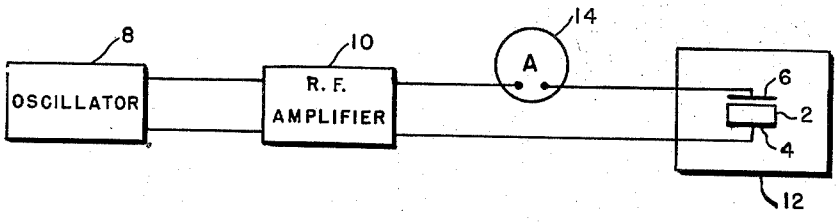
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CRYSTAL DETWINNING

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CRYSTAL DETWINNING

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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.

This invention relates to piezoelectric crystals and more particularly to a method of converting electrically twinned crystals to a uniform structure.

Piezoelectric crystals, especially quartz, are widely employed to control the operating frequencies of vacuum tube circuits, as elements in band-pass filters, and for many other related uses. The supply of natural quartz is limited and the expense and difficulties encountered in producing quartz crystals are quite substantial. It has been found that as much as half the quartz from which piezoelectric crystals are cut have to be discarded because of twinning, since uniform orientation of the crystalline material is necessary to produce satisfactory piezoelectric elements.

Twinning is the lack of homogeneity of the molecular structure of a crystal. The quartz crystal is subject principally to two primary twinning forms, namely the Dauphiné type in which one part of a crystal has suffered a 180° rotation about the Z axis with reference to the remainder of the crystal and the Brazilian type in which part of a crystal has the structure associated with the left-handed variety quartz and the remainder has the structure associated with the right-handed variety quartz, both parts being on a common Z axis. In a third form of twinning, the twinned part is of opposite hand to the parent crystal, the two parts being on a common Z axis, but the twin has suffered a 60° rotation so that the positive and negative rhombohedra comprising the crystals are coincident. In this latter form of twin, there is no reversal of the piezoelectric activity but the crystal is elastically and optically inhomogeneous.

Many methods have been attempted to correct the Dauphiné type of twinning. Invariably these methods have had little success in providing completely detwinned crystals. One difficulty encountered has been the effective application of heavy homogeneous unidirectional stresses. A second difficulity has been the inability to detwin crystal masses unless they are in the form of a thin blank.

It is, accordingly, the primary object of the present invention to provide a method for converting electrically twinned piezoelectric crystals to a uniform structure.

In accordance with the present invention, there is provided a method of treating a quartz crystal of symmetrical shape containing Dauphiné twinning so as to produce a uniform crystalline structure comprising driving the crystal at the fundamental frequnecy of a desired twinned portion for a predetermined period at a constant A. C. potential while simultaneously gradually heating the crystal to about the low-high inversion temperature and thereafter gradually cooling the crystal.

Also in accordance with the present invention, there is provided a method of treating a quartz crystal of symmetrical shape and containing Dauphiné twinning to

produce a uniform crystalline structure comprising the steps of tracking the frequency of a desired twin portion of the crystal while simultaneously gradually heating the crystal to a temperature where the response of the crystal becomes undetectable, the crystal having applied thereto during the tracking, an A. C. voltage of adjustable frequency, driving the crystal at this temperature for a predetermined period with an A. C. voltage which has a frequency equal to the frequency at the point of undetectable response, and gradually cooling the crystal.

For a better understanding of the invention, together with other and further objects thereof, reference is had to the following description taken in connection with the accompanying drawing and its scope will be pointed out in the appended claims.

It is known that quartz crystals for piezoelectric use such as AT and BT cut crystals change in response when their temperature is elevated until a point is reached where the response becomes undetectable. The frequncy of these cuts at various temperatures may be calculated or determined empirically. To observe the change as the crystal is gradually heated, a convenient method is to apply to the crystal, a constant A. C. voltage whose frequency can be adjusted to correspond to the fundamental frequency of the crystal at a given temperature. In effect, this enables the tracking of the response of a crystal as it is gradually heated.

In this manner, since the respective frequencies of the twin portions of a twinned crystal are known theoretically or empirically, a desired twin portion of a crystal may be tracked as it is gradually heated to the point where its response is practically undetectable. During the tracking operation, the crystal is driven at all times at the fundamental frequnecy of the desired twin portion.

Referring now to the drawing, there is shown an apparatus for practicing the method of the present invention in completely removing twinning from a twinned crystal when the crystal has one predominent mode of oscillation such as the thickness shear mode of AT and BT cut quartz crystals. It is to be understood, of course, that the method is effective with other type crystal cuts. The crystal **2** is positioned upon a metal plate **4** such as copper, silver, nickel, aluminum, etc., which acts as one electrode and a second metal plate **6** is positioned over the other surface of the crystal. Metal plate **6** need not be in contact with crystal **2**, a suitable air gap such as .003 inch therebetween providing means for preventing mechanical pressure or other deleterious effects. It is preferable that metal plates **4** and **6** have areas at least equal to the area of the crystal faces to subject the total crystal mass to the applied electric field uniformly. A ceramic crystal holder (not shown) suitably modified to withstand elevated temperatures may be used to contain the crystal. Crystal **2** is connected to an alternating current power source **8** such as an oscillator whose output frequency may be continuously varied over a considerable range which includes the frequency corresponding to that of the desired twinned portion of the crystal and whose output is applied to crystal **2** through an RF amplifier **10**. A heating device such as an oven **12** is provided to produce desired temperatures. A suitable meter **14** such as an RF milliameter is in circuit with crystal **2** to monitor crystal response. Crystal **2** may also be monitored by a device such as an incandescent lamp (not shown) whose light intensity would vary as the crystal response. The lamp would also serve the purpose of a current limiting device.

In operation the output of oscillator **8** whose frequency corresponds to the frequency of the desired twin portion is applied to crystal **2** through amplifier **10**. Oven **12** is turned on and heats crystal **2** at a rate small enough to

avoid thermal fracture of the crystal. It is preferable not to apply too great a voltage to avoid puncturing or fracturing the crystal, a value up to about 100 volts being practicable. The response or current of the crystal at its resonant frequency is indicated by meter 14. Since, at the crystal resonant frequency, its reactance is substantially zero, current is at a maximum at such frequency. Thus as the temperature of crystal 2 rises, its response changes due to a change in resonant frequency of the crystal and the frequency of oscillator 8 is varied constantly to follow or track the response of the crystal. Tracking the crystal may be accomplished by slowly sweeping the oscillator frequency through the desired frequency range, 25 to 50 cycles per minute being a good rate of sweep. The response is indicated on meter 14 under the above described conditions. As the crystal is slowly heated near to or above the inversion temperature of quartz, which is 573° C., the response changes, diminishing progressively. At a point near the inversion temperature, from about 450° C. to 550° C., the magnitude of the change of the response becomes so small that it may become undetectable. At this temperature, the frequency is slowly swept about the last noted frequency at which response was observed for a period of about 15 minutes, while the crystal is heated to about the inversion temperature, with care being taken to insure that the last noted response value is not permitted to approach closely the corresponding response value of the undesired twin portion of the crystal. The frequency is swept slowly and for a predetermined period as stated above to insure that crystal 2 has been subjected to the necessary force required for detwinning. The temperature of crystal 2 is then slowly reduced, while simultaneously sweeping it with the last noted frequency, until the desired detwinned crystal response of the crystal reappears. The crystal temperature is reduced further and its response is followed for a period to insure that the desired detwinned crystal response is being produced. At this point, the output of the oscillator may be removed from the crystal and the crystal is cooled to room temperature at a rate slow enough to avoid thermal shock. Twinned crystals which are lapped and etched after the application of the method of the present invention were found to be detwinned.

The following is believed to be the theoretical mechanism of detwinning in the present method. As the temperature of the crystal is increased, the mobility of the atoms in the lattice is increased. Now assuming it is desired to select one twinned portion of a crystal as the desired portion, upon following the response of this portion as the temperature increases, a point is reached at which the lattice mobility permits a change throughout the whole mass of the crystalline material to the desired lattice structure provided a strong enough field (enough energy being applied at the correct periodicity) is applied to the crystal at the correct frequency. The correct frequency to apply at elevated temperatures is the frequency noted at the temperature at which the crystal response disappears.

While there has been described what is at present considered to be the preferred embodiment of this invention, it will be obvious to those skilled in the art that various changes and modifications may be made therein without departing from the invention and it is therefore, aimed in the appended claims to cover all such changes and modifications as fall within the true spirit and scope of the invention.

What is claimed is:

1. A method of treating a quartz crystal of symmetrical shape containing Dauphiné twinning to produce a uniform crystalline structure comprising the steps of applying a constant alternating current voltage of variable frequency to a desired twin portion of the crystal while simultaneously gradually heating the crystal to a temperature near its high-low inversion point, the heating causing both an increase in the resistance and a change in the resonant frequency of the crystal, continually adjusting the frequency of the applied voltage during the heating to make it correspond substantially with the changing resonant frequency of the crystal, a temperature and a frequency being obtained at which the current passing through the crystal is substantially zero, applying said voltage with said obtained frequency at said obtained temperature to said crystal for a short period of such duration as to insure that the crystal has been subjected to the force required for detwinning and gradually cooling said crystal.

2. A method as in claim 1 wherein said crystal is an AT crystal.

3. A method as in claim 1 wherein said crystal is a BT crystal.

4. A method as in claim 1 wherein said potential is up to about 100 volts.

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