INVESTIGATIONS OF THERMO-SENSITIVE QUARTZ RESONATORS NLC-CUT AT CRYOGENIC TEMPERATURES

L. Spassov[1], R. Velcheva[1], Ts. Yordanov[1], L.Vergov[1], B. Dulmet[2], R. Bourquin[2]

[1] *ISSP-BAS, 72 Tzarigradsko Chaussee Blvd., 1784 Sofia, Bulgaria*
E-mail : lspassov@issp.bas.bg, Tel : (359-2) 77 25 10, Fax : (359-2) 975 36 32
[2] *LCEP/ENSMM, 26 Chemin de l'Epitaphe , 25000 Besançon, France*

ABSTRACT

The paper presents obtained results from the testing of motional parameters, Q-value, amplitude-frequency characteristics of a new miniaturized design of TSQR with *yxl*/-31°30' cut, and their dependency with respect to temperature. This design operates at 29.3 MHz on fundamental frequency of mode. The influence of mass loading on motional parameters and spectrum characteristics is detailed. Special attention is given to the disturbances of the frequency-temperature characteristic from room temperature down to the *He* temperatures.

1. INTRODUCTION

Thermo-sensitive quartz resonators (TSQRs) recently became more attractive as high sensitive temperature sensors for scientific and industrial applications, due to their advantages, for instance good reproducibility, long term stability and immunity to electromagnetic perturbations.

The most important advantage of quartz is its anisotropy, allowing the design of piezoelectric resonance devices, which possess either negligible or strong temperature-frequency dependence. The temperature-frequency characteristic (TFC) of each resonator can be represented by polynomial of *n-th* order and has the form of a straight line (LC cut), quadratic (BT, CT cuts) or cubic parabola (AT, SC cuts) [Ref.1]. On this base a family of different thermostable quartz resonators for telecommunication applications has been developed [Ref.2,3]. Since 1962 there has been a systematic approach to promoting the temperature sensitivity of the frequency from an unwonted effect in the frequency control application domain to an attractive temperature sensor concept [Ref.4,5]. Researchers at Hewlett-Packard [Ref.6] developed the first thermosensitive resonators with a linear TFC in the interval from minus 80°C to 230°C. These resonators were sold as temperature sensors over a period of more than 25 years [Ref.7]. The main disadvantage of this sensor is the significantly higher price of doubly rotated compared to singly rotated Y-cuts.

In 1984 Ziegler [Ref.8] introduced the HT-cut quartz temperature sensor based on a slightly singly rotated Y-cut (θ=-4°) with a frequency versus temperature sensitivity of 90 ppm/°C. The nonlinearly is only a few percent over the full temperature range.

In 1987 Spassov et al. [Ref.9] found a singly rotated quartz Y-cut (*yxl*/-31°30'), named as NLC (new linear coefficient) cut with a small linearity deviation of ± 0,2°C in temperature range from minus 30 to 130°C. The sensitivity is 30 ppm/°C corresponding to 1000 Hz/°C for 29,3 MHz fundamental mode crystal plate [Ref.10].

The present paper deals with further investigations of this type of TSQRs at cryogenic temperatures.

2. EXPERIMENT

2.1 Resonator theory and design

For quartz resonators with a rotated Y-cut and thickness-share mode of vibration, the resonance

$$f = \frac{m}{2h}\sqrt{\frac{c'_{66}}{\rho}}$$

frequency is determined by
where m is number of overtone, h-thickness of the plate, ρ - density of quartz, and

$$C'_{66} = C_{66}\cos^2 • + C_{44}\sin^2 • + C_{14}\sin 2•$$

C_{ij} – are elastic constants and θ the angle between the *Z*-axis and the main surface of the piezoelement.

$$f(t) = f_0\left[1 + \sum_{n=1}^{3} T_f^{(n)}\left(t - t_0\right)^n\right]$$

Temperature dependence of frequency extremely depends on orientation of piezoelement towards the crystallographic axis of quartz. It can be expressed by polynomial in 3rd order,

Temperature coefficients of frequency $T_f^{(n)}$

$$T_f^{(n)} = \frac{1}{n!}\frac{\partial^n f}{f_0 \partial t^n}\Big|_{t=t_0}$$

where n=1,2,3 is sum of the temperature coefficients of frequency of first, second and third order

$$T_f^{(n)} = T_f^{(1)} + T_f^{(2)} + T_f^{(3)}$$

Principal role in temperature dependence of frequency play the temperature coefficients of the elastic constants C_{ij}.

Knowing the temperature coefficients an orientation has been determined to ensure that temperature coefficient of the first order, $T_f^{(1)}$ has positive value, while the temperature coefficients of second and third order, $T_f^{(2)}$ and $T_f^{(3)}$ respectively have values with different signs, which leads to a decrease of nonlinearity.

Miniature resonator with *yxl*/-31°30' cut quartz plate on the fundamental frequency 29,3 MHz have been realized. The diameter of the plate is 5 *mm* and its thickness is 0.083 *mm*. After it has been polished silver electrodes with 2.9 *mm* diameter have been deposited on the main surfaces of the plate. The thickness of electrodes varies from 500 Å to 1200 Å for each side. Piezoelement is enclosed in a standard HC – 45U crystal enclosure and filled with dry nitrogen or helium.

2.2 Influence of mass loading on motional parameters and spectrum characteristics.

Five groups with different thickness of electrodes have been investigated. By vacuum evaporation at $4{,}6.10^{-6}$ bars *Ag* electrodes with thickness from 500Å to 1300 Å for each side have been deposited.

Typical values of the series resistance of the TSQRs are within 8 Ω to 25 Ω. It is observed some light increase of R_s with increasing the thickness of electrodes due to mass loading of the electrodes (fig 1).

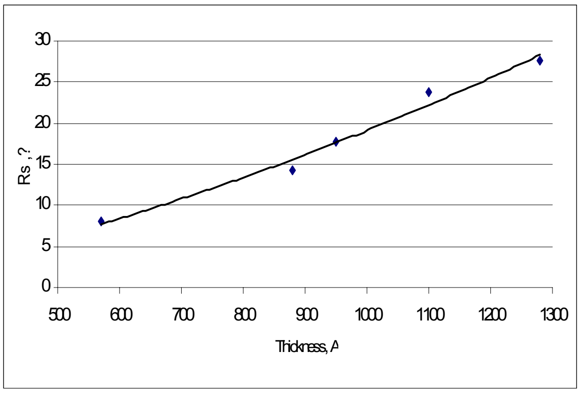

Fig.1. Series resistance shift vs. electrode thickness

Thickness of electrodes visible influence on the spectrum characteristics of the TSQRs. Resonators with thickness of electrodes in the range from 500 Å to 900 Å, which correspond of decreasing of frequency from 5.10^{-3} to 9.10^{-3} have comparatively pure spectrum characteristic. There are some unwanted modes which are located at distance more than 50 kHz and 20 dB attenuation (fig 2a). At thickness 950 Å to 1050 Å, which correspond to decreasing of frequency with $9{,}23.10^{-3}$ to $9{,}77.10^{-3}$, a new peak at 25 kHz higher towards main resonance comes into view (fig. 2b). It disappears at thickness over 1100 Å (fig.2c) i.e.1.10^{-2} mass loading.

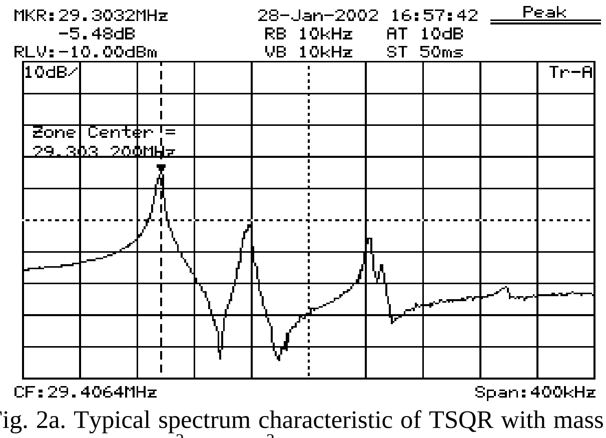

Fig. 2a. Typical spectrum characteristic of TSQR with mass loading from 5.10^{-3} to 9.10^{-3}

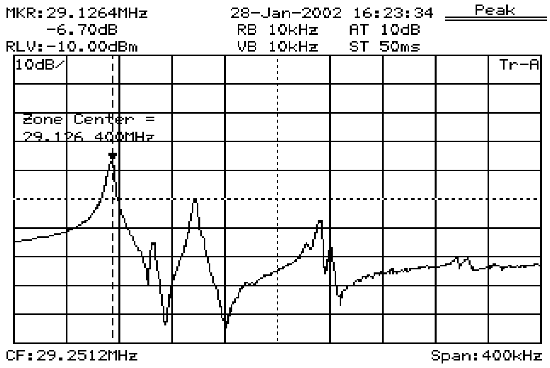

Fig. 2b Typical spectrum characteristic of TSQR with mass loading from $9{,}23.10^{-3}$ to $9{,}77.10^{-3}$

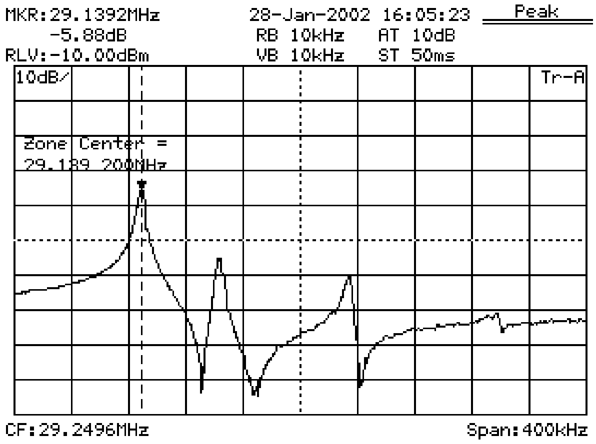

Fig. 2c. Typical spectrum characteristic of TSQR with mass loading over 1.10^{-3}

2.3. Influence of temperature on spectrum characteristics

A typical spectrum characteristic of TSQRs with 570 Å electrode's thickness is shown at figure 3. Unwanted resonances are depressed more than 25 dB. Spectrum characteristic retains with out significant changing within temperature interval from minus 40^o C to 120^o C. There is some redistribution of acoustic energy between second and third peaks at minus 40^o C and 120^o C (fig. 3a,c) compare to spectrum at room temperature (fig 3b).

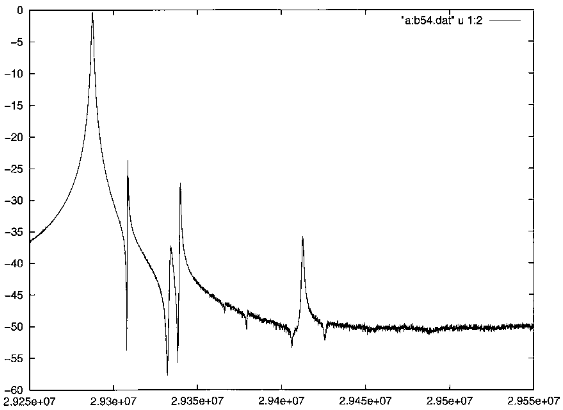

Fig. 3a. Spectrum characteristic of TSQR at room temperature

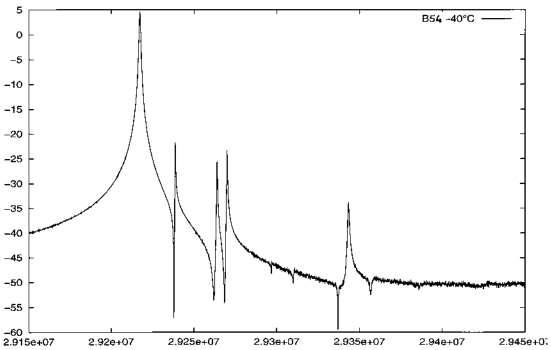

Fig. 3b. Spectrum characteristic of TSQR at minus 40°C

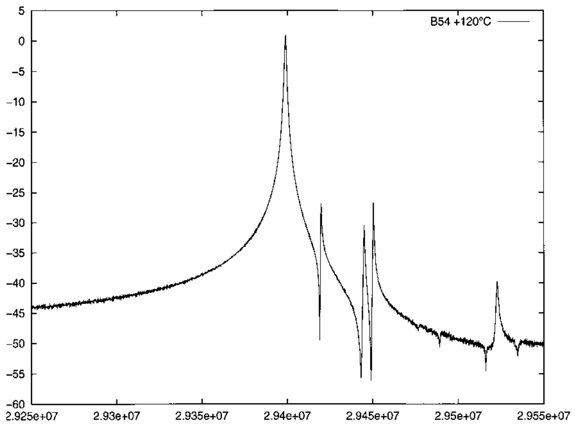

Fig.3c. Spectrum characteristic of TSQR at 120°C

3. INVESTIGATIONS OF TSQR AT CRYOGENIC TEMPERATURES

3.1. Experimental set-up

The TFC at cryogenic temperatures was measured using a special helium vessel equipped with stick where the thermosensitive quartz resonator and carbon thermoresistor (TVO) for temperature measurement were attached. The block diagram of the equipment for measuring the TFC is shown on Figure 4.

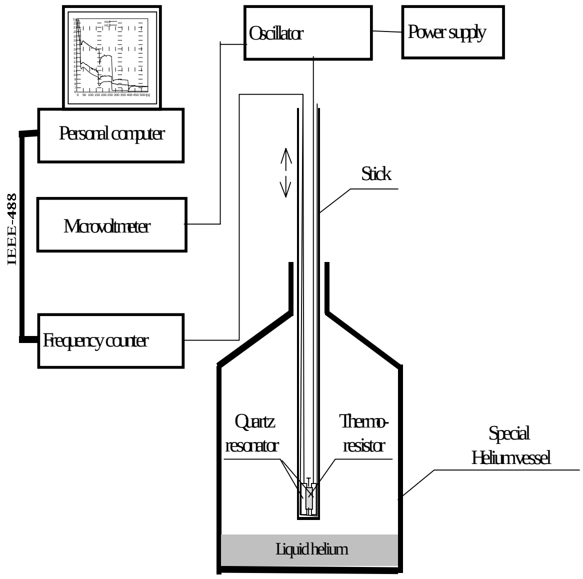

Fig.4. Experimental set up of measurement

A high-frequency matching cable to the measuring oscillator connected the quartz thermosensitive resonator. The frequency was measured by a Ч3-63 frequency counter with uncertainty about of $\pm$ 1,10^{-9}. By vertical movement of the stick, the temperature of TSQRs and TVO-sensor was changed from room temperature to the temperature of liquid Helium - 4,2K. Changing the temperature with 5K step at temperature range from 4,2K to 40K and with 10 K step at temperature range from 40K to 300K, we measured the TFCs of the TSQRs. The temperature and frequency data are read every second by the personal computer (PC), where they are displayed as graphics and saved in files.

3.2. Temperature-frequency characteristic

A polynomial of third order that describes temperature frequency characteristic with enough accuracy at temperature range from 40K to 300K.(Fig.5).

The coefficients of polynomial $T_f^{(1)}$=-5,9.10^{-3} ;

$$f(t) = -0.0059t^3 + 4{,}8867t^2 - 167{,}99t + 28844900$$

$T_f^{(2)}$=4.8867 ; $T_f^{(3)}$=167.99 ensure linear character of the TFC over 130K. In the temperature interval from 4,2 K to 130 K the temperature sensitivity of the resonators is monotonous increased function. In this interval the sensitivity increases with the increase of the temperature starting with a value of about 2 Hz/K at 4,2K to a value of 900 Hz/K at 130K. Over 130 K the TFC has linear character and the sensitivity is about 1000 Hz/K

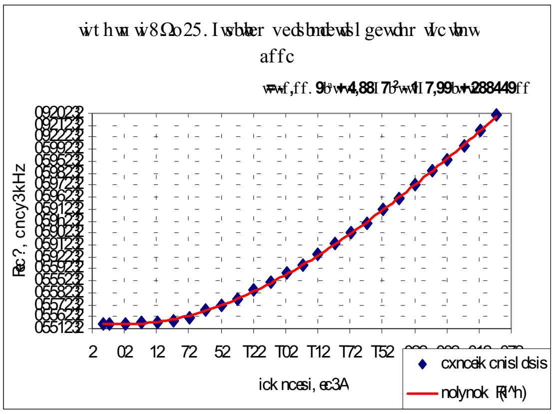

Fig.5. Temperature-frequency characteristic of TSQR at temperature range from 4,2K to 300K

Temperature-frequency characteristic at the temperature range below 40K to the temperature of liquid helium (4,2K) can be described better by follow equation:

$$f(t) = -0.018t^3 + 2.69t^2 - 47.3t + 28844200$$

as it is shown on Figure 6.

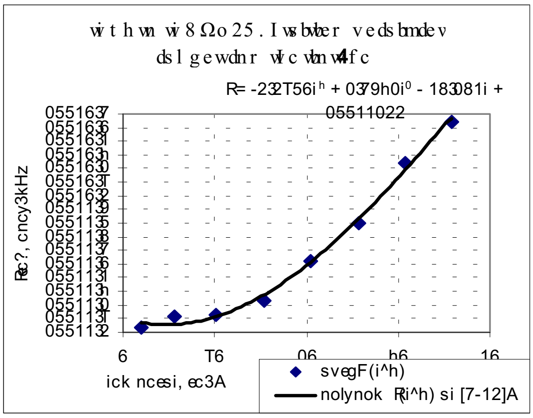

Fig. 6. Temperature-frequency characteristic of TSQR in temperature range from 4,2K to 40K

Frequency stability of TSQRs at the temperature of liquid helium was measured within time interval of 140seconds. Stability of $3,5.10^{-9}$ was registrated. (Fig.7), which is very closed to the frequency stability of the frequency counter.

One of the advantages of TSQR as temperature sensor is its high temperature sensitivity. It is different for different temperature range:

2 Hz/K at 4,2 K (temperature of liquid helium), 60 Hz/K at 20 K, 900 Hz/K at 130 K, 1000 Hz/K at 300 K,

This allows the measurement of the temperature fluctuations as small as:

at 4,2K, ΔTmin = 50mK,
at 20 K, ΔTmin = 8 mK,
at 130 K, ΔTmin = 0,12 mK,
at 300 K, ΔTmin = 0.085 mK.

This microdegree sensitivity allows measuring frequency deviation of a unit in 10^9. However, for sensitivity higher than 10^{-4} K is limited by measuring equipment.

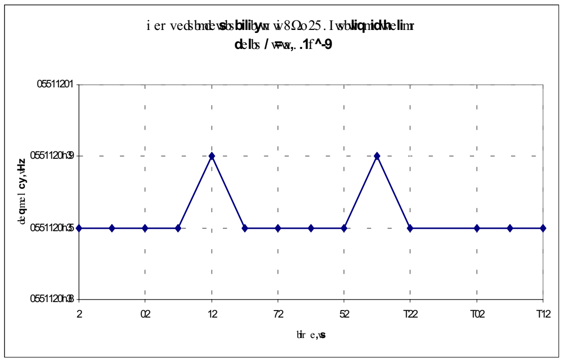

Fig. 7. Frequency stability of TSQR at He-temperature

3.3. Disturbances of TFC at cryogenic temperatures

In particular temperature intervals [50-60]K, [90-110]K and [120-135]K disturbances of the behavior of some of the resonators are observed. On Figures 8(a, b) is presented a kind of this disturbances in temperature interval [50-60]K.

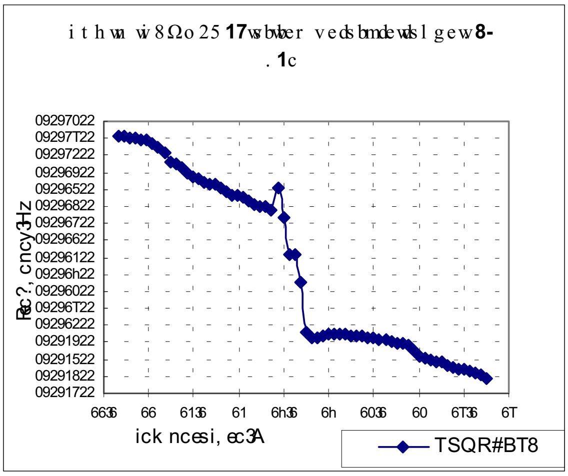

Fig.8a. Disturbance of TFC at decreasing of temperature

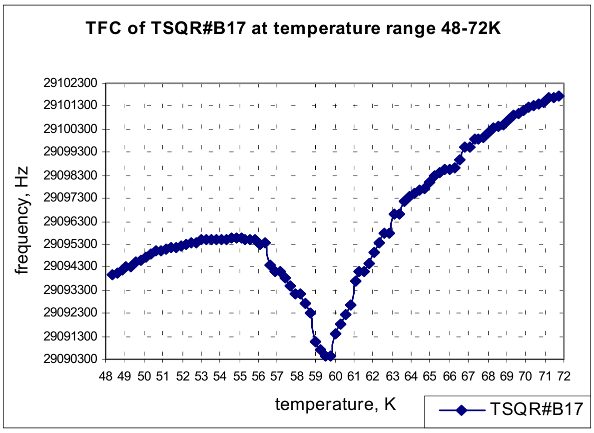

Fig.8b. Disturbance of TFC at increasing of temperature

One can remark suddenly jumping of frequency when the temperature decrease (Fig.8a) and when the temperature increase(Fig.8b). The most possible reason of the instabilities is the stimulation and coupling of other mode of vibration at some temperatures. This phenomenon is known as "activity dip".

4. CONCLUSION

New design of miniaturized TSQR yxl/-31°30' cut has been investigated at room and cryogenic temperatures. Influence of mass loading on motional resistance and spectrum characteristics are studied. It is shown that with increasing of the mass loading motional resistance slightly increase. At thickness 950 Å to 1050 Å, which correspond to decreasing of frequency with $9,23.10^{-3}$ to $9,77.10^{-3}$ a new peak in spectrum characteristic at 25kHz higher towards main resonance come into view. It disappear at thickness over 1.10^{-3} mass loading. Spectrum characteristic retains with out significant changing within temperature interval from minus 40°C to 120°C.

It is found a polynomial of a third order that describes temperature-frequency characteristic with enough accuracy at temperature range from 40K to 300K. In the temperature interval from 4,2K to 130K the TFC of the resonators are monotonous increased function. Frequency stability ($3,5.10^{-9}$) and high temperature sensitivity allows measurements of temperature fluctuation as small as from 50mK at 4,2K to 0,01mK at 300K.

In particular cryogenic intervals some disturbances of the TFC of some resonators are observed. The most possible reason of the instabilities is the stimulation of coupling of the modes at some temperature, well known as "activity dip" phenomenon.

5. ACKNOWLEDGEMENTS

This study is partly supported by CNRS (Direction Relation Internationales) and BAS (Bulgarian Academy of Sciences) under bilateral cooperation number 8686. Authors wish to thank both organizations for this precious help.

Special thanks authors wish to express to Mrs. Zdravka Raicheva, Mr. Stefan Staikov, and Mr. Julian Lazarov for preparating of samples and Mrs. Svetla Shumkova for her assistance in collecting and processing of results.

6. REFERENCES

1. R.Bechman, A.D.Balato and T.J.Lukaszek, Higher Order Temperature Coefficients of the Elastic Stiffness and Compliance of Alfa-quartz, Proc. of IRE, 50(1962),1812-1822
2. A.G.Smagin and M.Y.Yaroslavski, Piezoelectrichestvo quartza i quartzevie resonatori, Energia, Moscow,1970, p.488, (in Russian)
3. P.G.Pozdniakov, Handbook of Quartz Resonators, Sviaz, Moscow, 1978, p.49-54, (in Russian)
4. W.H.Wide and L.J.Slutsky, Quartz Crystal Thermometer, Rev.Sci.Instr., 33(1962), 212-213
5. W.L.Smith and W.J.Spencer, Quartz Crystal Thermometer for Measuring Temperature Deviations in 10^{-3} to 10^{-6}°C range, Rev.Sci.Instr., vol.34, 268-270, 1963
6. D.L.Hammoud and A.Benjaminson, Linear Quartz Thermometer, Instrum. Control Syst., vol.38, 115-119, 1962
7. Quartz Thermometer 2804A, Hewlet Packard catalog
8. H.Ziegler, A Low-cost Digital Sensor System, Sensors and Actuators, vol.5, 169-178, 1984
9. M.Borissov, L.Spassov et al. Quartz Resonator, Bulgarian Patent N 81377, 1987
10. L.Spassov, E.Yossiffov, V.Georgiev, L.Vergov, A Rotated Y-cut Quartz Resonator with a Linear Temperature-Frequency Characteristic, Sensors and Actuators, A58(1997), 185-189