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FABRICATION OF QUARTZ RESONATORS

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The present invention relates to the fabrication of quartz crystals for electric oscillators.

In accordance with one method of fabricating quartz crystal dielectric elements for electric oscillators or resonators, the crystal elements are shaped to approximate desired dimensions by mechanical operations, and then the crystals are reduced in thickness mechanically until a desired piezo-electric vibration frequency is attained. It has been proposed to subject the crystal elements to chemical erosion by hydrofluoric acid in order to supplement and facilitate the mechanical dimensioning of the resonators to obtain a desired frequency. It was found, however, that the dissolving action of the hydrofluoric acid was too variable and erratic to be dependable. The rate of removal of quartz per unit of time was not uniform and an undesirable uneven dissolving action occurred. One area on the surface of blanks being treated may be attacked with greater vigor than other areas, thereby producing irregularities in dimensions which, in turn, affect the vibration frequency.

As a consequence, an undesirable variability in characteristics results when a batch of oscillator elements of the same size is subjected simultaneously to the same hydrofluoric acid treatment for the same length of time. In other words, the time required to produce a desired increase in vibration frequency cannot accurately be predetermined under such conditions.

In accordance with my invention the chemical activity between silica articles being treated and the acid etching reagent is regulated by associating the acid reagent with an effective amount of ammonium bifluoride which functions as a stabilizer.

As a consequence of my invention the etching effect, or removal of silica from the surface of an article of crystalline silica, is rendered uniform in distribution and occurs with a uniform rate which makes it possible to secure a predetermined result in a given time.

In carrying out my invention resonator elements of quartz are fabricated by cutting and rough grinding the oscillators to approximately a desired order of frequency, for example, to approximately 6000 kilocycles. As is well known, the frequency of piezo-electric oscillation is critically dependent on the thickness of the oscillating dielectric elements. If a slightly higher frequency is desired, careful grinding by highly skilled operators is required until the desired frequency is attained. Chemical reduction in thickness, if accurately controllable, permits

further approximation of desired frequency by unskilled labor with greater speed and dependability. However, the final accurate adjustment of dimensions of the resonator to obtain exactly a desired frequency may be carried out by a hand-lapping operation.

If considerable abrasive from a grinding operation is embedded in the surface of the quartz blanks as a result of the rough grinding, then, preliminary to subjecting the blanks to a timed acid treatment, the abrasive should be removed by a cleaning operation in an etching reagent. It is also desirable that oil or grease on the surface, even in the minute amounts derived from the skin when handling blanks, should be removed before subjecting the blanks to the etching reagent. This may be done by washing with acetone.

The clean crystals are subjected for a predetermined period of time at room temperature to the etching bath comprising an aqueous solution of hydrofluoric acid and ammonium acid fluoride (NH_4HF_2). Concentrated hydrofluoric acid is preferred as with weak acid the process is too slow. Commercial hydrofluoric acid consisting of an aqueous solution of HF containing about 47 to 53 per cent of hydrofluoric acid may be used. It should be saturated with ammonium bifluoride. About 35 grams of the ammonium bifluoride is dissolved in a sufficient amount of the commercial acid to produce 100 cc. of solution.

The etching rate of a given reagent may be determined by measuring the frequency of a chosen oscillator plate, exposing it for a given length of time, for example one minute, to the etching effect of the reagent, and then, after carefully cleaning the plate, again determining the frequency of oscillation. When the etching rate is reproducible with time, as is true when using the mixture above given, then a multiple of the unit time will give a predictable increase of frequency. With a quartz oscillator crystal oscillating at 2500 kc., the etching rate at 25° C. may be 1.4 kc. per minute while an oscillator of 4600 kc. may have an etching rate of 1.8 kc. per minute. The etching rate will be different for different grades of quartz and even for different sections of the same crystal.

The crystal blanks should be supported by a chemically inert holder, as for example a holder consisting of platinum or chromium steel, or of suitable non-reactive, resinous material. Relative motion between the reagent and the quartz blanks should be produced either by moving the quartz

blanks or by stirring the solution, the object being to renew the solution in contact with the surfaces to be dissolved and to brush away gas bubbles. The duration of the treating time will vary in accordance with the conditions, such for example as the temperature of the reagent, the amount of material to be removed and the concentration of the reagent, but in general it may be said that the duration of etching ordinarily will fall within the limits of about ten minutes to one hour. For example, if a frequency of 6030 kc. is desired, a plate which resonates at 6000 kc. may be exposed to the etching reagent for about ten minutes.

As the construction of piezo-electric oscillators containing dielectric elements consisting of thin plates of crystalline quartz are well understood in the art, it is not necessary to describe them in connection with the present method.

With a predetermined set of conditions for the etching, it is possible by the practice of my invention to predetermine the amount of material removed during a given period and thereby to approximate closely the frequency desired in the oscillating elements. The amount of skilled hand labor for the final adjustment of frequency thus is reduced to a minimum.

Quartz oscillator plates or other shaped quartz articles after being treated by contact with a reagent embodying my invention have a smooth polished surface.

The concentration of the solution as etching proceeds should be restored by approximate measures as the reaction proceeds in quantity production, for example by introducing anhydrous hydrofluoric acid to replace acid lost by reaction with the silica.

After the etching has been completed, the oscillator element should be very carefully cleaned to remove traces of reagent. It is desirable to rinse first with distilled water, then with a dilute solution of ammonium hydroxide, then to again wash with water, which preferably should be

heated, and finally to rinse with acetone. The rinsing operation, after the etching has been completed, may be carried out in some cases by boiling the completed elements for about ten minutes in a relatively large quantity of distilled water.

Careful and thorough cleaning is desirable to maintain the "acidity" or liveliness of oscillation of the quartz resonators.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. The method of fabricating resonators of crystalline quartz which consists in mechanically shaping said articles to approximate sizes, subjecting the shaped articles to the dissolving action of an aqueous solution of hydrofluoric acid and ammonium bifluoride until a desired size has been further approximated, washing said resonators with sufficient thoroughness to remove traces of adhering reagent and finally completing mechanically accurate dimensional sizing of said resonators.

2. The method of effecting desired change in piezo-electric vibration frequency of crystalline quartz resonator elements which consists in immersing said elements in an aqueous solution substantially saturated with hydrofluoric acid and ammonium bifluoride and having a substantially constant predetermined chemical activity and removing said elements upon the elapse of a calculated time interval whereby a desired dimensional reduction is attained.

3. The method of treating a quartz oscillator crystal to adjust the rate of oscillation thereof which consists in subjecting said crystal to a reagent consisting of a concentrated aqueous solution of hydrofluoric acid and ammonium bifluoride for a sufficient length of time to cause the oscillation frequency of said crystal to approximate the desired working frequency thereof and then completing the adjustment of frequency by reducing the thickness of said crystal mechanically.

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