High Stability Evacuated HF Crystals

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Abstract.

This work is devoted to results of development of high frequency fundamental mode (HFF) crystal resonators fabricated with inverted mesa-structure procedure and sealed in HC-45 holders with high vacuum process. The paper deals with analysis of various parameters of the resonators. Most essential advantageous of the vacuum-sealed crystals over the conventional ones were observed in long-term frequency stability (aging). About 9 months aging tests with the HFF vacuum units operating at 103-156 MHz shown 0.5-1.0 ppm frequency drift for the first month and 2-3 ppm for the 8 months period at 85°C.

1. Introduction.

It's well known that a major cause of long-term instability (aging) of conventional HFF resonators is mass transfer onto resonator electrodes due to chemical reaction between electrode material and residual active gases in the sealed volume including oxygen and moisture. Residual gas analysis of hermetically sealed enclosures using resistance welding in "inert" atmosphere showed substantial amount of such contaminations. As a result frequency of the HFF crystals falls down almost linearly for years with about 5 to 10 ppm per year rate. Ambient temperature accelerating the chemical reaction on the electrodes can radically worsen the long-term stability as well as result into frequency changes. Obviously, sealing the HFF crystals in vacuum environment could radically improve "purity" of the crystal volume that should lead to considerable improvement of the frequency stability. As it's stated in [1] vacuum packaging the HFF strip crystals in the ceramic package provided excellent aging rate – within 2 ppm for 70 days at 85°C temperature.

Our goal was achievement of good aging results with standard HC-45 package and 5 mm diameter AT-cut inverted mesa-structure plate. To reach the goal we developed a technique, which consists of resistance welding the "holed" cover to the header, baking the units in high vacuum, and solder sealing the hole (using high melting temperature solder) without breaking the vacuum. As a result of the process at least 0.1 Pa vacuum level was reached inside the HC-45 holders.

Fabricated with described above technique HFF crystals for 103, 140 and 156 MHz were studied to compare their electrical parameters and frequency stability with those of similar crystals sealed using regular resistance welding procedure. The work describes and discusses results of the researches.

Practice of conventional HFF crystals proved the gold electrodes material as the best to attain better aging. However small conductivity to density ratio (CDR) restricts it's application in higher frequency range. Aluminum electrodes providing best electrical parameters of HFF crystals and good stability in vacuum conditions [1] create, however, some problems with connection to the mounting structure, requiring additional plating operation, as well as somewhat degrading frequency stability over temperature.

We've tried silver as basis electrodes material for the vacuum resonator as being an intermediate option between gold and aluminum in CDR factor and surface stability. The paper analyzes experimental data on electrical parameters and aging of the vacuum-sealed HFF resonators with silver and gold electrode materials in 103-156 MHz frequency range.

Important part of fabrication process of any crystal resonators is testing their aging parameter. However long-term measurement procedure becomes prohibitively expensive when large-scale production of the units takes place. We developed a simple method to replace long-term measurements of crystal aging by a few hours high temperature treatment. Simple expression for prediction of long-term behavior of the vacuum-sealed crystals upon frequency changes after the temperature treatment is considered in the paper.

2. Evacuated HFF crystals design and fabrication process.

Design of the vacuum HFF crystal consists of inverted mesa-structure AT-cut blank with deposited electrodes that is mounted in HC-45 holder. Sizes of silver or golden (optionally) electrodes vary from 0.4 – 0.6 mm depending on requirements to motional parameters of the unit. Thickness of the electrodes was chosen minimal to provide minimal loading on the crystal at sufficient electrical conductivity.

The process of fabrication of the vacuum units is schematically depicted in fig.1 and basically contains standard procedures except packaging which is implemented in two stages.

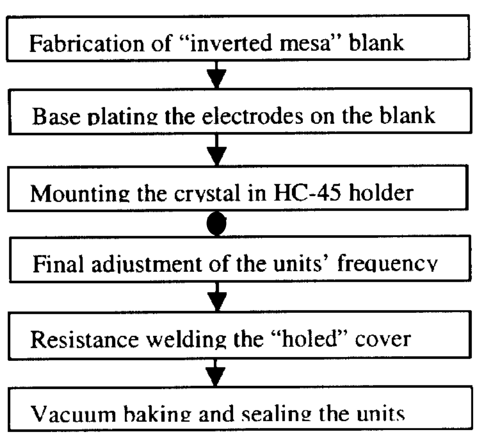


Fig.1. Process of fabrication of the vacuum HFF resonators in HC-45 packaging.

At the first stage the HC-45 base is welded with the cover using the resistance-welding machine. The cover is made “holed” for further evacuation of the packaging at the second stage. At the second stage the “hole” is hermetically sealed by closing it with solder in a vacuum chamber at 0.01-0.001 Pa vacuum level. For final cleaning the units are baked in the vacuum chamber at 200-250°C in the single cycle with the sealing procedure.

3. Electrical parameters of the vacuum resonators.

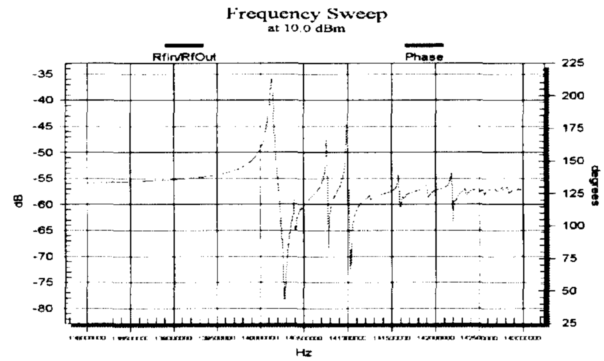
Electrical parameters of the vacuum units with Ag electrodes operating at 103 MHz and fabricated with above process were measured before and after packaging. Obtained figures are depicted in table 1 in comparison with the data for similar crystals with Al and Au electrodes sealed in vacuum environment. All the crystals have similar electrodes area (0.5 mm) providing equal motional capacitance of the units – about 4 fF. Thickness of the electrodes is different for the materials to provide acceptable conductivity at minimal mass loading.

Table 1. Electrical parameters of the 103 MHz HFF resonators

Type of the HFF resonators	Motional resistance, Ohms	Q-factor, K
Gas-filled units with silver electrodes	19	21
Evacuated units with silver electrodes	15	26
Evacuated units with aluminum electrodes	12	32
Evacuated units with gold electrodes	25	16

As one can see from the table evacuated crystals show about 20 per cent increase in Q-factor as compared with that of the gas filled units. As it could be expected the aluminum electrodes have minimal impact on the Q-factor providing lowest motional resistance of the units. At the same time motional resistance of the units with silver electrodes is 1.5 times lower than with gold ones.

It’s well known that mass loading affects inharmonic performance of HFF crystals. The less the CDR value of the electrodes material the more difficult is fulfillment of Bechman's criteria to suppress the inharmonic responses. Figure 2 illustrates inharmonic responses pattern for the 140 MHz vacuum resonators with silver electrodes with the thickness of 100 nm. As one can see level of the nearest inharmonic modes does not exceed –10 dB.



4. Long-term stability of the vacuum-sealed resonators.

The most significant difference between conventional and vacuum HFF resonators was observed in long-term frequency stability. Figures 3 shows results of aging for 57 pieces of 140 MHz vacuum-sealed crystals during first 3 months of operation at 85°C. Figure 4 shows aging of similar units during first 9 months period.

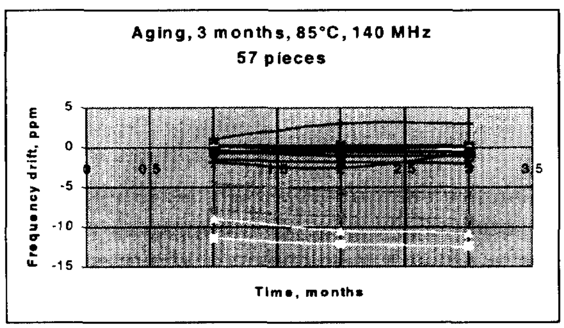


Fig. 3. Diagram for 3 months aging results of the vacuum crystals for 140 MHz.

As one can see from the data most of the units show frequency change within 2 ppm for 3 months test period and no more than additional 1 ppm for 6 months after that. Arbitrary sign of the frequency change is due to the fact that the mass transfer (a prevailing aging mechanism in gas-filled enclosures of HFF crystals) is comparable with other aging mechanisms. Obtained results radically differ from aging of conventional gas-filled resonators, which typically has negative sign with slope of 5-10 ppm per month at the same temperature.

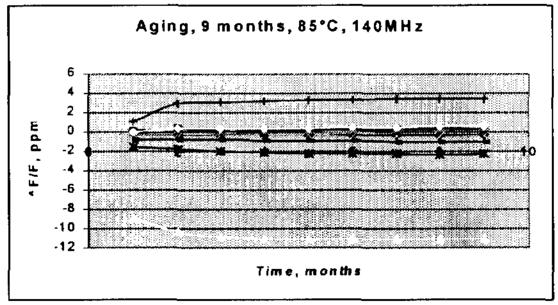


Fig. 4. Diagram for 9 months aging results of the vacuum crystals for 140 MHz.

To reveal influence of the electrode material on long-term stability of the vacuum-sealed HFF resonators we carried out comparative study of the 103.68 MHz units with Cr-Ag and Cr-Au electrodes. Obviously, usual "room" temperature conditions would require rather long test period to obtain reliable results, that's why we carried out accelerated aging tests at 150°C during 15 days (fig.5). As it follows from the figures, average aging rate for the Au-electrodes option is about 1.2 ppm per months, while for Ag-electrodes it's 0.7 ppm over test period. Excellent results for units with Ag-electrodes can be explained by high purity of the vacuum environment inside the sealed volume, and thus the absence of any active contaminating residuals.

Table 2. Comparative aging tests of the 103 MHz crystals with Au (a) and Ag (b) electrodes at 150°C during 15 days.

№	Au electrodes	Ag electrodes
1	0	0
2	0	0.1
3	0.1	-0.1
4	-4.8	0.1
5	-3.9	-2.9
6	-3.9	1.9
7	0	0
8	0	1.9
9	0	0
10	0.1	0.1
Average	1.2	0.7

Although obtained results are not sufficient for conclusion of superiority of silver over gold as a material for electrodes for vacuum-sealed resonators that, however, proves silver electrodes can be an attractive option taking into account cost reduction, as well as improvement in motional parameters of the resonators.

Accelerated aging test at 150°C described above can be used as a universal method to predict long-term stability of the vacuum-sealed HFF units. As it was shown in [2] the test period, test temperature and measured frequency shift of the units can be calculated by following equation:

$$\Delta f/f\ (T2, t2) = \Delta f/f\ (T1, t1), \quad (1)$$

where Δf/f (T2, t2) – frequency drift during t2 aging period at T2 ambient temperature;
Δf/f (T1, t1) – frequency change during t1 aging period at T1 ambient temperature;
t2= t1x2^[(T1-T2)/10].

Using above expression one can calculate, for example, that frequency shift observed with 103 MHz vacuum-sealed HFF resonators during 15 days at 150° C will be "reached" only after 4 years aging at 85°C. To predict aging rate for shorter time faster express test method is required. Table 3 compares frequency shifts of 155 MHz vacuum units after 150°C four hour treatment with their aging measured during 21 days at 85°C. The 21 days period was calculated with equation (1) as equivalent to 4 hours aging at 150°C.

Table 3. Frequency changes of the HFF crystals observed at different aging conditions, sealed with two different bake-out procedures.

Unit No.	Frequency change after 150°C, 4 hours treatment, ppm	Frequency change after 85°C, 21 days, aging, ppm
"Insufficient" bake-out process		
1	-2.1	-5.0
2	-3.5	-2.8
3	-1.4	-6,4
4	-2.8	-2.1
5	-2.8	-2.1
6	-2.1	-2.8
7	-8.5	-3.5
8	-9.3	-7.1
9	-2.1	-4.2
10	-8.5	-5.7
Σ/n	**4.3**	**4.2**
"Normal" bake-out process		
11	0.7	0.1
12	-0.7	-1.4
13	0.1	-0.7
14	-0.1	-0.7
15	2.1	1.4
16	-0.1	-0.7
17	-0.7	-0.7
18	-1.4	-2.1
19	0.1	0.7
20	0.7	0.7
21	0.7	0.1
Σ/n	0.74	0.93

As one can see from the data, there is a strong correlation between express test results and results obtained with usual aging test, that allows to use express test for both process validation and unit sorting. Another important conclusion can be drawn is of a necessity of proper bake-out processing.

5. Conclusions.

1. Developed process of fabrication of HFF vacuum-sealed crystals allows for significant improvement in long-term stability and frequency stability over temperature in comparison with HFF crystals using conventional fabricating processes. About 20% improvement of Q-factor of the vacuum-sealed units as compared with the gas-filled ones was also achieved.

2. The study discovers no noticeable difference in long-term stability of the vacuum-sealed resonators using Ag and Au electrodes. That allows application of silver electrodes instead of gold ones in the vacuum-sealed units in order to reduce cost and/or improve motional parameters and inharmonic responses even with tight requirements for long-term frequency stability.

3. Developed process opens up a possibility of application of the vacuum-

sealed HFF crystals in frequency control devices, such as VHF/UHF VCXO and XO used in SONET/SDH and other optical networks, where tight overall stability is a must.

4. High temperature aging test during a few hours allows for fast evaluation of the fabrication process and prediction of long-term behavior of the vacuum-sealed HFF resonators.

References.

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