Temperature Coefficients of Quartz at Cryogenic Temperature

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***Abstract*—The temperature coefficients of the elastic coefficients of quartz within [4 K, 15 K], the operating range of conventional cryocoolers, are predicted from experimental data. The raw data consist of a set of frequency-temperature curves recorded from bulk acoustic wave resonators made in different plates cut according to various angles relative to the crystallographic axes. Raw data are then processed as described in the paper to provide the 1st, 2nd, and 3rd order temperature coefficients of the seven elastic coefficients of quartz crystal.**

Keywords—quartz; elastic coefficients; temperature coefficients; liquid-helium temperature; low mechanical loss; high quality factor.

I. Introduction

Usually temperature sensitivity cannot be neglected when designing a quartz resonator-based device. Even at temperatures lower than 6 K, where the material properties are less sensitive to temperature than at room temperature, the remaining sensitivity has to be considered. The resonant frequency of a quartz crystal resonator is not an exception to this rule: the remaining fractional frequency change versus temperature is typically a few 10^{-9} K^{-1} close to 4 K [1-2]. This is still too much to realize a stable frequency source even with a temperature control exhibiting a typical gain of 1000: definitely, such an application would need a temperature compensated cut. It should be reminded that quartz resonators look very attractive around 4 K because of their very high quality factor that can be greater than one billion [3]. Conversely, quartz resonators could also be used as temperature sensors provided they are made in an appropriate cut, i.e. a very temperature-sensitive one. In both cases, to design a frequency reference or a temperature sensor the knowledge of the temperature coefficients of the elastic constants of the material is mandatory, the resonance frequency depending on these elastic constants. A set of coefficient values already exists for the quartz crystal since 1975 [4] but it covers the very wide range [4K, 400 K], and consequently becomes irrelevant inside the shorter operating range of conventional cryorefrigerators. These are the main reasons why we carried out a campaign of measurements to determine the 1st, 2nd and 3rd order temperature coefficients of the elastic constants of quartz within [4 K, 15 K].

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II. Method of Calculation

The thermal coefficients can be determine from various experimental principles. It can be from resonant ultrasound spectroscopy, phase velocity measurements by pulse echo overlap methods, laser ultrasonic techniques or combined resonance techniques. Nevertheless, their implementation at cryogenic temperatures arises technical difficulties. The selected alternative consists in probing the temperature sensitivity of resonance frequencies of dedicated quartz devices.

A. Theoretical principle

Basically, a flat plate exhibits bulk acoustic waves (BAW) resonance frequencies derived from $f_n = \frac{n}{2h}\sqrt{\frac{C_e}{\rho}}$ where n is the overtone order, $2h$ the plate thickness, ρ its density, and C_e an elastic coefficient that looks like a combination of the material elastic constants C_{ijkl} depending on the cut orientation with respect to the crystallographic axis in case of quartz because of its anisotropy. All these parameters are temperature dependent yielding to a temperature-dependent frequency. Usually, the frequency-temperature characteristic can be fitted effectively by a 3rd order polynomial approximation, and this operation can be applied to each parameter. Once experimental frequency-temperature records processed this way, the three corresponding temperature coefficients of the elastic coefficient(s) can be extracted accordingly. The calculation is just somewhat complicated by the fact that there are seven coefficients C_{ijkl} for the quartz crystal. A set of combinations should therefore be considered involving a set of resonator cuts to be tested.

B. Relationships and assumptions

Actually, the tested devices are not flat plates but plano-convex disks, a shape commonly used to trap efficiently the acoustic energy. This is one way to preserve the resonator against disturbances from its environment (i.e. parasitic stresses and then frequency changes), and consequently a kind of guarantee to qualify the material regardless of the device structure. However, the modelling is complicated and the resulting frequency is as follows, according to the Stevens-Tiersten model [5], one of the most accurate theories:

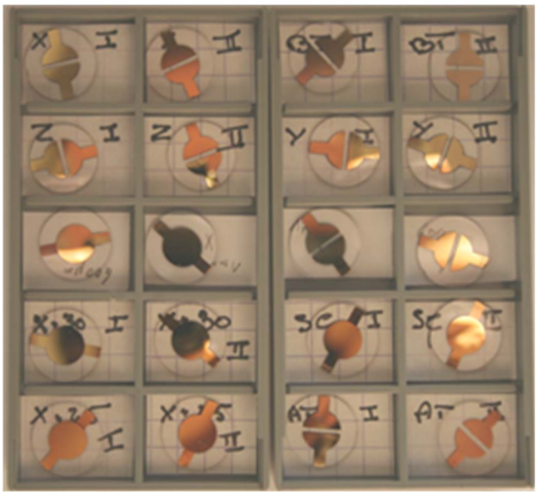

Fig. 1. Set of plano-convex resonators tested for determining the temperature coefficients of the elastic constants of quartz at 4 K.

$$f_{nmp}^2 = f_n^2\left[1+\frac{1}{n\pi}\sqrt{\frac{2h}{R_0}}\left(\sqrt{\frac{M_n}{C_e}}(2m+1)+\sqrt{\frac{P_n}{C_e}}(2p+1)\right)\right] \quad (1)$$

Where f_n are the eigenfrequencies of the corresponding infinite flat plate, R_0 the curvature radius of the convex face, m, p the number of modal lines in the resonator plane, M_n and P_n the dispersion constants.

Nevertheless, several assumptions can be made by considering some orders of magnitude [6], and calculation proceeds according to the described process.

III. Experiment

A. Experimental set-up

Resonators have been tested in a conventional pulse tube cryorefrigerator SRP082B. The operating temperature is controlled at ±3 mK within [4 K, 15 K] by a Lakeshore 335 probing a cernox sensor and actuating a heater embedded inside the copper block where the resonator under test is set.

B. Devices under test

As mentioned above, a set of dedicated BAW energy-trapped resonators with gold electrodes, as shown in Fig. 1, has been made and tested.

Table 1 shows, from left to right, from which cut and modes each experimental frequency-temperature characteristic is taken from, the corresponding frequency coefficients of the polynomial fit, and the targeted elastic coefficient(s).

TABLE I. Targeted elastic coefficients

Cut	From experimental data				Target
	Mode	***$T^{(1)}f$ In 10^{-7}***	***$T^{(2)}f$ In 10^{-8}***	***$T^{(3)}f$ In 10^{-9}***	
AT	C_{300}, C_{500}	0.769	0.748	0.105	C_{14}
SC	A_{300}, A_{500}	-1.178	-2.189	-2.747	C_{13}, C_{33}
SC	B_{300}, B_{302} B_{320}, B_{500}	-0.879	-1.672	-1.832	C_{13}, C_{33}
X	A_{100}, A_{1300}, A_{1500}	-0.143	-0.451	-0.581	C_{11}, (C_{12}[a])
Y	C_{300}, C_{500}	1.339	1.869	1.289	C_{66}
Z	C_{300}, C_{500}	-1.465	-2.662	-3.571	C_{44}

a. Calculated from C_{11} and C_{66}

IV. Results

Table II shows the resulting 1st, 2nd and 3rd order temperature coefficients of the *Cij*'s, for quartz, with T_{ref} = 8 K.

TABLE II. Resulting coefficients

C_{ij}	$T^{(1)}C_{ijkl}$ in 10^{-7}	$T^{(2)}C_{ijkl}$ in 10^{-8}	$T^{(3)}C_{ijkl}$ in 10^{-9}
C_{11}	-0.235	1.28	-3.25
C_{12}[b]	-6.39	-10.56	-4.22
C_{13}	-7.18	-7.42	-0.877
C_{14}	-1.35	-1.31	-7.93
C_{33}	-4.62	-5.65	-7.62
C_{44}	-3.17	-4.37	-11.6
C_{66}	3.08	5.92	0.486

b. Calculated from C_{11} and C_{66}

V. Conclusion

Resulting data will be used to identify temperature compensated cuts in the very useful range [4 K, 15 K], where quartz resonators exhibit Q-factors of about one billion.

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