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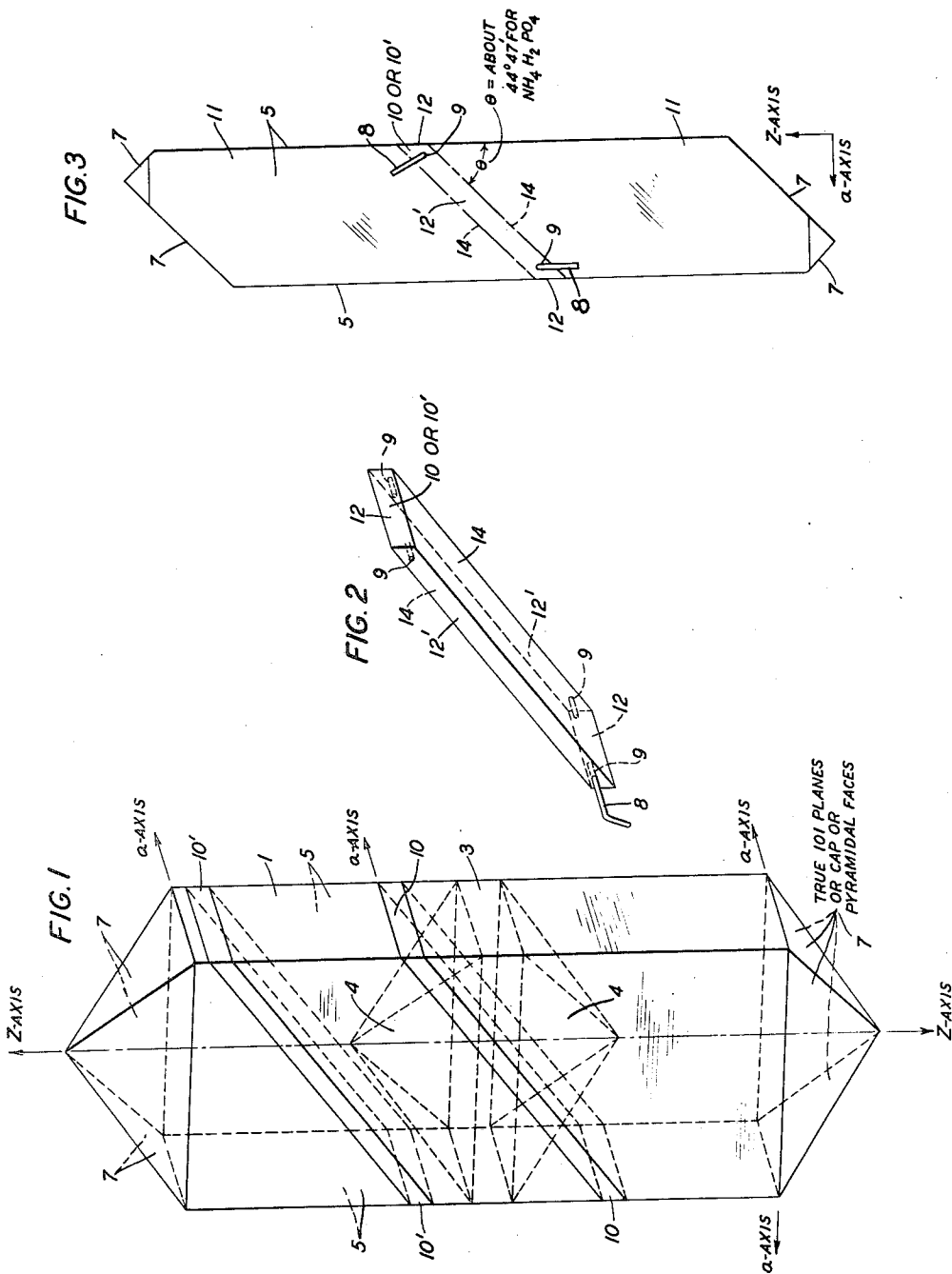
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2,442,755

PIEZOELECTRIC CRYSTAL GROWING METHOD

Filed June 11, 1945

2 Sheets-Sheet 1



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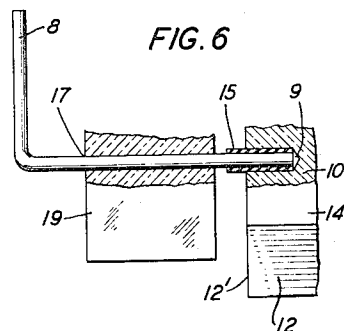
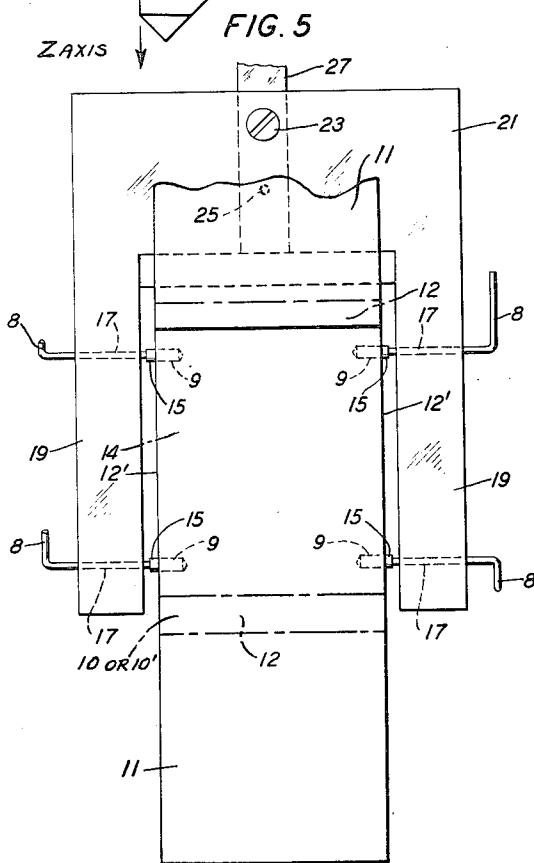
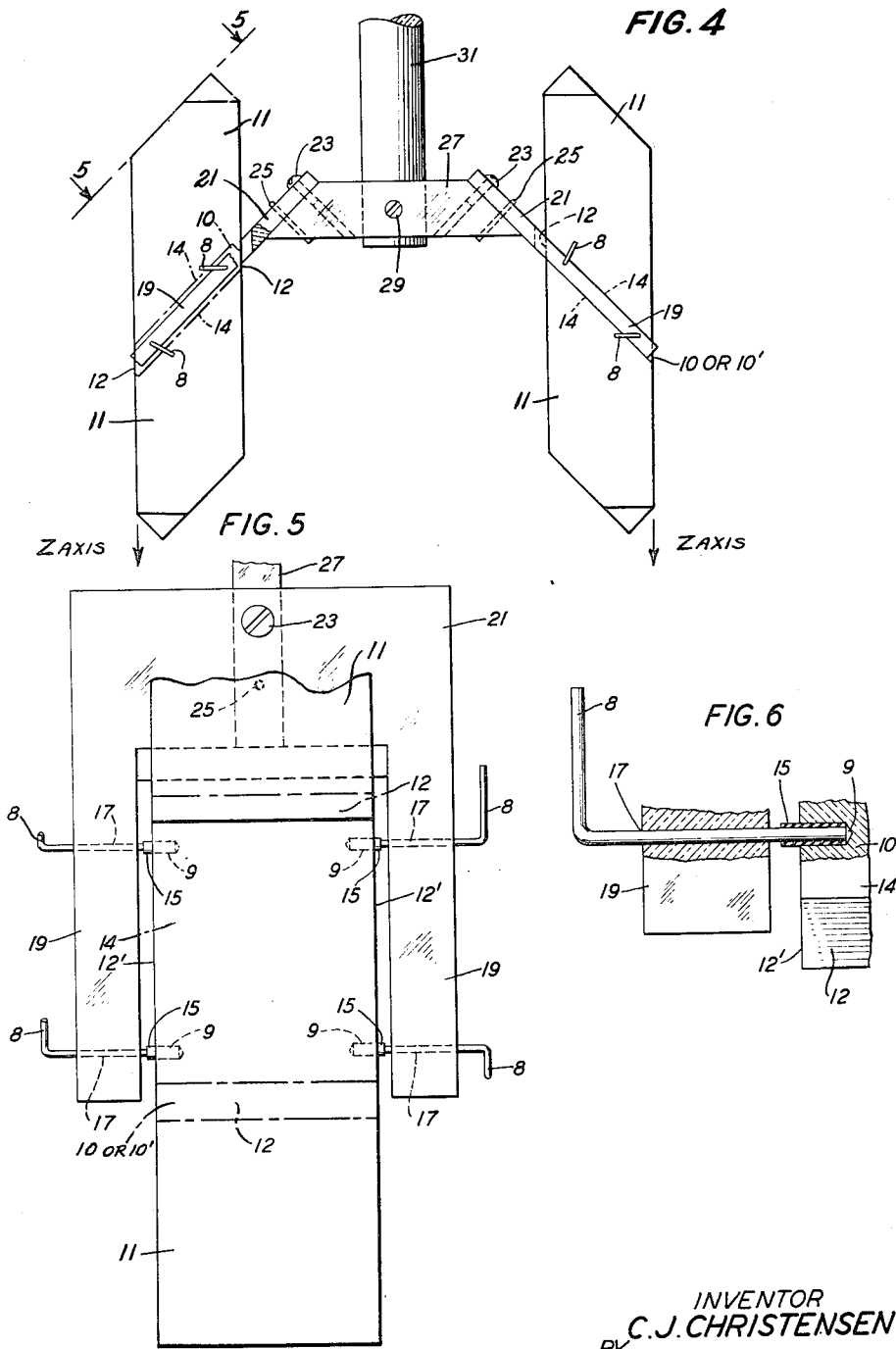
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2 Sheets-Sheet 2



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PIEZOELECTRIC CRYSTAL GROWING METHOD

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17 Claims. (Cl. 171-327)

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This invention relates to piezoelectric crystals and particularly to seed crystals that may be utilized for growing crystals in a nutrient solution.

One of the objects of this invention is to reduce waste in crystalline materials.

Another object of this invention is to provide crystals having a relatively large usable piezoelectric content free from flaws.

Another object of this invention is to provide a seed crystal that may be cut from waste crystalline material and yet which may be grown to produce a crystal having a large proportion of crystalline material usable for piezoelectric purposes.

This invention will be described particularly in connection with piezoelectric crystals comprising ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$), but it will be understood that it may also be embodied in many other types of crystalline substances of similar tetragonal form or otherwise.

In accordance with this invention, a seed crystal plate may be cut from a crystal bar, such as that of ammonium dihydrogen phosphate, so that the two major faces of the seed plate are parallel or nearly parallel with respect to the $\{01\}$ plane of any one of the pyramidal cap faces of the crystal bar. This type of seed plate is a useful form of seed for growing a crystal bar in a nutrient solution.

In the construction of many types of electrical systems, piezoelectric crystal elements are often used as electrical circuit elements therein, and such piezoelectric elements may be cut with any suitable size, shape and crystallographic orientation to suit the particular application. As an example in the case of crystalline ammonium dihydrogen phosphate, some of the useful piezoelectric cuts are the Z-cut crystal plate, and the 45 degree Z-cut crystal plate, and others. Such piezoelectric elements are cut from a suitable crystal bar which has been grown from a seed crystal in a suitable nutrient solution. While such a crystal bar may be grown from many different shapes and types of seed crystal, it is often desirable, especially in connection with large scale commercial production, to grow the crystal bar from a type of seed crystal which gives a relatively large content of usable piezoelectric material that is free from flaws. In accordance with this invention, a diagonal type seed plate is provided which is capable of growing a crystal bar having a relatively larger content of usable piezoelectric material therein than is obtained from other types of seed crystals of the prior art.

In order to conserve material in connection with large scale production of crystal material,

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it is often desirable that the seed crystals used for growing the crystal material be provided from sources other than the manufactured material that is to be or could be used in the construction of the piezoelectric elements intended for use as circuit elements in the ultimate electrical system. In accordance with this invention, the diagonal type seed plate may be cut from flawed or normally wasted crystalline material and yet be capable of growing a good crystal bar therefrom the grown part of the crystal bar being free from any flawed crystal material.

For a clearer understanding of the nature of this invention and the additional advantages, features and objects thereof, reference is made to the following description taken in connection with the accompanying drawings, in which like reference characters represent like or similar parts and in which:

Fig. 1 is a perspective view of an ammonium dihydrogen phosphate crystal bar, and a diagonal seed plate in accordance with this invention;

Fig. 2 is a perspective view of a diagonal seed plate as cut from the crystal bar shown in Fig. 1;

Fig. 3 is a side view of a crystal bar grown from a diagonal seed plate of the type shown in Fig. 2;

Figs. 4, 5 and 6 are views of a mount which may be used for mounting and growing the seed plate of Fig. 2 into a crystal bar as shown in Fig. 3, Fig. 5 being an enlarged view taken on the line 5-5 of Fig. 4, and Fig. 6 being an enlarged view of the lower left corner portion of Fig. 5.

Referring to the drawing, Fig. 1 is a perspective view of an ammonium dihydrogen phosphate crystal bar 1 which has been grown in a suitable nutrient solution from a seed crystal plate 3, shown in outline by the broken lines in Fig. 1. The crystal bar 1, as illustrated in Fig. 1, has four equal side or prism faces 5, and at the opposite ends thereof there are two sets of four principal cap or pyramidal faces 7 which by convention are designated $\{01\}$ planes. The optic axis Z of the crystal bar 1 extends along the direction of the length dimension of the crystal bar 1, and the mutually perpendicular a-axes extend along the directions of the width dimensions of the prism faces 5, the two mutually perpendicular a-axes being perpendicular to the optic axis Z. The optic axis Z is sometimes referred to as the c-axis, and the mutually perpendicular a-axes are sometimes referred to as the X and Y axes. The a-axis cross-section of the crystal bar 1, may be of square shape, or of other rectangular shape and of any desired size. The Z-axis over-all length of the crystal bar 1 may be made any

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desired length dependent upon the length of time of its growth from the seed crystal 3 and other factors. It will be understood that the crystal bar 1 may be grown from a suitable seed crystal 3 in a suitable nutrient solution by any suitable crystal growing apparatus such as for example by a reciprocating rotary gyrator type crystallizer as disclosed and claimed in a copending application, Serial No. 598,691, filed June 11, 1945, by A. N. Holden, assigned to Bell Telephone Laboratories, Incorporated.

The seed crystal 3 from which the crystal bar 1 of Fig. 1 is grown may be any suitable seed crystal. The seed crystal 3 illustrated in Fig. 1 is a Z-cut crystal plate 3 having its four naturally grown edge faces coextensive with the four prism faces 5 of the crystal bar 1, in which case the two cone sections 4 as shown in outline by broken lines in Fig. 1 may represent flawed cone sections in the crystal bar 1 which have been formed during the beginning of the growth of the crystal bar 1 from the Z-cut seed crystal plate 3. Alternatively, the sections 3 and 4 taken together may represent a naturally grown capped Z-cut seed crystal 3 and 4, from which the crystal bar 1 has been grown directly. The sections of the crystal bar 1 disposed beyond the ends of the sections 4 may represent clear good quality crystal material from which piezoelectric plates may be cut for use as circuit elements in electrical systems generally.

The crystal plate 10 or 10', as shown in Figs. 1 and 2, illustrates a diagonal seed plate in accordance with this invention, which has been cut from the mother crystal bar 1. The orientation of the diagonal seed plate 10 or 10' may correspond to the orientation of any of the natural cap or pyramidal faces 7 which follow the 101 planes of the crystal bar 1. This results in an orientation for the diagonal seed plate 10 or 10' wherein one set of the opposite edges of the faces 12 and 14 of the seed plate 10 extends in an a -axis direction, and the two major plane faces 14 of the diagonal seed plate 10 are parallel or nearly parallel to the plane of a cap face 7 and in the case of ammonium dihydrogen phosphate, are inclined at an angle of θ —about 44 degrees and 47 minutes with respect to the optic axis Z. The diagonal seed plate 10 may be cut to any convenient thickness dimension such as down to $\frac{1}{16}$ inch for example.

As illustrated in Fig. 1, the four small edge faces 12 and 12' of the diagonal seed plate 10 or 10' may be comprised of the four naturally grown prism faces 5 of the crystal bar 1, the two major plane faces 14 of the seed plate 10 or 10' being cut faces. While a series of such diagonal seed plates 10 or 10' of the same orientation may be cut from other portions of the crystal bar 1, the seed plate 10, as shown in Fig. 1, is cut from the original seed area 3 and 4 of the crystal bar 1. This region 3 and 4 ordinarily includes waste piezoelectric material, but in accordance with this invention, a useful seed plate 10 may be obtained from the region 3 and 4 of the crystal bar 1. Accordingly, the diagonal seed plate 10 cut from the flawed region 4 as shown in Fig. 1, may contain some flawed crystalline material 4 in the central regions of the seed plate 10 away from the peripheral edges 12 and 12' thereof, and still be used in growing a good crystal bar 11 therefrom, as illustrated in Fig. 3.

Fig. 3 is a side view of an ammonium dihydrogen phosphate crystal bar 11 as grown from a diagonal seed plate 10 or 10' of the type and

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orientation shown in Figs. 1 and 2. The crystal bar 11 of Fig. 3 may be grown from the diagonal seed plate 10 or 10' in a nutrient solution in a known manner, the Z-axis length of the crystal bar 11 being dependent upon the time of growth in the nutrient solution, and the a -axis cross-section of the crystal bar 11 being determined by the projected a -axis dimensions of the seed plate 10 or 10' from which the crystal bar 11 is grown. Due to the orientation of the seed plate 10 or 10' from which the crystal bar 11 is grown, it will be noted that the cap or pyramidal faces 7 thereof may be of different resultant lengths but of the same angular orientation with respect to the crystallographic axes Z and a .

For the purpose of growing the seed plate 10 or 10' into the crystal bar 11 of Fig. 3, the seed plate 10 or 10' may be mounted in suitable crystal growing apparatus in any suitable manner, such as for example by one or more supporting stainless steel wires 8 having ends extending into small holes 9 that may be drilled in the seed plate 10 at the peripheral region labeled 9 in Fig. 2. If these holes 9 are placed near the corners of the seed plate 10 and are shallow, the plate 10 can be suitably mounted to a plastic support as shown in Figs. 4 to 6 with the stainless steel wires 8 and so that the generally most useful 45-degree Z-cut piezoelectric plates can be cut therefrom without crystal wastage in or near the regions where the crystal seed 10 was mounted.

In commercial crystal bar growing, it is often desirable that the crystal bar 11 of Fig. 3 be grown with predetermined approximate a -axis dimensions, and this requirement involves the dimensions of the diagonal seed plate 10, which is oriented with its crystallographic axes Z and a coinciding with those of the grown crystal bar 11. The seed plate 10 so oriented with its crystallographic axes coinciding with those of the crystal bar 11 may be a suitable seed for growing the crystal bar 11, provided the peripheral edges of the seed plate 10 touch or coincide with each of the expected four prism faces 5 in at least one point. It is quite unimportant if the central portions of the seed plate 10 are imperfect since a suitable crystal bar 11 may be produced therefrom if the peripheral edges 12 and 12' of the seed plate 10 are of clear material of uniform orientation and at least $\frac{1}{16}$ inch thick. Also, seed plates 10 having small purely mechanical defects such as broken or cut corners and small chips out of the edges 12 and 12' will heal over in the growing process. It will be understood that diagonal seed plates 10 which are cut from the center regions 3 and 4 of the crystal bar 1 of Fig. 1 may have flawed crystal material on their major faces 14, but since these surfaces are surrounded by good solid crystal, such a diagonal seed plate 10 will heal over quickly when grown and give a good crystal bar 11, as illustrated in Fig. 3.

Alternatively, as shown in Fig. 3, a crystal bar 11 may be grown from an essentially unflawed diagonal type seed plate, such as 10' in Fig. 1, that shows such perfect joining of the crystal bar 11 onto the major faces 14 of the diagonal seed plate 10' that the whole crystal bar 11, even the portions containing the original seed material, can thereafter be used for cutting therefrom unflawed usable piezoelectric plates. While the major faces 14 of the diagonal seed plate 10' are cut parallel to and closely approximate the orientation of the naturally grown pyramidal cap faces

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7 of the crystal bar 1 of Fig. 1, they may depart slightly from that 101 plane by at least one-half degree for crystals 11 whose a -axis dimensions are 1½ inches and still give a diagonal seed plate 10' capable of yielding a substantially perfect joint with the crystal bar 11 of Fig. 3, and hence provide a crystal bar 11 practically all of which can be economically used for making piezoelectric plates therefrom for use as circuit elements in piezoelectric systems. Crystal bars 11 grown from diagonal seed plates 10' which have no flaws are practically free from veils and other crystal flaws running through the interior of the crystal bar 11. These and other improved results give the diagonal seed plate 10' an advantage over other types of seeds that are used for growing crystal bars.

Seed plates which are cut with their major surfaces approximating to the orientations of naturally growing crystal faces, of the type of cut 10, are useful not only as seed crystals in growing ammonium dihydrogen phosphate crystals 11 but also are useful for growing other kinds of crystals from a suitable nutrient solution. Depending on the growth habit and form of the particular crystal being grown, the diagonal plate type of seed 10 may be used for growing crystals other than ammonium dihydrogen phosphate crystals and similar tetragonal crystals such as potassium dihydrogen phosphate, ammonium dihydrogen arsenate and potassium dihydrogen arsenate. Analogous seed plates can also be cut from crystals of other classes, depending upon the crystallizing habit of the crystal. In general, where the seed crystal plate, such as the seed plate 10 or 10', can be cut with its two major plane faces disposed parallel to and in the same crystallographic orientation as that of a surface which is natural to the growing habit of the crystal material and on which crystal growth will take place, it may be utilized for growing a crystal therefrom in connection with other classes of crystal material than those particularly mentioned in this specification.

Figs. 4 to 6 are views illustrating a form of crystal support which may be utilized for mounting two seed crystal plates 10 or 10' and for growing such seed plates, in a suitable nutrient solution, into crystal bars 11 of the type illustrated in Fig. 3. In the particular mounting arrangement shown in Figs. 4 to 6, the two seed plates 10 or 10', which may be of different thickness dimensions as illustrated by the two crystal plates 10 in Fig. 4, are each carried by four wires 8, such as stainless steel or other suitable wires 8. The ends of each of the four supporting wires 8 may extend horizontally into four corresponding small and shallow holes 9 which may be drilled into the two opposite edge faces 12' of each seed plate 10 near the four corners thereof. The holes 9 being small, shallow and disposed near to the corners of the seed plate 10 do not extend far enough into the plate 10 to interfere with and cause damage or waste in the crystal material that comprises the interior regions of the plate 10 and bar 11 grown therefrom.

As particularly illustrated in Figs. 5 and 6, the ends of each of the supporting wires 8 may be provided with a short sleeve 15 composed of plastic or other suitable resilient material which is inserted in the corresponding hole 9 of the seed plate 10, the sleeve 15 being compressed between the wall of the hole 9 and the outer surface of the wire 8. The sleeves 15 accordingly provide a tight and resilient fit for firmly mounting the

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seed plate 10 on the supporting wires 8 without danger of cracking the seed plate 10.

As illustrated in Figs. 4 to 6, the supporting wires 8 may be carried by U-shaped supports 21, the wires 8 extending horizontally through corresponding openings 17 in the prongs 19 of the U-shaped supports 21, which may be constructed from plastic or other suitable material. The two U-shaped supports 21 may each be secured at an inclined position by means of suitable bolts 23 and pins 25 to the opposite ends of a horizontally disposed radial arm 27, which may be constructed of plastic or other suitable material. The radial arm 27 may be secured at its center by a bolt 29 to a slotted portion in the vertically disposed shaft 31. Other horizontally disposed radial arms similar to the radial arm 27 may be provided at suitable intervals along the entire vertical length of the shaft 31 for the purpose of mounting additional seed plates 10 to the shaft 31 in the manner that the two seed plates 10, as shown in Fig. 4, are mounted and carried by the vertical shaft 31.

Suitable means, as disclosed for example in the A. N. Holden application hereinbefore mentioned, may be provided for supporting and rotating the vertical shaft 31 in a reciprocating rotary motion about its vertical axis, and thereby moving the seed plates 10 of Fig. 4 in contact with the nutrient solution for the purpose of growing each of the seed plates 10 into crystal bars 11 of the type shown in Fig. 3.

As shown in Fig. 3, the growth of the crystal bar 11 consisting in particular of crystalline ammonium dihydrogen phosphate takes place mainly on the two major faces 14 of the seed plate 10 and is in the general direction of the optic axis Z which, in the example illustrated, is disposed at an acute angle θ with respect to the major faces 14 of the seed plate 10. Accordingly, when it is desired that the crystal bar 11 be grown in the vertical direction, the major faces 14 of the seed plate 10 may be mounted at the same angle θ with respect to the vertical direction as illustrated in Fig. 4, so that the growth on the major faces 14 of the seed plates 10 will take place in the vertical direction. For this purpose, the major faces 14 of the seed plates 10 and the planes of the U-shaped crystal supports 21 have been inclined at a suitable angle with respect to the vertical shaft 31, as illustrated in Fig. 4.

While the mounting of the seed plates 10 has been illustrated in Fig. 4 for growing the crystal bars 11 in the vertical direction, it will be understood that the seed plates 10 may be mounted for growing the bars 11 in any other direction by a suitable corresponding position for the mounting of the seed plates 10, and while a particular means has been disclosed for mounting and growing the crystal seed plates 10, it will be understood that any suitable crystallizer and mounting means may be utilized for growing the crystal bars 11 from the seed plates 10.

Although this invention has been described and illustrated in relation to specific arrangements, it is to be understood that it is capable of application in other organizations and is therefore not to be limited to the particular embodiments disclosed.

What is claimed is:

1. A piezoelectric crystal body grown from a seed crystal cut from another crystal, said seed crystal having a face upon which said crystal body is grown, said face of said seed crystal being a face extending substantially parallel to a

grown cap face of said grown crystal, and said face of said seed crystal having a plurality of peripheral edges corresponding in number with and being substantially aligned with the corresponding grown prism faces of said crystal body.

2. A piezoelectric crystal body grown from a seed crystal cut from another crystal, said seed crystal having a face upon which said crystal body is grown, said face of said seed crystal being a face extending substantially parallel to a grown cap face of said grown crystal, and said face of said seed crystal having a plurality of peripheral edges corresponding in number with and being substantially aligned with the corresponding grown prism faces of said crystal body, said grown crystal body being of tetragonal form having four of said prism faces, said grown cap face of said crystal body and said cut face of said seed crystal being substantially plane parallel surfaces.

3. A piezoelectric crystal body grown from a seed crystal cut from another crystal, said seed crystal having a face upon which said crystal body is grown, said face of said seed crystal being a face extending substantially parallel to a grown cap face of said grown crystal, and said face of said seed crystal having a plurality of peripheral edges corresponding in number with and being substantially aligned with the corresponding grown prism faces of said crystal body, said grown crystal body being of tetragonal form having four of said prism faces, said grown cap face of said crystal body and said cut face of said seed crystal being substantially plane parallel surfaces, said grown crystal body and said seed crystal comprising ammonium dihydrogen phosphate.

4. A piezoelectric crystal body grown from a seed crystal plate cut from another crystal, said seed crystal plate having a pair of substantially plane parallel major faces upon which said crystal body is grown, said major faces of said seed crystal plate being faces extending substantially parallel to a grown cap face of said grown crystal body, each of said major faces being of similar shape and having a plurality of peripheral edges corresponding in number with and being in substantial alignment with the corresponding grown prism faces of said grown crystal body.

5. A piezoelectric crystal body grown from a seed crystal plate cut from another crystal, said seed crystal plate having a pair of substantially plane parallel major faces upon which said crystal body is grown, said major faces of said seed crystal plate being faces extending substantially parallel to a grown cap face of said grown crystal body, each of said major faces being of similar shape and having a plurality of peripheral edges corresponding in number with and being in substantial alignment with the corresponding grown prism faces of said grown crystal body, said grown crystal body and said seed crystal plate comprising ammonium dihydrogen phosphate.

6. An artificially grown piezoelectric crystal bar comprising as a part thereof a diagonal seed crystal plate, the prism faces of said crystal bar being grown coaligned with the corresponding outermost peripheral edges of said crystal seed plate, and the two major faces of said crystal seed plate extending substantially to said prism faces being faces cut substantially parallel to a pyramidal cap face, or 101 plane, of said crystal bar, said crystal bar comprising uniformly oriented crystalline material that is substantially free from

flawed regions adjacent said major faces of said seed plate.

7. A piezoelectric crystal grown from a seed crystal plate, said seed plate having two major faces cut parallel to, or in the same crystallographic orientation as, that of a surface which is natural to the growing habit of the crystal and on which crystal growth will take place, and said seed plate being mounted for growing by supports extending into shallow holes drilled into a selected region of said seed crystal plate, said region corresponding to a region where said holes will not damage those portions of said grown crystal bar which are desired for cutting into piezoelectric crystal elements, said region for said holes being in the periphery adjacent the corners of said seed crystal plate, and said supports being wires inserted in said holes.

8. A piezoelectric crystal bar grown from a seed crystal plate cut from another crystal, said seed crystal plate having two major faces, said major faces being faces cut parallel to a surface natural to the growing habit of the crystal and on which crystal growth will take place, said surface and said cut major faces being substantially parallel to a grown cap face of said crystal bar, and means for mounting said seed crystal plate comprising supports extending into holes in said seed crystal plate.

9. A diagonal type seed crystal plate cut from a piezoelectric mother crystal and adapted for growing another piezoelectric crystal in a nutrient solution, said seed plate having a pair of major faces and a plurality of peripheral edge faces extending between said pair of major faces, said major faces being substantially parallel with respect to a 101 plane of said mother crystal, said peripheral edge faces extending in the direction of the Z-axis and comprising the naturally grown prism faces of said mother crystal, said seed plate comprising a part of the initial seed region of said mother crystal.

10. A diagonal type seed crystal plate cut from a piezoelectric mother crystal and adapted for growing another piezoelectric crystal in a nutrient solution, said seed plate having a pair of major faces and a plurality of peripheral edge faces extending between said pair of major faces, said major faces being substantially parallel with respect to a 101 plane of said mother crystal, said peripheral edge faces extending in the direction of the Z-axis and comprising faces cut substantially parallel to the naturally grown prism faces of said mother crystal, said seed plate comprising a part of the initial seed region of said mother crystal.

11. A diagonal type seed crystal plate cut from a mother crystal and adapted for growing another crystal in a nutrient solution, said seed plate having a pair of major faces and a plurality of peripheral edge faces extending between said pair of major faces, said major faces being substantially parallel with respect to a 101 plane of said mother crystal, said peripheral edge faces extending in the direction of the Z-axis and comprising the naturally grown prism faces of said mother crystal, said seed plate comprising a part of the initial seed region of said mother crystal, and comprising one of the crystalline substances ammonium dihydrogen phosphate, potassium dihydrogen phosphate, ammonium dihydrogen arsenate and potassium dihydrogen arsenate.

12. A diagonal type seed crystal plate cut from a mother crystal and adapted for growing another crystal in a nutrient solution, said seed

plate having a pair of major faces and a plurality of peripheral edge faces extending between said pair of major faces, said major faces being substantially parallel with respect to a 101 plane of said mother crystal, said peripheral edge faces extending in the direction of the Z-axis and comprising faces cut substantially parallel to the naturally grown prism faces of said mother crystal, said seed plate comprising a part of the initial seed region of said mother crystal, and comprising one of the crystalline substances ammonium dihydrogen phosphate, potassium dihydrogen phosphate, ammonium dihydrogen arsenate and potassium dihydrogen arsenate.

13. A seed crystal plate cut from a tetragonal mother crystal, said seed plate having its four edge faces substantially parallel to the Z-axis and its two major faces substantially parallel to a naturally grown pyramidal cap face of said mother crystal, said seed plate comprising a part of the initial seed region of said mother crystal.

14. A seed crystal plate cut from a grown tetragonal mother crystal, said seed plate having its four edge faces substantially parallel to the Z-axis and its two major faces substantially parallel to a grown pyramidal cap face of said mother crystal, said seed plate comprising a part of the initial seed region of said mother crystal and said seed plate comprising crystalline ammonium dihydrogen phosphate.

15. A crystalline seed plate having two major faces adapted for growing a tetragonal crystal bar from at least one of said two major faces in the direction of the Z-axis, said seed plate having at least a part of its peripheral edges lying in each of the four prism faces of said crystal bar to be grown therefrom, said seed plate having its said two major faces disposed substantially parallel to a grown pyramidal cap face of the mother crystal from which it is cut, said seed plate comprising at least a part of the initial seed region of said mother crystal.

16. A crystalline seed plate having two major faces adapted for growing a tetragonal crystal

bar from at least one of said two major faces in the direction of the Z-axis, said seed plate having at least a part of its peripheral edge lying in each of the four prism faces of said crystal bar to be grown therefrom, said seed plate having its said two major faces disposed substantially parallel to a grown pyramidal cap face of the mother crystal from which it is cut, said seed plate comprising at least a part of the initial seed region of said mother crystal, said seed plate comprising crystalline ammonium dihydrogen phosphate.

17. A crystalline ammonium dihydrogen phosphate seed crystal plate cut from a mother crystal, said seed plate having its major faces substantially parallel to the plane of one of the grown pyramidal cap faces or a 101 plane of said mother crystal, said seed plate comprising at least a part of the initial seed region of said mother crystal.

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