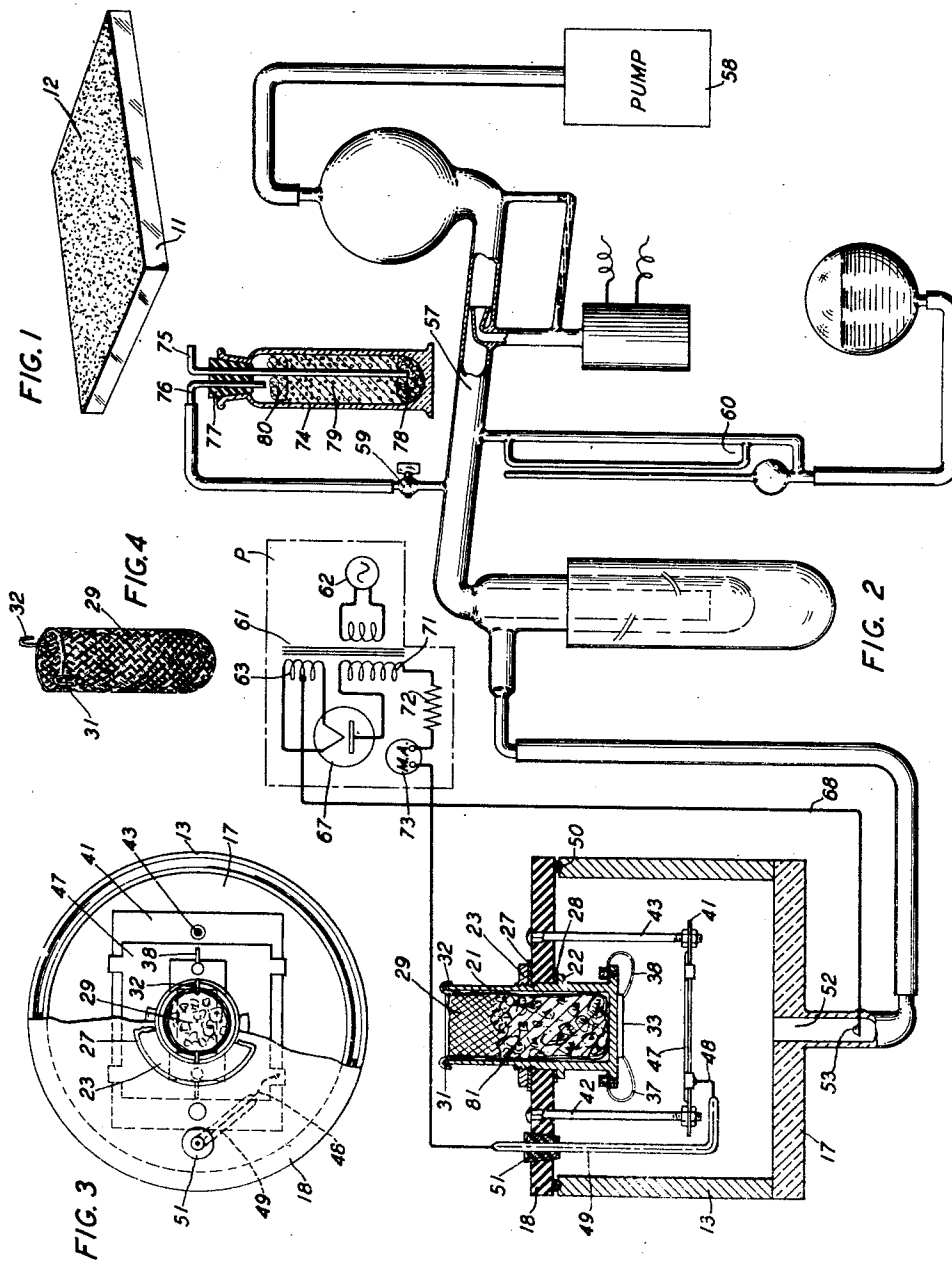


Nov. 2, 1937.

T. G. KINSLEY
PIEZOELECTRIC DEVICE
Filed Jan. 31, 1936

2,097,488



INVENTOR
T. G. KINSLEY
BY
E. V. Snigge
ATTORNEY

UNITED STATES PATENT OFFICE

2,097,488

PIEZOELECTRIC DEVICE

Thomas G. Kinsley, Bloomfield, N. J., assignor to
Bell Telephone Laboratories, Incorporated,
New York, N. Y., a corporation of New York

Application January 31, 1936, Serial No. 61,674

2 Claims. (Cl. 91—12.2)

This invention relates to piezoelectric elements and particularly to metallic coatings for such elements.

5 An object of the invention is to improve the performance of piezoelectric crystals.

Another object of the invention is to facilitate the conductive association of piezoelectric crystals with electrical circuits.

10 A more specific object of the invention is to provide a metallic coating on the surface of a piezoelectric crystal.

15 Piezoelectric crystals are, as is well known, commonly used as elements in electrical circuits where a constant frequency is essential and are of increasing importance in connection with wave filters and similar electrical networks. When the piezoelectric elements are used in such electrical circuits, it is necessary, of course, that some means be provided whereby the piezoelectric elements may be operatively associated with the circuit. It is common practice to provide for this purpose a metallic coating on one or more of the surfaces of the piezoelectric crystal element, this coating being electrically connected to the crystal surface to which it is applied. The desired conductors of the circuit may then be attached, for example by means of clamps, to the coatings. These coatings are commonly referred to as the electrodes of the crystal.

20 It has previously been proposed to apply metallic coatings to piezoelectric crystal elements by the sputtering process; U. S. Patent 1,848,630 issued March 8, 1932 to E. O. Hulburt containing reference to such an expedient. However, it has been found, particularly with respect to Rochelle salt crystals, that dehydration of the crystal results to a damaging degree during the sputtering process due to the fact that the vapor pressure of the crystal is much higher than the necessary sputtering pressure.

25 In accordance with a feature of the present invention, the crystal is cooled during the sputtering process until the vapor pressure of said crystal is lower than the necessary sputtering pressure whereby dehydration of the crystal is prevented.

30 In accordance with a specific embodiment of the invention, the sputtering process is carried out by means of a sputtering machine which includes a cooling chamber. After the Rochelle salt crystal has been placed in the sputtering machine but before the sputtering process itself is started, the cooling chamber is partly filled with a liquid having a very low freezing point such, for example, as acetone. Small lumps of solid carbon

dioxide are then dropped into the liquid, one after another, gradually cooling the liquid, cylinder and crystal down to the temperature of the solid carbon dioxide. After this, the sputtering process proper is started. Upon completion of the sputtering process, the solid carbon dioxide and liquid are removed from the cooling chamber and steps are taken to gradually bring the chamber and crystal back to room temperature.

35 It has been found that the Rochelle salt body is not damaged in any way by the sputtering process as practiced by applicant and that the resulting piezoelectric device is particularly satisfactory both mechanically and electrically. Speaking first from a mechanical standpoint, the electrode coating applied as proposed adheres closely to the body of the crystal, it having been observed that the bond between the coating and the body actually improves with age. From an electrical standpoint, the characteristics of the resulting piezoelectric device are particularly good, the "Q" value, for example, being substantially higher than that of Rochelle salt piezoelectric elements, the electrode coatings of which have been applied by other known methods. (The term "Q" is derived from consideration of the so-called equivalent electrical circuit of the piezoelectric device, "Q" being the ratio of the reactance to the resonance resistance of such a circuit. The theory and derivation of the equivalent electrical circuit of piezoelectric devices is explained on page 45 of "Quartz Resonators and Oscillators" by P. Vigoureux published by His Majesty's Stationery Office, London.)

40 A complete understanding of the invention and of its various advantages and possible modifications may be gained from a consideration of the following detailed description and the attached drawing, in which:

45 Fig. 1 is a perspective view of a piezoelectric crystal plate coated by the method contemplated by the invention;

50 Fig. 2 is a view of the sputtering apparatus contemplated by the invention, the sputtering chamber being shown as a sectioned side elevation and the power supply being illustrated schematically;

55 Fig. 3 is a plan view of the sputtering chamber, the cover being partially broken away to show the interior of the chamber; and

Fig. 4 is a perspective view of a basket utilized as a container for the cooling material.

Referring now to the drawing, a Rochelle salt piezoelectric crystal plate 11 is shown in Fig. 1 which plate is provided with a metallic coating 12. A similar coating may be applied on the other

major surface of the plate if desirable as well as on the sides and ends.

Referring now to Figs. 2 and 3, a sputtering chamber is shown which comprises a cylindrical metal shell 13 provided with a permanently attached base 17, preferably of glass, and a removable cover 18 of an insulating material such as a phenolic condensation product as, e. g., that identified by the trade-mark "Bakelite".

A cooling chamber 21 is mounted on cover 18. Flange 22 on the wall of the cooling chamber, nut 23 and gaskets 27 and 28 all cooperate to form an air-tight union between chamber 21 and cover 18. A mesh basket 29 (Fig. 4) is adapted to be positioned in cooling chamber 21, hooks 31 and 32 being provided to hold the basket in proper position as well as to facilitate its removal upon occasion. A Rochelle salt piezoelectric crystal plate 33 is shown mounted on the base of cooling chamber 21, being removably supported thereon by springs 37 and 38.

A mica plate 41 is supported from cover 18 by means of bolts 42 and 43. A thin sheet of gold, or other sputtering material, 47 is supported on plate 41; this sheet of sputtering material being the cathode of the sputtering apparatus. Cathode lead 48 is carried through cover 18 by glass tube 49, the tube being sealed at each end and an air-tight union between said tube and cover 18 being obtained by use of fitting 51.

A gasket 50 is mounted on the upper edge of shell 13 and cooperates in effecting an air-tight union between cover 18, when in closing position, and the sputtering chamber. The cover may, if desired, be provided with hinges or other attachment means (not shown).

Tube 52 is positioned centrally in base 17, anode 53 being located in this tube as shown. Tube 52 connects with mercury vapor pump 57 and oil pump 58 which pumps work in series to evacuate the sputtering chamber. Valve 59 serves to regulate the degree of evacuation. A mercury pressure gauge 60 is associated with the pumps.

A source of power supply P is schematically illustrated which source may comprise a transformer 61, the primary of which is connected to a suitable source of alternating current 62. A low voltage section 63 of the secondary is adapted to be connected to and to energize the cathode of rectifier 67, a center tap of this winding being connected to anode lead 48. One terminal of high voltage section 71 of the secondary is connected to the plate of rectifier 67 while the other terminal of section 71 is connected through resistance 72 and milliammeter 73 to cathode lead 48. Resistance 72 is provided to prevent arcing while milliammeter 73 may be used to indicate indirectly the degree of evacuation within the sputtering chamber since the current flowing between the electrodes is dependent upon the amount of gas remaining within the chamber.

It is very important that thoroughly dried air be available for supply to the sputtering chamber, such an expedient being necessary in order to prevent the collection of moisture on plate 33 during the cooling process to be described subsequently. Upon completion of the sputtering process it is important also that the air admitted to the sputtering chamber in order to reduce the vacuum therein be thoroughly dried as otherwise moisture may collect on the cold piezoelectric plate and dissolve the surface and damage the deposited metal coating. In order to assure a supply of thoroughly dried air, gas drying jar 74 is provided. Air inlet tube 75 and air outlet tube

76 are mounted in stopper 77, the last mentioned tube being connected to air inlet valve 59. Gas drying jars of this general type are commonly used and it is not considered necessary to describe its operation other than to point out that air enters through tube 75, passes through cotton 78, calcium chloride 79, more cotton 80 and finally to supply tube 76, the air being thoroughly dried during its progress through the jar.

Turning now to a description of the method of operation of the sputtering device, let us assume that it is wished to sputter Rochelle salt crystal plate 33. Cover 18 is removed from the chamber and plate 33 is then carefully mounted on the base of cooling chamber 21, being held in place by the pressure of springs 37 and 38. Cover 18 is then placed in closing position on the chamber and pumps 57 and 58 are set in operation, valve 59 being in open position. This (the fact that valve 59 is open) allows air thoroughly dried by passage through jar 74, to be pumped into the sputtering chamber, the pumps usually being left operating for ten or fifteen seconds after which they are stopped. Cooling chamber 21 is now partially filled with a liquid having a low freezing point, such as acetone, after which lumps of solid carbon dioxide 81 are dropped into the liquid, one after another, gradually cooling the liquid, the sputtering chamber and Rochelle salt crystal plate 33 down to the temperature of the solid carbon dioxide (approximately -57°C.).

It is well to start the pumps a few times during this chilling process, with valve 59 open, thereby assuring dry air in the sputtering chamber in order to prevent moisture and frost collecting on crystal 33.

The vapor pressure of Rochelle salt crystal plate 33 is now well below the pressure to which it will be subjected during the subsequent steps in the sputtering process, this lowering of the vapor pressure resulting from the substantial reduction of the temperature of the plate just described. While applicant has not determined the exact vapor pressure of the Rochelle salt crystal at the approximate temperature of -57°C. he has established conclusively that it is definitely lower than the lowest pressure at which gold is successively deposited. Gold is capable of being sputtered between the pressures of .06 and .2 millimeter of mercury and applicant has demonstrated by actual tests that a Rochelle salt plate is not dehydrated or damaged otherwise when sputtered according to the method which he has invented even though the sputtering process be carried out at the lowest pressure at which gold can be successfully sputtered, i. e., .06 millimeter of mercury.

After the sputtering chamber and crystal plate 33 have been thoroughly chilled, valve 59 is closed and pumps 57 and 58 are again set into operation and allowed to run until the pressure in the sputtering chamber as indicated by gauge 60 has been reduced to the desired sputtering pressure; some value between .06 and .2 millimeter of mercury. The high voltage, approximately 2500 volts, is then applied across cathode 47 and anode 53 and the actual sputtering is started. The current is kept constant (approximately .016 ampere) by means of valve 59 for a period of about twelve minutes at the end of which time, it will be found that the exposed surface of crystal plate 33 has been properly coated.

Before removing plate 33 from the sputtering chamber it is desirable that its temperature be gradually raised to room temperature in a dry

atmosphere. In order to accomplish this, basket 20, which contains the lumps of solid carbon dioxide 21, is first removed from cooling chamber 21 after which the liquid is absorbed by means of a cloth. After the contents of chamber 21 have been removed, dry air, heated if desired, is forced therein by means of a pump and tubing, the supply of air being continued until the sputtering chamber and plate 33 have been restored to room temperature. Cover 18 may then be removed, dry air being first admitted to the sputtering chamber through valve 50, and crystal plate 33 detached. If it be desired that the other side of plate 33 be sputtered also, the plate is replaced on the mounting in reversed position, cover 18 is replaced on the chamber and the sputtering process is repeated.

While certain specific embodiments of the invention have been selected for detailed description, the invention is not, of course, limited in its application to those embodiments. The embodiments described should be taken as illustrative of the invention rather than as restrictive thereof.

What is claimed is:

25 1. Apparatus for applying a metal coating to Rochelle salt crystals by the vacuous dispersion of electrodes comprising a chamber, a removable cover for said chamber, means for evacuating said chamber, an anode and a cathode, a source

of suitable electrical potential connecting said anode and said cathode, a second chamber mounted on said removable cover, a portion of said second chamber being within said first chamber and another portion of said second chamber being outside said first chamber when said cover is in closing position, and means for removably supporting a Rochelle salt crystal on the first-mentioned portion of said second chamber. 5

2. Apparatus for applying a metal coating to Rochelle salt crystals by the vacuous dispersion of electrodes comprising a chamber, a removable cover for said chamber, means for evacuating said chamber, an anode and a cathode, a source of suitable electrical potential connecting said anode and said cathode, a second chamber mounted on said removable cover, a portion of said second chamber being within said first chamber and another portion of said second chamber being outside said first chamber when said cover is in closing position, means for removably supporting a Rochelle salt crystal on the first-mentioned portion of said second chamber, and means associated with said second chamber for lowering the vapor pressure of the Rochelle salt crystal supported thereon below the normal value thereof. 10 15 20 25

THOMAS G. KINSLEY.