Investigations on 10 MHz LGS and LGT crystal resonators

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***Abstract*— Materials in the LGT family are promising for designing bulk acoustic wave resonators with high quality factor. In our laboratory, we have manufactured a lot of plano-convex 10 MHz 5th overtone Y-cut resonators using LGS (langasite $La_3Ga_5SiO_{14}$) and LGT (langatate $La_3Ga_{5.5}Ta_{0.5}O_{14}$) crystals.**

Our initial aim was to do noise measurements on these home-made resonators but problems occurred during manufacturing. It was the opportunity for further investigations. Indeed, we observed that the quality factor depends strongly on the energy trapping, the polishing method and the materials quality from one supplier to another. As for the quartz crystal, we have found that the material quality can be qualified by IR spectrometry whose resulting spectra exhibit absorption peaks more or less deep, linked to defects. These predominant criteria are not surprising but although they are nowadays quite well-defined in the case of quartz crystal resonators, they have to be defined again in the case of these LGS and LGT crystals. Then, a satisfying machining and polishing method has been first applied to elaborate high Q resonators.

A comparison between different grades of LGS and LGT materials is established. In addition, LGT resonators are characterized by their motional parameters and frequency-temperature curves. Nevertheless, one of the main results is that the measured Q-f product is not the expected one. We present results of Q-factor versus radius of curvature and their comparison with the theoretical approach. It appears that an optimization should be performed.

Right now the best resonator that we have made has got a Q-f product of 1.4 10^{13} on its 5th overtone (1.7 10^{13} on its 9th overtone). This result is slightly higher than the similar parameter obtained on a SC-cut quartz crystal resonator working at the same frequency.

I. Introduction

Today, langasite and langatate ($La_3Ga_5SiO_{14}$ LGS and LGT $La_3Ga_{5.5}Nb_{0.5}O_{14}$) piezoelectric materials are less well known than quartz material. They have very interesting and attractive properties [1, 2, 3] like: high coupling coefficient, low acoustic losses, no Curie transformation point... These properties are required to make high performance bulk acoustic wave resonators (BAW). But before this step, we need to develop a good machining process and to evaluate radius of curvature for an optimal energy trapping. At these conditions, it could be possible to make high Q-factor langatate crystal resonators.

Our aim is to realize Y-cut LGT crystal resonator with plano-convex configuration, bridges linking active part to the dormant one and electrodeless (so called BVA structure). But during fabrication, problems occurred due particularly to a worse quality of material than awaited. So, we decide to do investigations to know where and why problems occur.

II. Machining process of LGS and LGT

Final goal is to develop a satisfying manufacturing method to manufacture high Q-factor resonator. With an empirical method, we have evaluated parameters of relevant machining. For lapping and polishing [4], we tried different types of slurry. For the lapping, different abrasive powders were used: silicon carbide (SiC), aluminum oxide and synthetic diamond. These abrasive powders are melt with unionized water and used on a brass form. We noticed that, with grains size diameter lower than 5µm on brass lappers, surface of LGS and LGT can present a defect similar to a work hardening. On glass lappers the problem seems less important but still exists. Cleaning phases between each machining step are done with unionized water, Decon 90, alcohol and/or acetone to avoid any chemical attack.

The last step of lapping is made with grains size higher than 5µm to avoid the phenomena of work hardening. Polishing does not pose problems. For SiC and aluminum oxide it is done on a brass support covered with felt and for diamond with brass tools covered with silk.

For the manufacture of our resonators, we chose to use a machining process entirely with diamond powder carried by unionized water. Final roughness obtained is very satisfying

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(Ra ~ 1nm, Ra meaning arithmetic mean of the roughness obtained by mechanical measurement along a straight line). List of grains size is classified in the following table.

TABLE I. LIST OF GRAINS SIZES SUCCESSIVELY USED AND THICKNESS REMOVED ON BOTH FACES

Grains nominal diameter	***Removed thickness (both faces)***
9µm	100 µm
6µm	40 µm
3µm	20 µm
1µm	12 µm
1/2 µm	4 µm
1/4 µm	2 µm
1/8 µm	1µm

III. QUALITY AND VISIBLE ASPECT OF MATERIAL

We own langasite and langatate "boules" from different suppliers. They are different by their:

- Aspects [5] (colors and visible defects in volume)
- Infra-Red spectrum
- quality factor of the manufactured resonators

A. Visible aspect

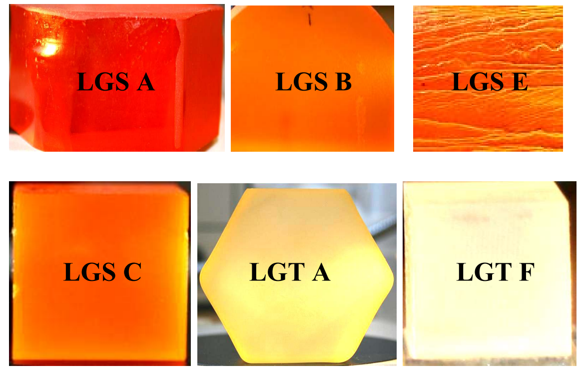
The first thing that we notice is the strong coloring of the 5 blocks of LGS and the 2 of LGT. Two LGS of two different suppliers may have different color aspects (Table II and Caption 1). In the block of the C source, an intern colored straight and thick line is visible, aligned along the crystallographic X-axis (Caption 2).

TABLE II. SOURCE AND COLOR OF THE DIFFERENT BLOCKS

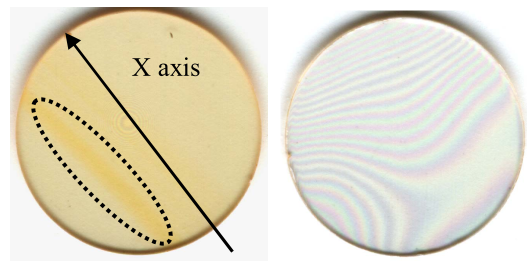
Supplier	***Color***
LGS	
A	Orange/red
B	Orange
C	Orange
D	Pale red
E	Orange
LGT	
A	Green/yellow
F	Colorless

In the A block of LGS, we observed striations aligned along the X-axis. Moreover, the color of one LGT sample (from A) has changed from green to red in a few months after cutting, as a demonstration of the instability of the material due probably to internal stresses trapped during pulling.

We noticed that the less colored material with the smallest volume defects, present the best quality factor of the Y-cut 5th overtone 10 MHz resonators.



Caption 1. Color of the different blocks



Caption 2. Color and defect of two different LGS Y-cut resonators (right: C supplier and left: D supplier)

B. IR spectrometry: LGS and LGT crystals comparison

To analyze the quality of each crystal sending by our different suppliers, we have used the same tools allowing the characterization of the quartz crystal. The standards, applied to quartz quality study, define particularly an intrinsic coefficient alpha obtained on the IR spectrum of a thick (~ 5 mm) Y-cut sample with polished faces.

So, we present here the IR spectra of the 5 different LGS and the 2 LGT samples obtained at N_2 liquid temperature. Almost of these spectra exhibit narrow and more or less deep bands at 3413 and at about 5420 cm^{-1}. We observe that the second one disappears almost on the best quality crystals (D for LGS and F for LGT). To obtain more details on the process and few explanations on the existence of these absorption bands, we advise with the reader to see [6] and [7].

To give an idea of the comparative qualities of these different crystals of LGS and LGT, we present in the table below the α-value translating the depth of the 3413 cm^{-1} narrow band. We assume here that the absorption level of the lattice can be given, as for quartz, at 3800 cm^{-1}.

TABLE III. α-VALUES OF DIFFERENT LGS AND LGT SAMPLES, GIVEN AT 3413 CM^{-1} (- : NOT MEASURED)

	LGS samples					LGT samples	
	A	B	C	D	E	A	F
α(3413 cm^{-1})	0.346	0.377	-	0.035	0.141	0.013	0.008

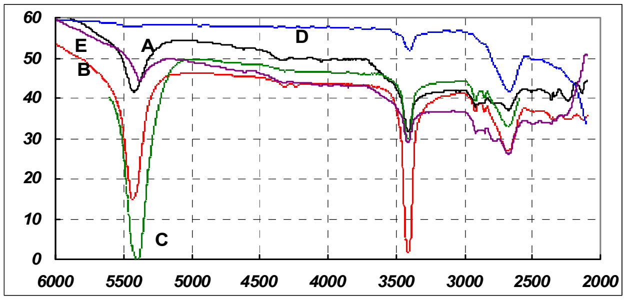


Figure 1. IR spectra of LGS Y-cut samples from 5 suppliers (absorption in % versus wave number in cm^{-1})

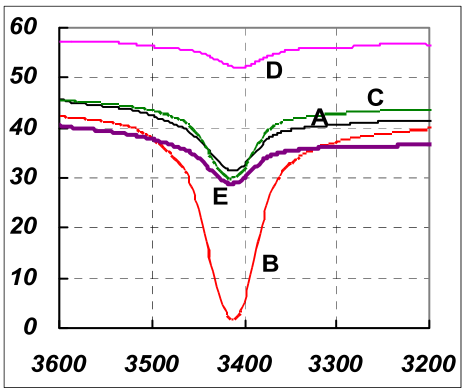


Figure 2. IR spectra of LGS Y-cut samples from 5 suppliers (absorption in % versus wave number in cm^{-1}), details of the Fig. 1.

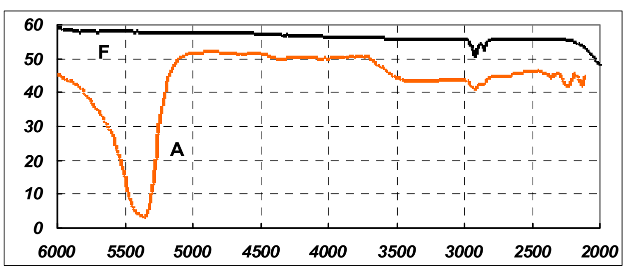


Figure 3. IR spectra of LGT Y-cut samples from various suppliers (absorption in % versus wave number in cm^{-1})

C. *Quality factor versus supplier*

For LGS as for LGT, it seems that resonators resulting from the less colored blocks have the highest Q-f product, i.e. D source for LGS and F source for LGT. We can notice that the best sample of LGS is five times better than the worst one (Figure 4), whereas there is a factor fifteen between LGT samples (Figure 5) on the 5th overtone. Moreover, these results are also completely validated by the infra-red analyses presented above.

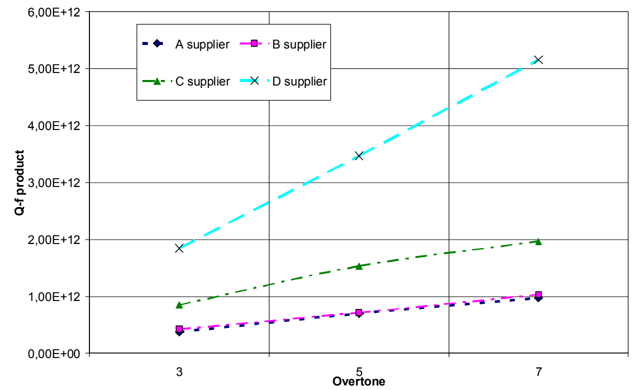


Figure 4. Q-f product (Quality factor × Frequency) versus overtone rank of LGS resonators from various suppliers

For LGS as for LGT, it seems that the resonators resulting from the the less colored blocks have the highest Q-F product. There is a factor 5 between worse and the best LGS and a factor 15 for the LGT (Figure 4 and 5).

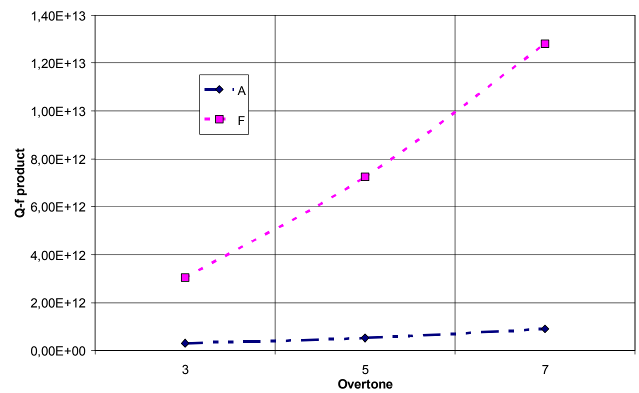


Figure 5. Q-f product (Quality factor × Frequency) versus overtone rank of LGT resonators from two suppliers

IV. Optimization of the Resonator

The material used for the resonator is the one from the F LGT supplier (which presents the best quality factor). Measurements presented below are made on resonators manufactured with the process presented in chapter II, we have adjusted the 5th overtone at 10 MHz.

We have optimized resonator parameters regarding their quality factor and motional resistance (Figure 6 and 7). On the 5th overtone, the optimum Q is obtained with a radius of curvature of 115mm with “usual” resonators (Table IV). For BVA ones (for which the diameter of the active part is equal to 10.2 mm instead of 13.2 mm in our standard resonator), the optimum is obtained with a radius of curvature of 100mm (Table V).

For resonators presented in Table V, the polishing step has been stopped at the 1µm grains size level. This explains why their Q factor is lower than the other ones (Table IV).

It is interesting to note the very low motional resistances of all 1st, 3rd and 5th overtones, which will imply a specific oscillator design. Unfortunately on both, resonators with optimized radii of curvature, the lowest motional resistance is not on their 5th overtone!

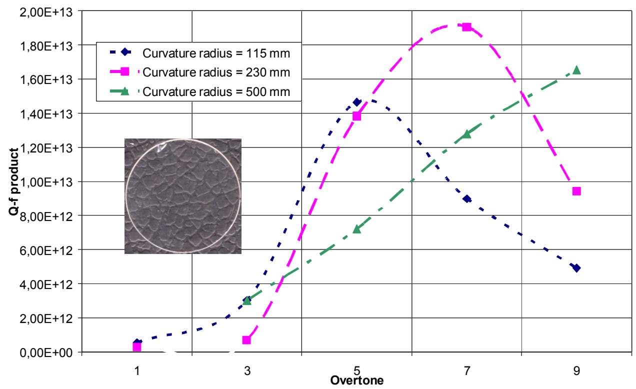


Figure 6. Q-f product (Quality factor × Frequency) versus overtone rank of LGT crystal resonators without bridges with various curvature radius

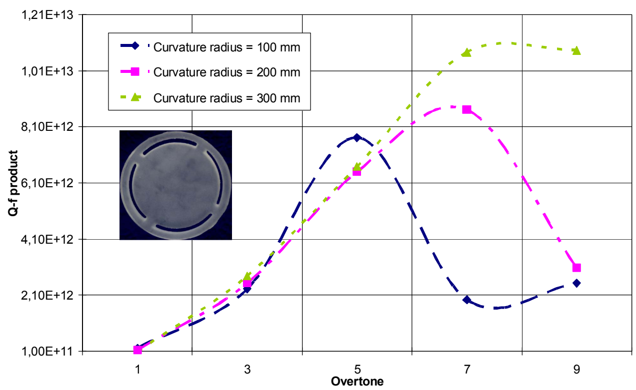


Figure 7. Q-f product (Quality factor × Frequency) versus overtone rank of LGT crystal resonators with bridges and with various curvature radius

TABLE IV. PLANO-CONVEX RESONATORS PARAMETERS (WITHOUT BRIDGES)

Curvature radius = 115 mm				
Overtone	1	3	5	7
Motional resistance (Ω)	1.7	11.7	7.8	34.3
Quality factor (10^6)	0.25	0.48	1.40	0.61
Frequency (MHz)	2.1	6.2	10.4	14.5
Curvature radius = 230 mm				
Overtone	1	3	5	7
Motional resistance (Ω)	2.7	3.7	5.7	10.3
Quality factor (10^6)	0.13	0.11	1.38	1.36
Frequency (MHz)	2.0	5.9	9.9	13.9
Curvature radius = 500 mm				
Overtone	1	3	5	7
Motional resistance (Ω)	-	9,5	12,2	17,4
Quality factor (10^6)	-	0.52	0.74	0.94
Frequency (MHz)	-	5.8	9.6	13.5

TABLE V. PLANO-CONVEX RESONATORS PARAMETERS (WITH BRIDGES)

Curvature radius = 100 mm					
Overtone	1	3	5	7	9
Motional resistance (Ω)	13	14.1	14.7	160	228
Quality factor (10^6)	0.11	0.42	0.83	0.15	0.15
Frequency (MHz)	1.8	5.5	9.2	12.8	16.5
Curvature radius = 200 mm					
Overtone	1	3	5	7	9
Motional resistance (Ω)	417.6	40.8	43.8	69.6	88.6
Quality factor (10^6)	0.01	0.11	0.21	0.23	0.28
Frequency (MHz)	1.8	5.5	9.2	12.8	16.5
Curvature radius = 300 mm					
Overtone	1	3	5	7	9
Motional resistance (Ω)	81.5	8.9	11.9	16.9	32.7
Quality factor (10^6)	0.04	0.50	0.72	0.83	0.65
Frequency (MHz)	1.8	5.5	9.2	12.8	16.5

V. CONCLUSION

As for quartz crystal, LGS and LGT crystals exist with different quality grades. We have proved it on different materials by two different ways: quality factor measurements on Y-cut resonators and infra-red absorption comparative spectra. Stability and quality of material are not yet guaranteed by all suppliers.

At this moment, LGT from certain supplier seems more adapted and more promising to realize ultra-stable oscillators. Moreover, for resonators built in the best material, we have optimized the energy trapping of the vibrating mode (5th overtone) on two different designs.

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