

# FREQUENCY TEMPERATURE COMPENSATION IN COMPOSITE MICROWAVE DIELECTRIC RESONATORS

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## ABSTRACT

In this paper we present the performance of two different composite dielectric resonators. The composite whispering gallery mode (WGM) dielectric resonator consists of sapphire puck sandwich between two thin rutile rings and supported by two quartz rods in a cylindrical copper cavity. Frequency-temperature turnover temperature above liquid nitrogen temperature with a Q-value  $\sim 10^6$  in WG mode is observed in the frequency range 8 GHz to 12 GHz. Frequency-temperature compensation is also observed in a stack resonator comprising of two polycrystalline bulk ceramics having opposite coefficient of permittivity. The materials studied are  $\text{Ba}_5\text{Nb}_4\text{O}_{15}$  (BN) and  $5\text{ZnO-Nb}_2\text{O}_5$  (ZN), synthesized by the conventional solid-state ceramic route. It is observed that the stack configuration yields temperature compensation over a large temperature range but at the cost of degraded Q-value.

## INTRODUCTION

Dielectric resonators based on microwave ceramics are of great importance for applications in frequency stabilized oscillators or as resonators in microwave filters and antennas. In the last decade new dielectric compositions have been developed in achieving high permittivity, high quality factor, low temperature coefficient of the resonant frequency,  $\tau_f$  and low fabrication cost. Microwave whispering gallery dielectric resonators based on single crystal sapphire or  $\text{LaAO}_3$  have the potential for very high unloaded quality factor,  $Q_0$  in the range  $10^5$  to  $10^6$  at cryogenic temperatures.  $\text{TE}_{011}$  cylindrical mode in a sapphire loaded dielectric resonator between two high temperature superconducting thin films yielded relatively high  $Q_0$  and considered useful as stabilizing elements in oscillators providing superior phase noise performance. Unfortunately, high  $\tau_f$  of single crystals has limited its applications. In polycrystalline dielectrics the  $\tau_f$  are tuned by appropriate doping so as to make  $\tau_f$  approaching zero at some particular temperature. Several other methods of tuning  $\tau_f$  of resonators have also been proposed and tested. This includes (i) stacking two cylindrical resonators made of different materials with  $\tau_f$  of opposite sign [2] (ii) mechanical compensation in which two similar sapphire resonators are held apart by a copper post. The relative difference in the thermal expansion of sapphire and the copper post, at 87 K, cancelled the temperature induced frequency shift of the sapphire's resonant mode due to strong tuning effect of the gap spacing between the two crystals [3] (iii) by using paramagnetic susceptibility effects of impurity ions in sapphire crystal to compensate the permittivity-temperature dependence. Temperature compensation was observed in the range 5 to 13 K with  $\text{Cr}^{3+}$  and  $\text{Ti}^{3+}$  ions and between 4 and 80 K depending on the magnetic-energy-filling factor in the sapphire and the  $\text{Ti}^{3+}$  ion concentration [4]. (iv) using composite dielectric structure consisting of more than one low loss monocrystal. Tobar et. al.[5] developed a composite dielectric structure consisting of two thin slices of rutile single crystal clamped tightly against the upper and lower surfaces of a cylinder of sapphire. The temperature coefficient of permittivity (TCP) of sapphire and rutile are of opposite sign and thus the temperature coefficient of the composite resonator may be cancelled. The composite structures, however, suffer from its complexity, cost, and presence of a large number of spurious modes. To reduce the spurious modes density, rutile rings, rather than discs, were employed [6]. A high Q-factor of  $3 \times 10^7$  was achieved with an annulment temperature of 56 K for the WG quasi TM modes

In this paper we present the performance of composite sapphire-rutile dielectric resonators for WG modes and stack resonators comprising of two polycrystalline bulk ceramics having opposite coefficient of

permittivity. The composite resonator operating in the WG mode in the range 8 GHz to 12 GHz shows the turnover temperature around liquid nitrogen temperature with a Q-value  $\sim 10^6$ . The frequency temperature dependence of the stack  $\text{Ba}_5\text{Nb}_4\text{O}_{15}$  (BN)/  $5\text{ZnO-Nb}_2\text{O}_5$  (ZN) resonator is achieved compensation over a large temperature range depending on the volume fraction of the dielectrics.

## Experimental

Microwave dielectric property measurement as a function of temperature was done using a microwave setup with a cryogenic attachment. The cryogenic system is a commercial cryo-cooler (APD-204S) in which cooling is performed by means of a thermodynamic close cycle of helium gas. The dielectric resonator was mounted on the cold head of the cryocooler taking utmost care to establish good thermal contact between the cavity and the cryocooler using indium foil. The chamber was evacuated to pressure less than  $10^{-5}$  mbar in order to reduce the convection losses. Radiation losses are minimized using a thermal shield maintained at 77K by attaching to the cryostat first stage. Microwave was coupled in and out of the DR through two opposite loop probes using low loss microwave cables. The DR was excited with microwave sweep oscillator HP8350B and the resonant frequency and Q-value of excited mode was measured using the full bandwidth at half power point method by scalar network analyzer (HP8757D). The DR was cooled down to 30 K and the temperature was then raised in steps. A temperature controller (Lake Shore 331S) was used to control the temperature by a PID regulator using a Si diode sensor and a heater fixed on the cold finger. A second Si-sensor was attached to the top of the cavity. The resonator was then allowed to warm up sufficiently slowly to keep it in thermal equilibrium. At each temperature point, the resonant frequency ( $f$ ), 3-dB bandwidth ( $\Delta f$ ) and insertion loss (IL) were recorded using a PC with an IEEE 488 data bus linking the sweep oscillator, scalar network analyzer and the temperature controller. A visual programming language HP VEE 7.0 was employed for controlling instruments, developing the data interface, creating a customized data display and data collection and storing the data into a data base for evaluation of the unloaded Q-value. The unloaded Q-value of the resonator was obtained, using the relation,  $Q_0 = Q_L [1 - 10^{-(IL/20)}]^{-1}$ , where  $Q_L (= f / \Delta f)$  is the measured loaded Q-value.

## WG MODE COMPOSITE DIELECTRIC RESONATOR

Whispering gallery mode (WGM) are modes of electromagnetic resonance that can be excited in a cylindrical monocrystal. In general it provides strong confinement of the electromagnetic field around the periphery of the dielectric/air interface and thereby the resonator unloaded quality factor, Q-value, is nearly inversely proportional to the loss tangent,  $\tan\delta$ . At low temperatures,  $T < 77$  K, the  $\tan\delta$  of sapphire, with positive temperature coefficient of permittivity (TCP) and rutile, with negative TCP, are fairly low, and the composite of the two can provide frequency-temperature compensation. We designed and developed a sapphire/rutile composite resonator consisting of two rutile rings, of 0.5mm thickness, an inner diameter of 5 mm and an outer diameter of 15 mm, held to the ends of a sapphire of diameter 30 mm and length 15 mm by two quartz crystals. The assembly was mounted in a silver coated copper cylindrical cavity of diameter 60 mm and length 36 mm, the composite being held together by a spring arrangement. The center symmetry of the sapphire/quartz was maintained by providing grooves in the sapphire crystal at both ends and notches at one end of the quartz crystals. The notches and the grooves were approximately 1 mm thick. Fig. 1(a) shows the photograph of the WGM resonator. The WG modes were excited in the dielectric, using coaxial loop, over the frequency range 8 GHz to 12 GHz. The resonator was initially measured at room temperature and data for WG mode with highest Q-value was recorded and then installed in the cryostat.

Measured variation of the resonant frequency and Q-value of WG mode resonance with sapphire only shows increase in the Q-value and the frequency as the temperature is lowered. The Q-value increases with decrease in temperature as loss tangent of sapphire,  $\tan\delta$  is strongly temperature dependent, falling rapidly as the temperature is reduced below room temperature. Q-value of  $4.93 \times 10^6$  is measured at 50K that corresponds to  $\tan\delta \sim 2 \times 10^{-7}$ .

Fig. 1(b) shows the experimental results obtained for frequency and Q-value of the WGH and WGE mode in sapphire/rutile composite, demonstrating the frequency-temperature compensation. The frequency of the same mode in the composite resonator in comparison to the bare sapphire is lower as the axial boundary condition at the sapphire-rutile causes the field to be squashed further into the sapphire. The Q-value of the composite resonator can be estimated from the  $\tan\delta$  values of sapphire and rutile :

$$Q(T) = [\kappa \tan \delta_r(T) + (1 - \kappa) \tan \delta_s(T)]^{-1} \quad (1)$$

where  $\kappa$  represents the electromagnetic energy filling factor of rutile. On cooling, the unloaded Q-value of the composite increases from  $7.5 \times 10^4$  at room temperature reaching maxima of  $1.8 \times 10^5$  at around 98 K and then decreases until it reaches a low value of  $1.09 \times 10^5$  at 43 K.

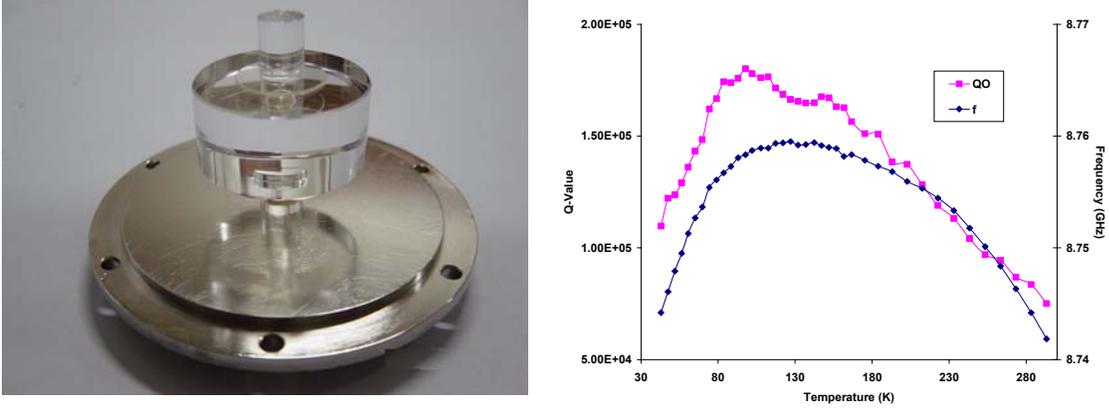


Fig. : Sapphire cylinder with perturbing rutile rings mounted on the base plate of the copper housing (b) Variation of measured frequency and Q-value of the composite WGH mode with temperature.

The change in the resonant frequency due to change in the dimensions of the sapphire and rutile is relatively very small compared to the change in permittivity with temperature. The frequency-temperature relationship for the composite resonator can be estimated from:

$$f(T) = f(0) \left[ \kappa \sqrt{\frac{\epsilon_r(0)}{\epsilon_r(T)}} + (1 - \kappa) \sqrt{\frac{\epsilon_s(0)}{\epsilon_s(T)}} \right] \quad (2)$$

Fig.1(b) shows annulment in the frequency-temperature dependent for the WGH mode at about 125 K. At turning point of 125K we recorded a maximum Q-value of  $1.69 \times 10^5$ , which is lower than in sapphire resonator at 125 K ( $=2.63 \times 10^5$ ). The Q-value, eq (1), depends on the ratio of the electrical filling factor in the sapphire and the perturbing rutile discs. This depends on the field structure of the excited resonant mode as well as on the field distribution at the sapphire/rutile interface. Other possible source of Q degradation is the air gap between the sapphire and rutile and dislocation in the crystals.

## STACK RESONATOR

For stack resonator two dielectric materials  $\text{Ba}_5\text{Nb}_4\text{O}_{15}$  (BN) and  $5\text{ZnO-Nb}_2\text{O}_5$  (ZN), having opposite  $\tau_f$  in the temperature range 20K to room temperature are placed on top of a single crystal sapphire crystal in a silver coated copper enclosure. The BN and ZN ceramics were synthesized by the conventional solid-state ceramic route under optimized conditions [7]. The temperature dependent permittivity was measured from the change in frequency in a  $\text{TE}_{011}$  mode shielded dielectric resonator [8]. The dielectric permittivity of BN is  $\epsilon_r \sim 38.39$  at 300 K and increases to 39.73 at 50 K, whereas for ZN  $\epsilon_r \sim 22.08$  at 300 K and decreases to 21.39 at 50K. The pallets of ZN, with different volume fraction  $V_{fZN}$  ( $=V_{ZN}/(V_{ZN}+V_{BN})$ ) were placed with axial symmetry on the top of BN. The schematic of the cavity is shown in the Fig.2(a). The samples were nearly 12 mm in diameter and were well polished and glued together using a very thin layer of low loss ceramic glue. The stack resonator acts as a single resonator resonating with a particular  $\text{TE}_{011}$  mode

frequency. The resonant frequency, Q-value and measured  $\tau_f$  varied with  $V_{fZn}$  and the measured values were within the experimental error when the order of BN and ZN are reversed.

Fig.2(b) shows the variation of the relative frequency shift of ZN (ZN1 to ZN5) stacked with BN with temperature and for different  $V_{fZn}$ . The  $V_{fZn}$  varied from 0.451 to 0.668. As  $V_{fZn}$  is increased the non-linear f-T behavior turns towards linear behavior. The stack resonator BN-ZN4 and BN-ZN5 shows almost a linear f-T behavior but with opposite  $\tau_f$ . The  $\tau_f$  becomes zero at different temperature depending on  $V_{fZn}$ . At 190 K the  $\tau_f$  estimated for BN, ZN and the stack BN- ZN4 and BN-ZN5 resonators are respectively +39.816 ppm/K, -35.483 ppm/K, +6.267 ppm/K and - 5.47 ppm/K. The Q-value of the stack resonators showed that although BN-ZN4 or BN-ZN5 yields temperature compensation but at the cost of degraded Q compared to BN-ZN2 that showed maximum Q-value  $\sim 11000$ . In general, it is observed that the  $Q \times f$  product decreased with the increase in  $V_{fZn}$  upto 0.61 and thereafter tends to increase.

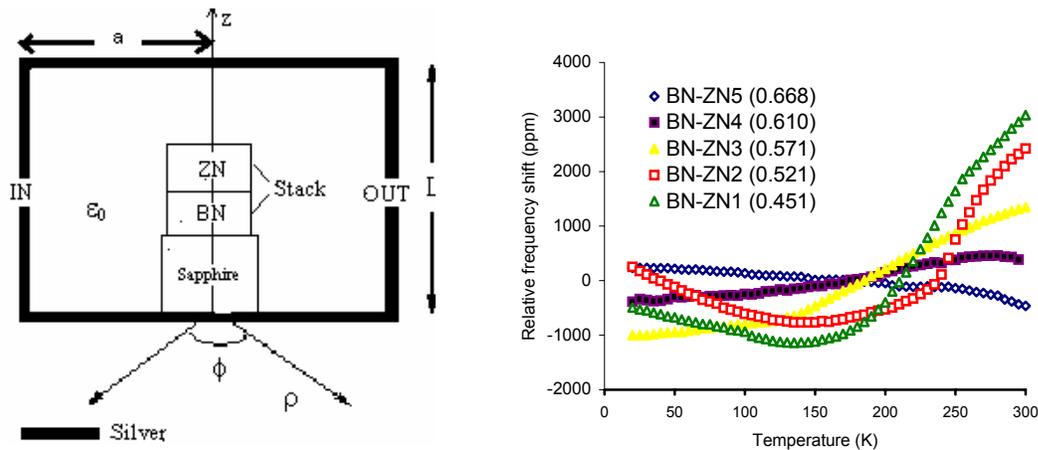


Fig.2(a): Schematic of the stack dielectric resonator, (b) temperature variation of relative frequency shift of the stack resonators with different  $V_{fZn}$ .

## CONCLUSION

High Q resonators can be used as a frequency discriminator for a stable oscillator proposed to be used for frequency standard. High sensitivity of the discriminator lowers the phase noise of the frequency stabilized oscillator. To minimize the frequency fluctuation in the discriminator the Q-value must be kept as high as possible. The present composite resonator had the annulment temperature around 125 K with  $Q \sim 1-2 \times 10^5$ . The Q-value of the composite will be increased if the annulment temperature is obtained towards lower temperature that can be obtained using thinner rutile rings. In the stack f-T compensation is observed by varying the volume fraction of the dielectrics with opposite  $\tau_f$  and can be used to design frequency-stabilized MIC oscillator.

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