Oct. 6, 1959 J. M. STANLEY 2,907,808

INSULATED ELECTRODE

Filed Sept. 5, 1957

FIG. 1

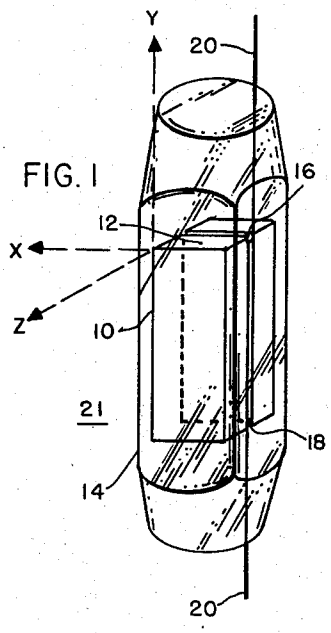


FIG. 2

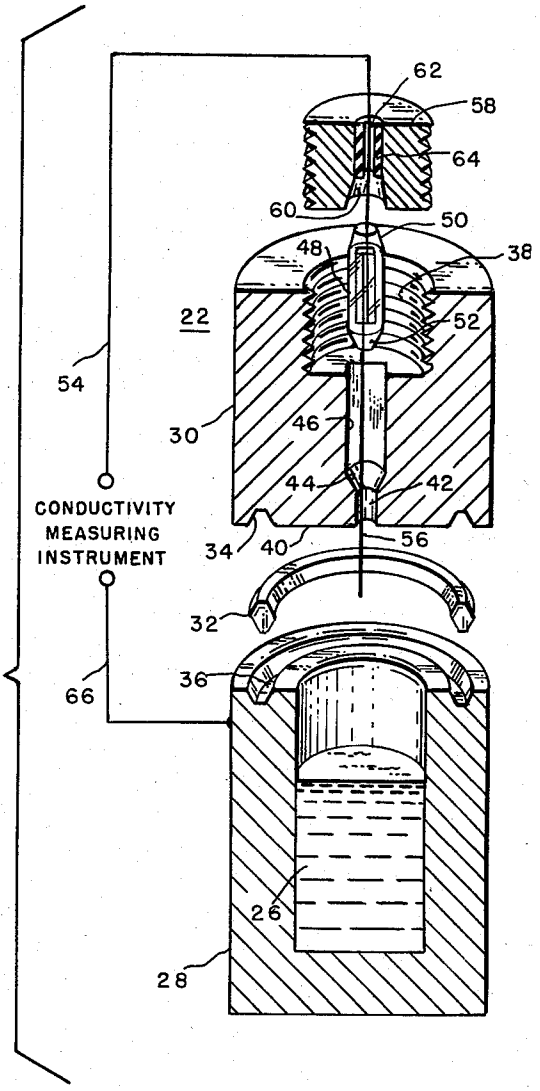
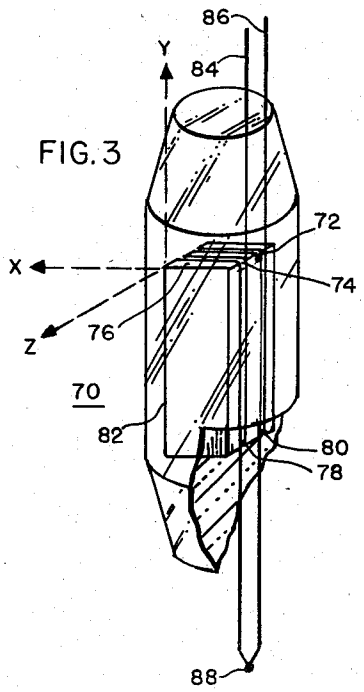


FIG. 3



INVENTOR,
JOSEPH M. STANLEY
BY
Harry M. Saragovitz
ATTORNEY

United States Patent Office 2,907,808 Patented Oct. 6, 1959

2,907,808

INSULATED ELECTRODE

Joseph M. Stanley, Spring Lake, N.J., assignor to the United States of America as represented by the Secretary of the Army

Application September 5, 1957, Serial No. 682,264

6 Claims. (Cl. 136—4)

(Granted under Title 35, U.S. Code (1952), sec. 266)

This invention relates to a method of producing a quartz and used by or for the Government for governmental purposes, without the payment of any royalty thereon. This invention relates to a method of producing a quartz crystal insulated electrode and to the quartz crystal insulated electrode produced by such a method. The present invention is particularly applicable to the production of an insulated electrode that is suitable for use in measuring the temperature and/or determining the conductivity conditions inside a sealed autoclave used for quartz growth though not limited thereto.

It has been customary in the art of growing crystals by hydrothermal techniques to make indirect temperature measurements of this type using thermocouple wells. Usually, such measurements may not have given the actual temperature of the crystallizing solution in the autoclave due to the mass of metal comprising the thermocouple well existing between the thermocouple and the crystallizing solution. In addition, previous measurements of conductivity of the crystallizing solutions were not uniformly successful due to the development of leaks in the ceramic seal of the insulated electrode and to the occurrence of corrosion therein.

The present invention is intended and adapted to overcome the difficulties and disadvantages of the prior art and to provide a new and improved method of making a more efficient insulated electrode which substantially avoids one or more of the limitations of the described prior devices used for making conductivity and/or temperature measurements of the crystallizing solution in an autoclave.

In accordance with the invention the quartz crystal insulated electrode is formed by winding a non-corrosive wire to be used as the electrode around a Y-bar quartz crystal seed, and depositing synthetic quartz thereon, under conditions suitable for the growth of synthetic quartz, so as to completely cover the wire.

The features of the invention which are believed to be novel are set forth with particularity in the appended claims. The invention itself, however, both as to its organization and method of operation, together with further objects and advantages thereof, may best be understood by reference to the following description taken in conjunction with the following drawing, wherein:

Fig. 1 is a isometric view of a Y-bar quartz crystal including a wire electrode constructed in accordance with the invention;

Fig. 2 is a cross-sectional view of an autoclave with which the electrode of Fig. 1 may be used; and

Fig. 3 is an isometric view, partially cut away, of another embodiment of the invention showing a Y-bar quartz crystal including a pair of thermocouple wire electrodes.

In Fig. 1 of the drawing, there is shown a water-insoluble Y-bar of natural or synthetic quartz seed 10 illustrating its X, Y and Z axis. Crystal 10 is suitable for depositing on its surfaces synthetic crystalline quartz and

has a natural tendency to grow more rapidly along its X and Z axes than along its Y axis. Satisfactory dimensions for Y-bar seed 10 may be 1 inch in length by ⅛ inch square in cross section.

Crystal 10 is in the form of a rectangular block, and the face 12 and its opposite face are slotted parallel to the X-axis, substantially at their mid-point to provide grooves 16 and 18 respectively therein. A non-corrosive conducting wire 20 such as silver, gold, platinum or any alloy of gold or platinum is looped around the length of crystal 10 and is recessed in the grooves 16 and 18 to uniformly position the wire thereon. The ends of wire 20 extend beyond the ends of crystal 10 along the Y-axis to terminate on opposite ends of bar 10, as shown.

The assembly is then placed in an autoclave containing quartz growth crystallizing solution, and using standard techniques for hydrothermal growth for synthetic quartz, the Y-bar seed 10 is built up to a quartz crystal. The growth is continued until a layer 14 of synthetic quartz completely seals and covers Y-bar seed 10 and wire 20. When this treatment is completed, the grown quartz crystal is removed from the autoclave and is cut and processed to form a quartz crystal 21 tapered at each end to provide a suitable air-tight seat when the invention is used as an insulated electrode in an autoclave setup, which will be described hereinafter. The body of the completed quartz crystal 21 may retain its square cross-sectional form as shown in Fig. 1, or it may be processed into a cylinder, so long as wire 20 is not exposed.

For a detailed description of the use of the invention, reference is had to Fig. 2, wherein the device shown in Fig. 1 is described in connection with an autoclave used in making measurements of the conductivity of the crystallizing solution. The autoclave does not constitute any part of the invention.

The autoclave 22 is a pressure vessel useful for growing synthetic quartz crystals from a crystallizing solution 26, which is contained in a stainless steel tank or other suitable metal container 28 arranged to be heated electrically or by any other means and maintained at an elevated temperature of between 350 and 400 degrees centigrade and operating at a pressure roughly between 400 and 800 atmospheres. Container 28 may be closed at its top with a cover 30 made of stainless steel or other suitable metal which may be held against container 28 with a suitable clamp and makes an air-tight seal therewith by means of a soft copper gasket 32 fitted into grooves 34 and 36.

The cover 30 is bored vertically at its top to provide the threaded recess 38 therein. The opposite end 40 is provided with a central bore or aperture 42 which extends vertically through the cover, flaring outwardly at 44 and opening into the elongated channel 46. The channel 46 in turn opens out into recess 38. Confined within the channel 46 is the Y-bar insulated quartz electrode 48 illustrated in Fig. 1, having both tapered ends 50 and 52 provided with wire terminals 54 and 56 respectively. Quartz insulator 48 is supported in an airtight sealed position by a metal gland nut 58, which has a hollowed-out portion 60 to receive tapered end 50 of electrode 48 when gland nut 58 is threaded into recess 38. Tapered end 52 fits securely in flared out portion 44 and forms an air-tight seal therewith.

When cover 30 is clamped against container 28, wire 56 is immersed into crystallizing solution 26. The other electrode 54 is brought through a reduced central opening 62 in gland nut 58 and is insulated therefrom by means of a ceramic tube 64 that completely surrounds electrode 54. A wire 66, connected to the outer wall of container 28 making electrical contact therewith and wire

54, respectively, are connected with a standard conductivity measuring instrument (not shown).

Fig. 3 shows an embodiment of the invention used for measuring the temperature of a crystallizing solution at elevated temperatures and pressures.

In this embodiment dimensions for the rectangular block of Y-bar seed **82** of natural or synthetic quartz that is used as a nucleus to grow the completed quartz crystal **70** may be 1 inch in length by ¼ inch square in cross-section. A pair of spaced grooves **72** and **74**, parallel to the X-axis are provided in face **76** of the Y-bar seed **82** and a corresponding pair of grooves **78** and **80** are provided in the opposite parallel face. Two non-corrosive thermocouple wires **84** and **86**, having different thermal characteristics such as platinum and platinum-rhodium or the like, are spaced from each other and separately looped around the length of Y-bar seed **82** parallel with the Y-axis. Wire **84** is wound in grooves **74** and **78**, and wire **86** is wound in grooves **72** and **80** in the face **76** and its opposite parallel face. The lower extremities of wires **84** and **86** are fused together at bead **88**. The assembly is then suspended in an autoclave and synthetic quartz is deposited thereon by conventional hydrothermal methods until the Y-bar seed **82** and the thermocouple wires **84** and **86** are completely covered by synthetic quartz in a manner similar to that described hereinbefore for the Y-bar seed **10**. When sufficient quartz has been grown on Y-bar seed **82** it is removed from the autoclave and the ends are tapered to form the completed crystal **70** as shown. In this one the body of the quartz crystal **70** is formed as a cylinder.

The completed crystal **70** may then be used in an autoclave, as described above in Fig. 2. However, in this embodiment the two ceramic insulated thermocouple wires **84** and **86** are brought through the reduced central opening **62** in gland nut **58**. Bead **88** is immersed into the crystallizing solution sufficiently to make proper thermal contact therewith, and a standard current measuring instrument (not shown) is connected to electrode wires **84** and **86**. The current produced by the thermocouple is a measure of the temperature.

Although in the specific embodiments only quartz is shown in producing the insulated electrode, other crystalline materials grown either hydrothermally or at approximate room temperatures and at atmospheric pressures may be used. However, such other crystalline materials should necessarily be water insoluble and mechanically strong.

While there has been described what are at present considered to be the preferred embodiments of the 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. The method of producing an insulated unitary electrode comprising looping a continuous non-corrosive conducting wire around a substantially rectangular body of Y-bar seed of water insoluble crystalline quartz having opposite elongated faces parallel to its Y-axis, said looping being parallel to said Y-axis, and growing synthetic quartz on said seed and over said wire to form a single crystal.

2. The method of producing an insulated unitary electrode comprising looping a continuous non-corrosive conducting wire about a substantially rectangular body of Y-bar seed of crystalline quartz having opposite elongated faces parallel to its Y-axis, said looping being parallel to said Y-axis, extending the discrete ends of said wire beyond the boundaries of said seed to form terminals, and growing synthetic crystalline quartz on said seed and over said wire to form a single crystal.

3. The method of producing an insulated unitary electrode comprising looping a pair of adjacent continuous non-corrosive conducting wires having different thermal characteristics on a substantially rectangular body of Y-bar seed of crystalline quartz having opposite elongated faces parallel to its Y-axis, said looping being parallel to said Y-axis, extending to discrete ends of said wires beyond to boundaries of said seed to form terminals, fusing one pair of said extended adjacent wires to form a thermocouple, and growing synthetic crystalline quartz on said seed and over said wire.

4. An insulated unitary electrode comprising a substantially rectangular body of Y-bar seed of crystalline quartz having opposite elongated faces parallel to the Y-axis of said seed, a continuous non-corrosive conducting wire on said elongated faces parallel to said Y-axis and having its ends extending beyond the boundaries of said seed to form terminals, and synthetic crystalline quartz grown on said seed and over said wire.

5. An insulated unitary electrode comprising a substantially rectangular body of Y-bar seed of water insoluble quartz having opposite elongated faces parallel to its Y-axis, and a non-corrosive conducting wire imbedded therein parallel to said Y-axis having ends extending from each side of said seed to form terminals.

6. An insulated unitary electrode comprising a substantially rectangular body of Y-bar seed of crystalline quartz having opposite elongated faces parallel to the Y-axis of said seed, a pair of adjacent continuous non-corrosive conducting wires having different thermal characteristics on said elongated faces parallel to said Y-axis and having their ends extending beyond the boundaries of said seed to form terminals, one pair of said ends of said extended adjacent wires being fused to form a thermocouple, and synthetic crystalline quartz grown on said seed and over said wire.

References Cited in the file of this patent

UNITED STATES PATENTS

2,456,831	Kjellgren	Dec. 21, 1948
2,546,305	Jaffe et al.	Mar. 27, 1951
2,638,408	Friedman et al.	May 12, 1953
2,680,677	Broge et al.	June 8, 1954
2,794,059	Smith	May 28, 1957