MONOLITHIC SAPPHIRE – RUTILE TEMPERATURE COMPENSATED WHISPERING GALLERY MODE RESONATOR OSCILLATOR AT 47K

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ABSTRACT

A 2µm thick film of Rutile (TiO_2) was deposited on a sapphire disk using a sol-gel method to realize a temperature compensated microwave resonator operating in whispering gallery mode configuration. Our resonator though not yet completely optimized shows turnover temperatures ranging from 40 to 60K depending on the mode azimutal number. For the $WGH_{7,0,0}$ mode at 9.4 GHz, the Q-factor is of the order of 1.5×10^6 at 45.7K. This resonator, stabilized near its turnover temperature, has been introduced in an oscillator loop. Preliminary measurements demonstrate frequency stability better than 2.10^{-12} for $1 < \tau < 10$ s.

1 INTRODUCTION

High frequency stability microwave oscillators are now required for radar, space and metrological applications. The most stringent requirements are found in the development of new time and frequency standards using cold atoms. These atomic clocks require microwave flywheel oscillators with frequency stability better 5.10^{-14} over integration time ranging from 0.01 s to 100 s. Moreover in the frame of the PHARAO project such a cold atoms clock will be placed on board of the international space station [1]. In that case, some other technological requirements such as low weight and low vibration sensitivity are also imposed to the microwave source. Considering only the phase noise induced by the loop amplifier, better than 5×10^{-14} frequency stability can be theoretically achieved with microwave oscillator incorporating high-Q near 77K Sapphire Whispering Gallery Mode Resonator as frequency reference. Unfortunately, the sapphire permittivity presents a large temperature sensitivity leading to a frequency temperature sensitivity of the order of -10 ppm/K at 77K. Then the oscillator frequency stability is no longer limited by the flicker noise of the electronic amplifier but by temperature fluctuations. Considering the temperature can be hardly regulated within a few µK, it is then result a frequency stability not better than 10^{-11}. Then there is a great interest in the development of temperature compensated sapphire resonators operating near the liquid nitrogen temperature. Thermal compensation can be obtained by doping the sapphire crystal with paramagnetic ions [2], by using a thermo-mechanical system [3,4] or by associating sapphire with another dielectric showing opposite temperature coefficient of permittivity as Rutile (TiO_2). The latter was already proposed and experimented in a composite structure consisting in a sapphire disk and two thin Rutile rings maintained by sapphire holders and springs [5].
In this paper, we propose another approach consisting in the deposition of a few micrometers thick Rutile film directly on the entire surface of the sapphire resonator. Such a structure is more compact and should be less sensitive to mechanical vibrations. After a brief description of the sol-gel method used to obtain the Rutile film, we present the measurement of turnover temperatures for the WGH mode family. Frequency stability measurement of the temperature compensated resonator oscillator is also given for the $WGH_{7,0,0}$ mode.

2 RESONATOR DESIGN

The sapphire disk has a diameter of 36 mm and a thickness of 10 mm. A 5 mm diameter brass screw passing through the hole made along the sapphire disk axis is used to maintain the resonator in the center of a copper cavity. Two electrical probes (antenna) parallel to the symmetry axis enable to excite the quasi-TM Whispering Gallery (WGH) mode family.

In this first attempt we use a low cost sapphire sample presenting relatively high dielectric losses at low temperatures. At 50 K, the Q-factor of the $WGH_{7,0,0}$ mode at 9.4 GHz measured before Rutile deposition was only 2.10^6 whereas Q-factors as high as 60 millions have been demonstrated with better quality crystals [6].

Compared to sapphire, rutile relative permittivity is higher and shows a negative temperature coefficient [7]. Then a small amount of rutile deposited on the sapphire resonator surface should greatly affect its temperature frequency sensitivity. Our deposition procedure consists in the polymerization of a sol composed of Titanium (IV) i-propoxide, ethanol (solvent) and acetic acid. Then, solvent and water are evaporated from the film by drying at 300°C. Such a procedure enables to deposit a 125 nm thick film on the entire sapphire surface. Then the same operation was repeated 16 times in order to reach 2 µm thickness. The resonator was then annealed at 1100°C to crystallize the titanium oxide film into the rutile or TiO_2 phase. Dominance of TiO_2 phase has been verified by X-ray measurements.

After these treatments, the sapphire disk appears white and is no longer transparent. Nevertheless, the resonant modes can be observed without difficulty and the Q-factor at ambient temperature remains unchanged and equal to 10^5.

3 TEMPERATURE COMPENSATION

The copper cavity containing the Rutile covered sapphire disk has been then fixed on the cold finger of a cryocooler. Heaters and a silicon diode temperature sensor are also fixed on the cold finger to enable temperature stabilization thanks to a PID regulator. The cavity was firstly cold down to 10K and then the temperature was raised by step. At each temperature point, resonance frequency and Q-factor of some resonant modes were recorded thanks to a network analyzer referenced to a cesium clock to ensure long term frequency stability. Fig.1 shows the evolution of the $WGH_{7,0,0}$ mode frequency as a function of temperature.

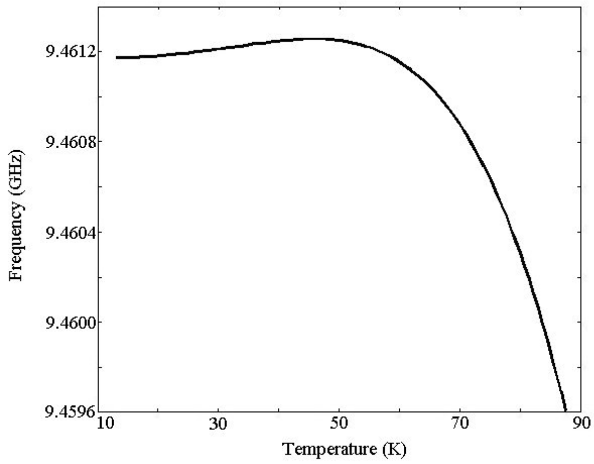

Figure 1: Temperature – frequency characteristic for the $WGH_{7,0,0}$ mode.

It clearly appears on these data a turnover temperature (T_0) of about 45.7K. A quadratic approximation in the vicinity of the peak gives:

$$\frac{\Delta f}{f} = 6\times10^{-8}(T-T_0)^2 \qquad (1)$$

A residual linear thermal coefficient due to imperfect temperature adjustment ∂T = $T\text{-}T_0$ can be derived from the slope of the curve as:

$$\frac{1}{f}\frac{\Delta f}{\Delta T} = 1.2\times10^{-7}\,\partial T \qquad (2)$$

At its turnover temperature, the unloaded Q factor is about 1.8 million and then does not differ appreciably from the Q factor measured before TiO_2 film deposition.

Observed turnover temperature for $WGH_{m,0,0}$ modes with m ranging from 4 to 9 are recorded in figure 2.

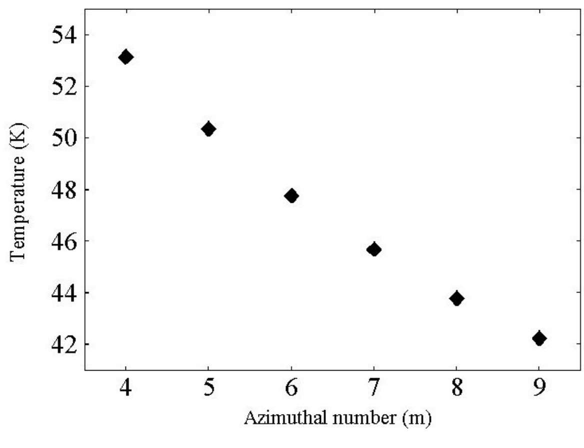

Figure 2: Turnover temperatures for the $WGH_{m,0,0}$ mode family.

As expected the turnover temperature is higher for low order modes. Indeed, lower is the value of the azimutal number m, less efficient is the electric field confinement in the sapphire and then higher is the perturbation induced by the Rutile film.

4 TEMPERATURE CONTROLLED CONPENSATED SAPPHIRE RESONATOR OSCILLATOR AT 46 K.

Figure 3 represents the schematic of the oscillator loop. It is constitued by the temperature compensated sapphire resonator, a voltage controller attenuator (VCA), a voltage controller phase shifter (VCPS), a commercial amplifier and a mechanical phase shifter.

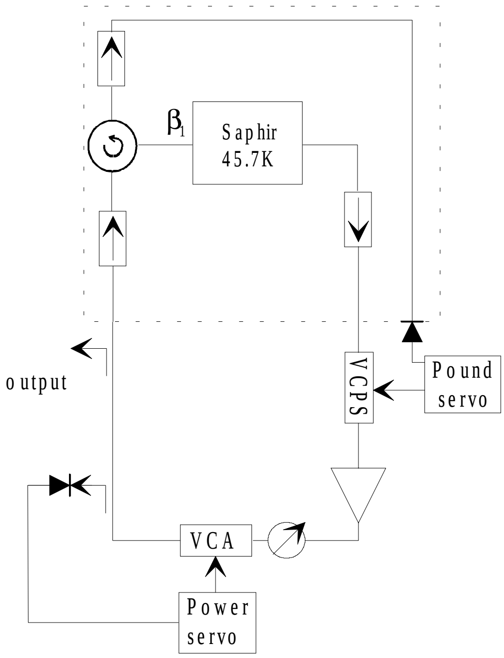

figure 3 : oscillator loop

The resonator is temperature controlled thanks to a commercial PID using a silicon diode as sensor with 4mK of resolution near 46K.
The insertion loss of the resonator is of the order of 17 dB and the obtained loaded Q factor is about 1 million.
Isolators and the circulator have been fixed just near the resonator in the cryogenic environment. The resonator couplings have been optimized to $\beta_1 = 0.87$; $\beta_2 = 0.02$ to provide a good reflected signal for the Pound frequency stabilization servo [8]. This signal is then converted to a DC signal by a tunnel diode placed at room temperature.
We have also implemented a power stabilization servo to prevent temperature fluctuations due to power fluctuation and also to limited the effect of the radiation pressure on the resonator frequency [9].

5 FIRST FREQUENCY STABILITY EVALUATION

To estimate the frequency instability of our oscillator we have compared it to a microwave synthesizer referenced to a cesium clock. This reference oscillator was mixed with the sapphire resonator oscillator creating a 6 kHz beat frequency. A frequency counter interfaced to a computer was used to calculate the frequency deviation of the 6 kHz beat note.
On the figure 4 we can see the evolution of the beat note frequency versus time.

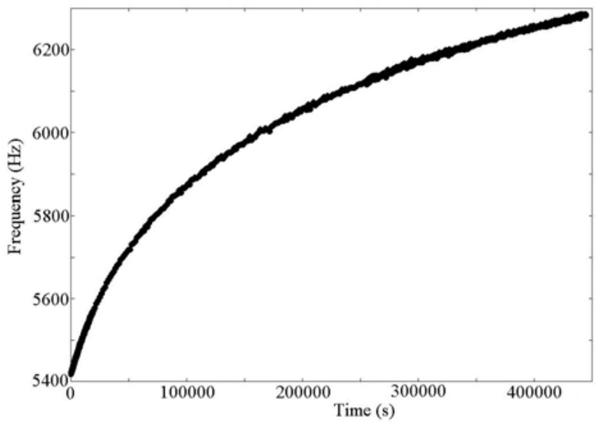

figure 4 : evolution of the beat note frequency versus time.

It clearly appears an ageing of the resonator frequency. Such a resonator ageing could be due to residual stresses induce by the mechanical sapphire fixation inside the copper cavity, or to the thermal stabilization of the resonator itself. Nevertheless after 5 days of operation the frequency drift is of the order of $6.5.10^{-9}$ per day, and it still decreases when time increases.
The figure 5 shows the standard deviation $\sigma_y(\tau)$ of the relative frequency fluctuations evaluated for 1s of integration times after 10 days of continuous operation of the oscillator.
The obtained frequency stability for $1 \leq \tau \leq 10$ s (i.e. 2.10^{-12}) is consistent with the performances of the thermal regulator. Indeed, if we consider the equation 2 with 4mK of temperature resolution measurement, we obtained a relative frequency stability $\sigma_y(\tau) = 1.9.10^{-12}$. After 10 seconds of integration times we can see the effect of the resonator frequency ageing. The obtained result at 1000 seconds leads to a frequency drift of the order of 2.10^{-9} per day.

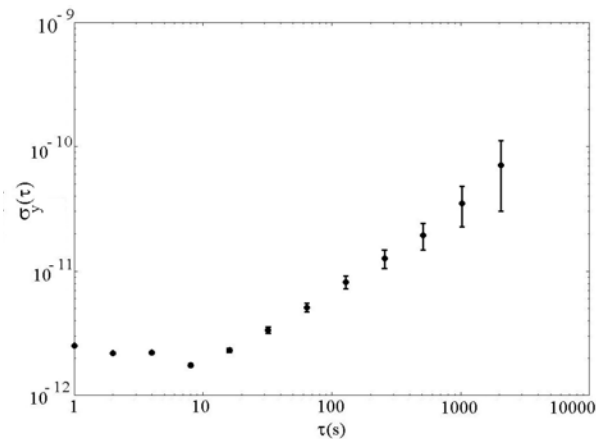

figure 5 : relative frequency fluctuations of the temperature compensated sapphire oscillator at 9.4 GHz

6 CONCLUSION

We have developed a new temperature compensated sapphire resonator achieving turnover temperatures of the order of 50K. For the $WGH_{7,0,0}$ mode operating at 9.4 GHz the turnover temperature is 45.7K and the unloaded Q is 1.8 million. The main advantages of this new compensated structure are its compactness, its intrinsic immunity to mechanical vibrations and its low cost. With this first prototype we have obtained a relative frequency stability of the order of 2.10^{-12} for integration times less than 10 seconds. Nevertheless at long integration times we observed a frequency ageing that we attribute to the relaxation of mechanical stresses in the sapphire crystal or to the thermal stabilization of the resonator.

Further experiments are needed to reach higher turnover temperatures by increasing the TiO_2 film thickness. Moreover, the influence of the TiO_2 film on Q-factor is not yet well determined and we plan to deposit TiO_2 film on a higher quality sapphire crystal.

8 REFERENCES

[1] A.G. Mann, G. Santarelli, S. Chang, A.N. Luiten, Ph. Laurent, C. Salomon, D.G. Blair and A. Clairon. "A high stability atomic fountain clock using a cryogenic sapphire interrogation oscillator".
Proc. of the IEEE IFCS, Pasadena, CA, USA, 1998, pp 13-17

[2] J.G. Hartnett, M.E. Tobar, A.G. Mann, J. Krupka and E.N. Ivanov. "Temperature dependence of Ti^{3+} doped sapphire whispering gallery mode resonator"
Electronics Letters, 1998, **34** (2), pp 195-196.

[3] G.J. Dick, D.G Santiago, R.T Wang "Temperature - compensated sapphire resonator for ultra-stable oscillator capability at temperatures above 77K"
IEEE Trans. UFFC, 1995, **42** (5), pp 812-819.

[4] V.Giordano, Y.Kersalé, O.DiMonaco, M.Chaubet "'Progress report on the development of a microwave spectral references at the LPMO"
European Physics Journal, Applied Physics, 1999, **8**, pp. 269-274.

[5] M.E. Tobar, J.G Hartnett, E.N. Ivanov, D. Cros, P. Blondy and P. Guillon. "Cryogenically cooled sapphire-rutile dielectric resonators for ultrahigh frequency stable oscillators for terrestrial and space applications".
IEEE Transactions on Microwave Theory and Techniques, 2000, **48** (7), pp. 1265-1269.

[6] J. Krupka, K. Derzakowski, M.E. Tobar, J.G. Hartnett and R.G. Geyer. "Complex permittivity of some ultralow loss dielectric crystals at cryogenic temperatures".
Meas. Sci. Technol., 1999, **10** (4), pp. 387-392.

[7] M.E. Tobar, J. Krupka, E.N. Ivanov and R.A. Woode. "Anisotropic complex permittivity measurements of mono-crystalline rutile between 10-300K."
J. of Appl. Phys., 1999, **83** (3), pp. 1604-1609.

[8] A.N Luiten, A.G.Mann, N.J.McDonald and D.G Blair. 'Latest results of the U.W.A cryogenic sapphire oscillator'. Proc. 1995 IEEE Int. Frequency Control Symposium, pp 433-437.

[9] S. Chang, A.G.Mann, A.N Luiten, and D.G Blair. "Measurements of radiation pressure effect in cryogenic sapphire dielectric resonators". Physical Review Letters, 1997, 79 (11), pp 2141 – 2144.