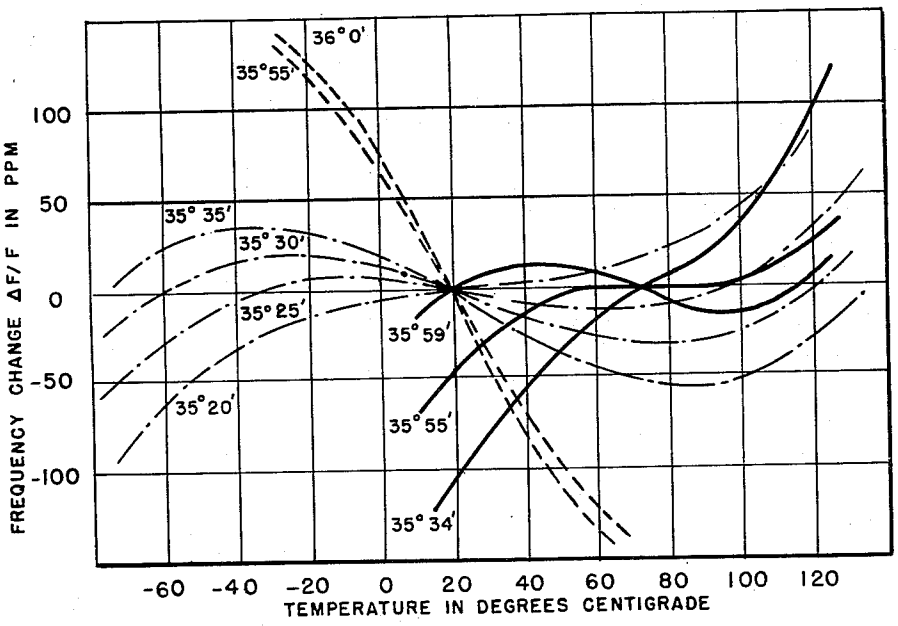
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METHOD OF FABRICATING A SYNTHETIC QUARTZ CRYSTAL

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METHOD OF FABRICATING A SYNTHETIC QUARTZ CRYSTAL

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The invention described herein may be manufactured and used by or for the Government for governmental purposes without the payment of any royalty thereon.

The present invention relates to synthetic quartz crystals and a method of making the same.

Quartz crystals in natural form have long been used as piezo-electric resonators. It has been found that quartz crystals in their natural form display certain inherent electrical properties. However, in many instances it has been found that quartz crystals having electrical properties varying from that of natural quartz might be more desirable and appropriate in certain particular usages.

It is therefore a primary object of this invention to provide a synthetic quartz crystal having electrical properties differing from those of natural quartz crystals.

It is another object of the invention to provide a synthetic crystal having the same desirable high Q and stability of natural quartz.

Another object of the invention is to provide a means for increasing the growth rate of fabricated crystals incorporating the desirable properties indicated above without imparting any undesirable characteristics thereto.

The above objectives and other advantages have been obtained by introducing certain impurities during the fabrication of synthetic crystals, such impurities being selected generally from the group of metals including aluminum, germanium, lead, tin and silver used either singly or in combination to secure certain preferred and desired characteristics.

The invention can best be understood from the following description and further explained in the accompanying sheet of drawing in which the single figure of the drawing shows the frequency-temperature characteristics of three different categories of crystals to be explained in greater detail hereinafter.

In the preparation of crystals where it is desired to incorporate such impurities, generally the same conditions were used in all the growths differing substantially only in the type of impurity element used. While the size of the autoclave to be used is determined by the size of the ultimate crystal desired to be obtained it has been found that excellent experimental test sized crystals can be obtained by using autoclaves which ranged in size from 250–500 cc. operated in pressure ranges from 5000–6000 p.s.i.

The mean temperatures at which such operations were carried out averaged approximately 350° C. as measured at the top and bottom of the autoclave; however, the specific temperatures for individual runs are indicated in the table below.

The individual runs were carried out under conditions generally used for the growth of synthetic quartz wherein sodium carbonate solution was utilized. The concentration of the solution was 0.5 N and the degree of fill in substantially all of the cases was approximately 70 percent of the capacity of the autoclave and the length of runs ran from eight to fifteen days. In the table below

are set forth individual runs setting forth specific additive impurities; the concentration of the impurity in solution; the concentration of impurity in the finished crystal as indicated in parts per million; the ionic radius; the temperature at top and bottom of the autoclave in which the run was completed, and the form of seed.

Run No.	Conc. of Impurity in sol.	Conc. of Impurity in Crystal (p.p.m.)	Ionic Radius (A)	Temp. at Top and Bottom of Autoclave, ° C.	Seed
A	0.2 N Ge+4	Ge—3,000	.53	339–381	Doubly oriented.
B	0.2 N Ge+4	Ge—1,000	.53	334–385	Do.
C	0.5 N Ge+4	Ge—3,000	.53	325–358	AT seed.
D	0.2 N Ge+4	Ge—1,000	.53	287–344	Do.
E	0.015 N Al+3	Al—100	.51	330–370	Doubly oriented.
F	0.015 N Al+3	Al—200	.51	351–399	AT seed.
G	0.03 N Al+3	Al—50	.51	320–376	Do.
H	0.015 N Al+3	Al—100	.51	332–348	Z seed.
I	0.06 N Pb+4	Pb—1,000	.84	315–395	AT seed.
J	0.05 N Pb+4	Pb—200	.84	345–378	Do.
K	0.018 N Sn+4	Sn—300	.71	311–341	Do.
L	0.05 N Ag+1	Ag—200	1.26	350–365	Do.

The additive impurity element was introduced into the quartz growing solution in a reactive form, for instance as a ready soluble compound such as its alkali salt, e.g., sodium aluminate for aluminum or in the form of its oxide, e.g., germanium dioxide. Similarly where desired the additive element can be added as a carbonate or other compound which can be used conveniently.

By referring to the graph shown on the drawing there can be seen the appreciable changes of the "doped" units as represented by the solid line which represents aluminum doped synthetic quartz resonators wherein the inflection temperature, that is, the temperature for which the second derivative of the temperature coefficient of frequency becomes zero, is indicated as approximately 75° C., whereas the inflection temperature of natural quartz resonators having approximately the same orientation angles as the doped crystals and represented by the broken lines, and the natural quartz resonators having the same frequency temperature characteristics as those with the doped units, as represented by the dot-dash lines have an inflection temperature of 20° C.

While there have been herein described certain preferred examples of the doped crystals, it is obvious that modification may be made without departing from the spirit and scope of the invention as set forth in the appended claims.

What is claimed is:

1. In the fabrication of a synthetic quartz crystal, the improvement comprising introducing aluminum into the solution from which said quartz crystal is made in an autoclave operated in pressure ranges from 5000–6000 p.s.i. at a temperature of about 350° C. for a period of from 8 to 15 days.

2. In the fabrication of a synthetic quartz crystal, the improvement comprising introducing aluminum into the solution from which said quartz crystal is made in an autoclave operated in pressure ranges from 5000 to 6000 p.s.i., at a temperature of about 350 degrees centigrade for a period of from 8 to 15 days and in an amount sufficient to produce aluminum in solid solution within a range of 50 to 200 parts per million.

3. In the fabrication of a synthetic quartz crystal, the improvement comprising introducing aluminum into the solution from which said quartz crystal is made in an autoclave operated in pressure ranges from 5000 to 6000 p.s.i., at a temperature of about 350 degrees centigrade for a period of from 8 to 15 days and in an amount

sufficient to produce aluminum in solid solution equal to 50 parts per million.

4. In the fabrication of a synthetic quartz crystal, the improvement comprising introducing aluminum into the solution from which said quartz crystal is made in an autoclave operated in pressure ranges from 5000 to 6000 p.s.i., at a temperature of about 350 degrees centigrade for a period of from 8 to 15 days and in an amount sufficient to produce aluminum in solid solution equal to 100 parts per million.

5. In the fabrication of a synthetic quartz crystal, the improvement comprising introducing aluminum into the solution from which said quartz crystal is made in an autoclave operated in pressure ranges from 5000 to 6000 p.s.i., at a temperature of about 350 degrees centigrade for a period of from 8 to 15 days and in an amount sufficient to produce aluminum in solid solution equal to 200 parts per million.

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