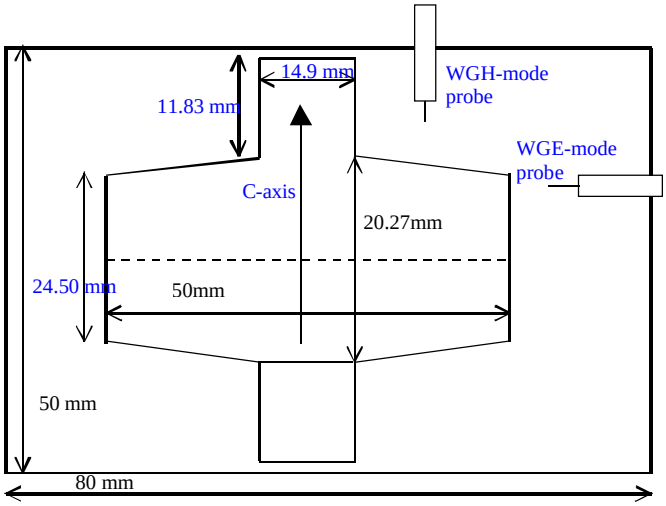

Fig. 1. The dimensions of the sapphire monocrystal and cavity

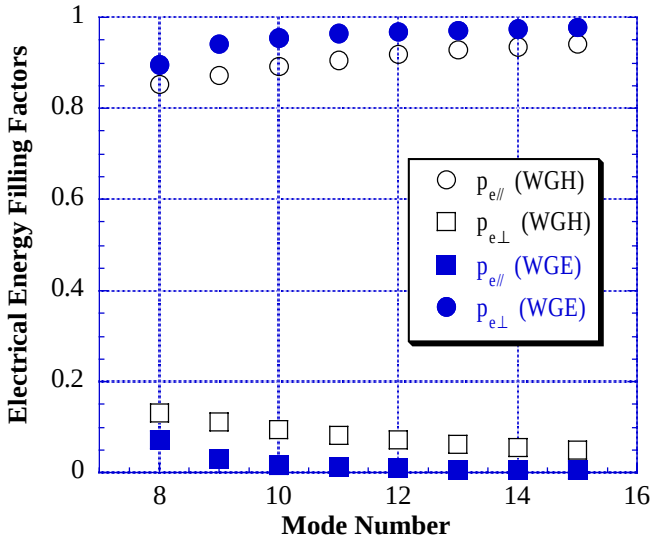

Fig. 2. Parallel and perpendicular electric filling factors for WGH and WGE modes at T=60K

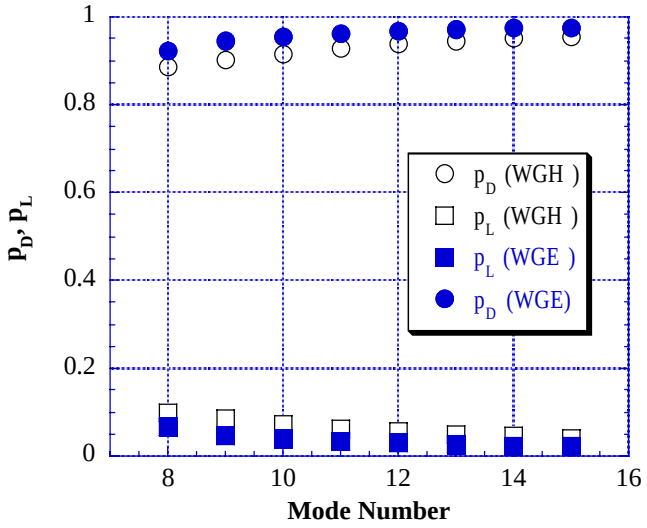

Fig. 3. Parallel and perpendicular dimensional filling factors for WGH and WGE modes at T=60K

Once the filling factors are calculated it is straightforward to calculate the fractional TCF of the resonator from the TCP and expansion coefficients of the material [7-9], using;

$$\frac{1}{f}\frac{\partial f}{\partial T} = -p_{e\perp}\frac{\alpha^*_{\varepsilon\perp}}{2} - p_{e//}\frac{\alpha^*_{\varepsilon//}}{2} - p_D\alpha^*_D - p_L\alpha^*_L$$
$$\alpha^*_\varepsilon = \frac{1}{\varepsilon}\frac{\partial \varepsilon}{\partial T} \qquad \alpha^*_D = \frac{1}{D}\frac{\partial D}{\partial T} \qquad \alpha^*_L = \frac{1}{L}\frac{\partial L}{\partial T} \tag{3}$$

where, $\alpha^*_{\varepsilon\perp}$ is the fractional TCP perpendicular to the anisotropy axis, $\alpha^*_{\varepsilon//}$ is the fractional TCP parallel to the

anisotropy axis, α_D^* is the fractional thermal expansion coefficient parallel to the anisotropy axis, and α_L^* is the fractional thermal expansion coefficient perpendicular to the anisotropy axis.

The physical dimensions of most materials expand with heating and contract with cooling. This is also true for sapphire, the expansion coefficients perpendicular and parallel [10] are shown as a function of temperature in Fig. 4., along with the TCP's divided by two [7, 11]. It is chosen to compare them as such because the resonance frequency is inversely proportional to dimension and inversely proportional to the square root of the permittivity. Thus the TCF given in Equation (3) is affected by these quantities in these proportions.

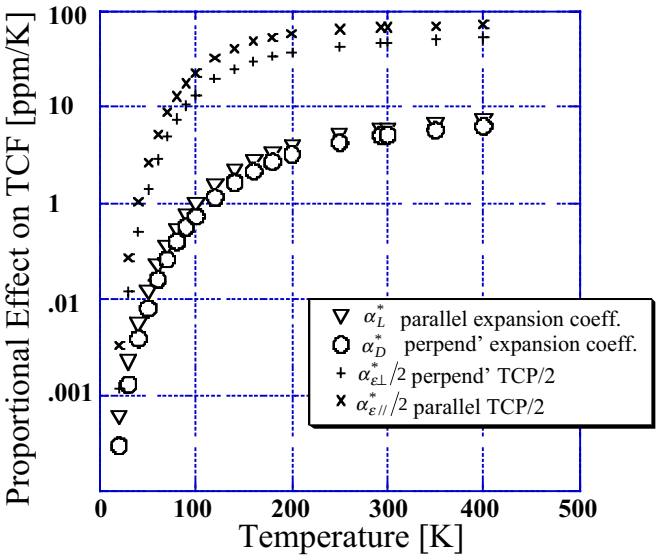

Fig. 4. Sapphire expansion coefficients $\alpha^*_D(T)$, $\alpha^*_L(T)$ [10] and TCP's $\alpha^*_{\varepsilon//}(T)/2$, $\alpha^*_{\varepsilon\perp}(T)/2$ from data collected at UWA from T = 4 K to T = 400 K [7].

It should be noted that the effect of the TCP's on the TCF's are over an order of magnitude larger than the expansion coefficients, and thus are the dominant source of the resonance frequency temperature dependence. The expansion coefficients, although smaller, are still significant and must be taken into account when analysing frequency-temperature compensation effects.

II. Experimental

The sapphire monocrystal and cavity were cleaned with ethanol, nitric acid and hydrofluoric acid before being placed into the copper cavity and vacuum chamber. The sapphire was clamped in a copper cavity (see Fig. 1.) of dimensions 80 mm diameter and 50 mm height. The sapphire crystal has diameter 50 mm with height 20.27 mm at the centre tapering to 24.50 mm at the edges. Only one of the support posts of the sapphire was clamped in the cavity, in order to avoid compression and inevitable resultant drifts in the resonance. Probes were set through holes in the cavity walls. Then the experimental chamber was evacuated, and placed in a cryogenic dewar. An indium seal was utilised to maintain a good vacuum. This was monitored via a Parani Gauge. The dewar was filled with liquid nitrogen and pumped on, using a rotary pump, until it was solid. Helium exchange gas was introduced into the cavity during this process to increase the rate of cooling of the dielectric resonator. Once at 50 K, the Helium gas was pumped out to create a vacuum. Measurements of temperature were made using a calibrated platinum resistance thermometer inserted into the base of the cavity.

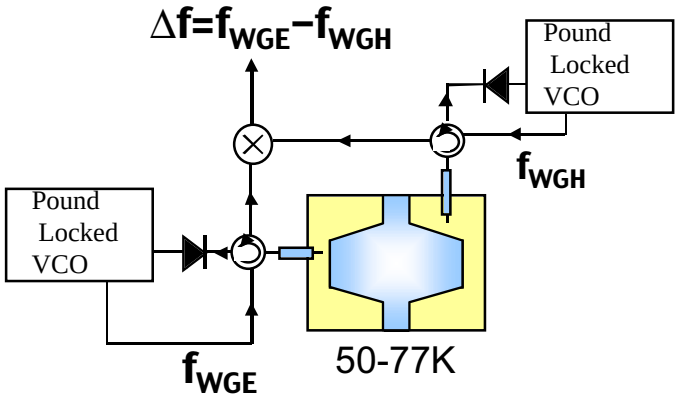

Fig. 5. Schematic of the Dual-Mode oscillator circuit.

Two identical Pound frequency-locking systems were implemented; one for each resonance. Fig. 5. shows a schematics of the 'locked' dual mode oscillator. Initially, a signal close to the resonance frequency is generated by mixing a 1 GHz RF signal from a Voltage Controlled Oscillator (VCO), with a stable microwave signal from a microwave synthesizer. The desired signal is filtered to select one side band, then amplified, and monitored via a frequency counter. It is then sent through a voltage controlled phase shifter, which is driven with a 100 kHz signal from a Lock-in amplifier. Thus a phase modulated microwave frequency interrogates the resonance. This resonance is detected in reflection via a diode detector, which demodulates the reflected Amplitude Modulation (AM) component. The output of the detector is then sent back to the Lock-in Amplifier. The Lock-in Amplifier compares the AM Demodulated signal from the detector to the 100 kHz signal that modulates the Voltage Controlled Phase shifter, which demodulates the 100 kHz signal down to DC. This is set phase sensitive by choosing the phase of the 100 kHz signal generated by the lock-in amplifier. The result is a DC signal, which traces out an error curve as the microwave signal is swept across the resonance frequency, which is used to lock the synthesizer to the resonance frequency. Two near identical systems were built for each mode.

Once the two modes are locked, the temperature characteristics of the dual mode oscillator were measured. The system was cooled to solid nitrogen temperatures by means of pumping on the liquid nitrogen bath, then the pump was turned off and the system slowly warmed to liquid nitrogen temperature. During this period the difference frequency, temperature (measured in ohms from the platinum thermometer), and the frequency of one of the mode pairs were measure continuously.

III. New Method for the Measurement of Temperature Coefficient of Permittivity

We have developed a new technique dubbed "Dual-Mode Frequency Locked" (DMFL) technique to determine the Temperature Coefficients of Permittivity for anisotropic dielectric resonators. It is similar to the previous "Whispering Gallery" (WG) mode method described in [7-9]. The WG mode method consists of measuring the frequency of differently polarised modes as a function of temperature. These modes are measured individually and in succession. Since the temperature points in the data are not equal, polynomial fits to the frequencies are necessary. Then the derivative of the frequencies is taken and Eq. (3) is solved simultaneously for the two modes, in order to find the TCP's. Drawbacks to this technique include polynomial fit errors in the slope, and systematic errors in the temperature due to cycling between measurements. For example, if the two individual measurements are taken as the resonator warms from solid to liquid nitrogen temperatures, the rate of the measurements may be different, resulting in different systematic errors. Also, it was clear before the outset of this work, the precision of the previous measurements of the TCP's using the WG method (see fig. 4) were not accurate enough to predict turning points precisely [2].

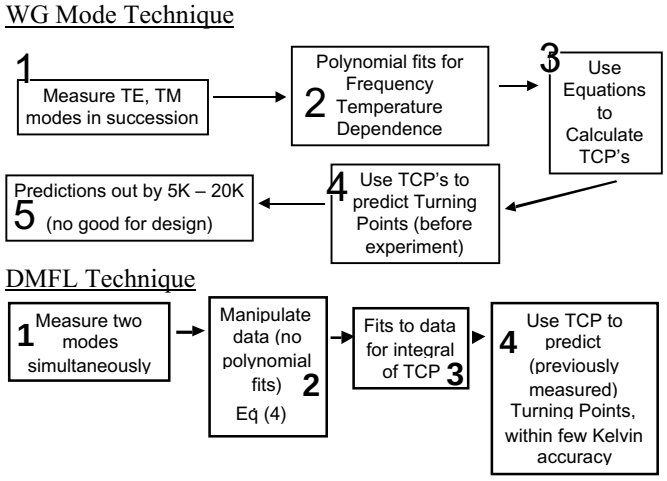

Fig. 6 Flow-charts that compare the method of calculating TCP's and using them to predict turnover temperatures.

The DMFL technique overcomes these problems by taking the frequency measurements of both modes simultaneously. This eliminates systematic effects associated with successive measurements. Moreover, as both frequencies are measured at the same temperature, data sets may be processed easily without the necessity of polynomial fits or interpolation early in the data processing. Flow charts representing a comparison between the WG mode and DMFL techniques are shown in Fig. 6.

By integrating Eq. (3) for both the WGH and WGE mode we may write;

$$\begin{pmatrix} \int_{T_S}^{T} \alpha^*_{\varepsilon//}(T)dT \\ \int_{T_S}^{T} \alpha^*_{\varepsilon\perp}(T)dT \end{pmatrix} = -2P_e^{-1}P \begin{pmatrix} \int_{T_S}^{T} \alpha^*_{L}(T)dT \\ \int_{T_S}^{T} \alpha^*_{D}(T)dT \end{pmatrix} - 2P_e^{-1} \begin{pmatrix} \frac{f_{WGE}(T)}{f_{WGE}(T_s)} \\ \frac{f_{WGH}(T)}{f_{WGH}(T_s)} \end{pmatrix} \quad (4)$$

The left-hand side of (4) is the matrix of unknowns (the TCP's $\alpha^*_{\varepsilon//}$ and $\alpha^*_{\varepsilon\perp}$). On the right hand side, P_e and P are the electric and dimensional filling factor matrices, given by (5).

$$P_e = \begin{pmatrix} p^{WGE}_{e//} & p^{WGE}_{e\perp} \\ p^{WGH}_{e//} & p^{WGH}_{e\perp} \end{pmatrix} P = \begin{pmatrix} p^{WGE}_{L} & p^{WGE}_{D} \\ p^{WGH}_{L} & p^{WGH}_{D} \end{pmatrix} \quad (5)$$

Filling factors were evaluated using finite element analysis (see Fig. 2. and 3.), and α^*_L and α^*_D were obtained from the literature (Fig. 4). The furthermost right matrix in (4) gives the normalised frequencies of the two modes. The integrations are performed around an arbitrary temperature T_s. The frequencies are thus normalised with respect to the frequency f_s at T=T_s. T_s is chosen to be near to the mid point of the temperature range of interest. Consequently all of the variables are known except for the integrals of the perpendicular and parallel TCP's. After processing the data according to the simultaneous Equations (4), data sets for the integrals of the TCP's were obtained. Polynomial fits to the final data sets were differentiated to determine the TCP's. Since the differentiation was only completed at the end of the data analysis process, polynomial fit errors do not propagate through the data processing.

IV. Is the Temperature Coefficient of Permittivity Dispersive?

To properly predict turning points of large frequency differences, it was imperative to determine whether the TCP

was dispersive or not. In order to investigate the possibility of dispersion within the TCP's, three pairs of orthogonally polarised modes with minimal frequency difference were measured over the entire temperature range. These three pairs and their frequencies at solid nitrogen temperature are listed in Table 1.

TABLE I
Listing of the WGH/WGE mode pairs used to calculate the TCP's. Frequencies are given at solid nitrogen temperature, near 50 K.

Label	WGH $_{m,0,0}$	Frequency	WGE $_{m,0,0}$	Frequency
A	m = 10	7.857 [GHz]	m = 8	7.8097 [GHz]
B	m = 11	8.473 [GHz]	m = 9	8.498 [GHz]
C	m = 12	9.086 [GHz]	m = 10	9.1882 [GHz]

Three different calculations of the TCP were made from the mode pairs listed in table 1, however no clear frequency dependence was observed within the precision of the technique. Fifth order polynomials were fitted to the calculated integrals of TCP (from the data process) and the TCP was calculated by differentiating, resulting in a fourth order polynomial description of the TCP. Results are shown in Figs. 7 and 8.

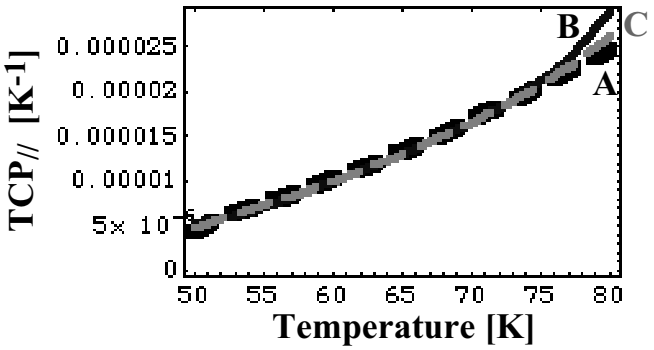

Fig. 7. Parallel TCP's from the similar frequency measurements as a function of temperature [K], with curves labeled A, B, and C as the TCP's calculated from the mode pairs given above.

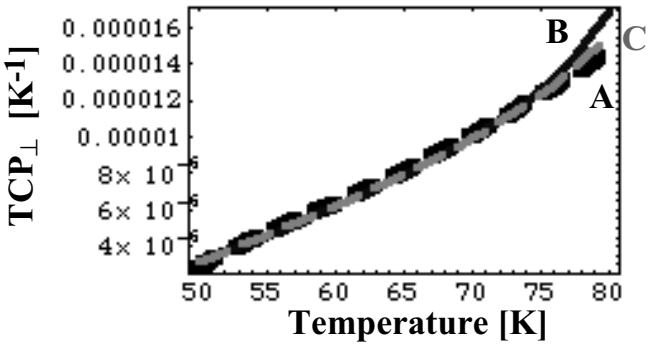

Fig. 8. Perpendicular TCP's from the similar frequency measurements as a function of temperature [K], with curves labelled A, B, and C as the TCP's calculated from the mode pairs given above.

These polynomials intersect at many values of temperature. Near the boundaries of the temperature range the curves diverge, this is believed to be due to edge effects of the polynomial fits and not due to any frequency dependence. From these results we take the average of the three polynomials. To discount the edge effects the polynomial fits are only considered to be valid between 55K to 75K. The average polynomials are given by (now referred to as the DMFL average):

$$\alpha^*_{//} = 1.74832*10^{-4} - 1.22892*10^{-5}T + 3.15919*10^{-7}T^2 - 3.50363*10^{-9}T^3 + 1.48153*10^{-11}T^4 \quad (6)$$

$$\alpha^*_{\perp} = 7.36247*10^{-5} - 5.50947*10^{-6}T + 1.48597*10^{-7}T^2 - 1.70298*10^{-9}T^3 + 7.41397*10^{-12}T^4 \quad (7)$$

Another method was attempted to determine whether there was any measurable frequency dispersion at higher frequency in the TCP's. This method determined the perpendicular TCP's only, by exploiting the fact that the WGE modes dependence on the parallel TCP is minimal. Thus, it was assumed that the DMFL average for the parallel TCP given Eq. (6) was valid between 9.8 to 12.6 GHz. Eq. (3) was then applied to the data set of the WGE modes listed below in table 2. The perpendicular TCP's using this method were then calculated. There was no definite indication of any frequency dependence up to 12.6 GHz within the precision of this technique, although more scatter in the calculation of the TCP's was prevalent in the data.

TABLE II
Listing of the WGH/WGE mode pairs, that exhibited a turnover temperature between solid and liquid nitrogen temperature. They were also used to check if the TCP was dispersive up to 12.6 GHz. Frequencies are given at solid nitrogen temperature, near 50 K.

Label	WGH $_{m,0,0}$	Frequency	WGE $_{m,0,0}$	Frequency
D	m = 8	6.624 [GHz]	m = 11	9.876 [GHz]
E	m = 9	7.242 [GHz]	m = 13	11.246 [GHz]
F	m = 10	7.857 [GHz]	m = 14	11.946 [GHz]
G	m = 12	9.086 [GHz]	m = 15	12.605 [GHz]

V. Calculation of Turnover Temperatures From the Temperature Coefficient of Permittivity

Previous calculations of the TCP were shown to be not accurate enough to make consistent predictions of the turn over temperature of the Dual-Mode oscillator [2, 7, 9, 12, 13]. In the following analysis calculations of the turnover temperatures are compared with measurement. Turnover temperature points were predicted using the following methodology. First the calculated TCP's were substituted into Equation (3) along with appropriate filling factors and the dimensional coefficients, in order to find the TCF's for each of the modes listed in Table II. Then the turnover temperature was determined by evaluating the temperature at which the

TCF's of the mode pairs (given in table II) were identical. These predictions were then compared to the measured values (table III). Since the DMFL average was not valid in the temperature range of the WGH_{12} and WGE_{15} mode pair, another comparison was made to assess the DMFL technique. Comparisons were made between the TCP's from mode pairs that could be identified as valid in the temperature range of the turning point.

TABLE III.
Comparison between measured and predicted values of turnover temperatures, brackets indicate the difference from the measured value. Here † indicates no prediction of a turning point, and * indicates that the DMLF average in (6) and (7) was not used as it was not valid for the temperature turnover range. Instead only the data generated from mode pair D (rather than the average) was implemented as it was the only mode valid over this temperature range.

Mode pair / Beat Freq.	Measured [K]	Flory TCP [K]	UWA TCP [K]	DMFL technique
D / 3.252 [GHz]	58.6	†	66.2 (+7.6)	59.7 (+1.1)
E / 4.004 [GHz]	70.2	†	70.3 (+0.1)	69.6 (-0.6)
F / 4.089 [GHz]	63.4	†	66.0 (+2.6)	60.5 (-2.9)
G / 3.519 [GHz]	50.6	70.3 (+19.7)	57.4 (+6.8)	51.5* (+0.9)

It is clear from the comparison in table 3 that the more consistent predictions are made with the DMFL technique. Most calculations are only of order 1 K of the measured value using the DMFL technique. It is believed the mode pair F exhibits a larger error due to the 11.9 GHz mode being in the proximity of a Cr^{3+} paramagnetic resonance.

VI. Conclusion

Precision measurements utilizing the DMFL technique have allowed the accurate characterization of the TCP's of sapphire. This technique is general and may be applied to any uniaxial anisotropic material, over any temperature range. For future design of temperature compensated oscillators at other temperature ranges (such as room temperature) similar measurements and calculations are necessary to determine the TCP over a broader temperature range.

Acknowledgements

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