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 APPARATUS FOR GROWING CRYSTALS  
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2,907,643

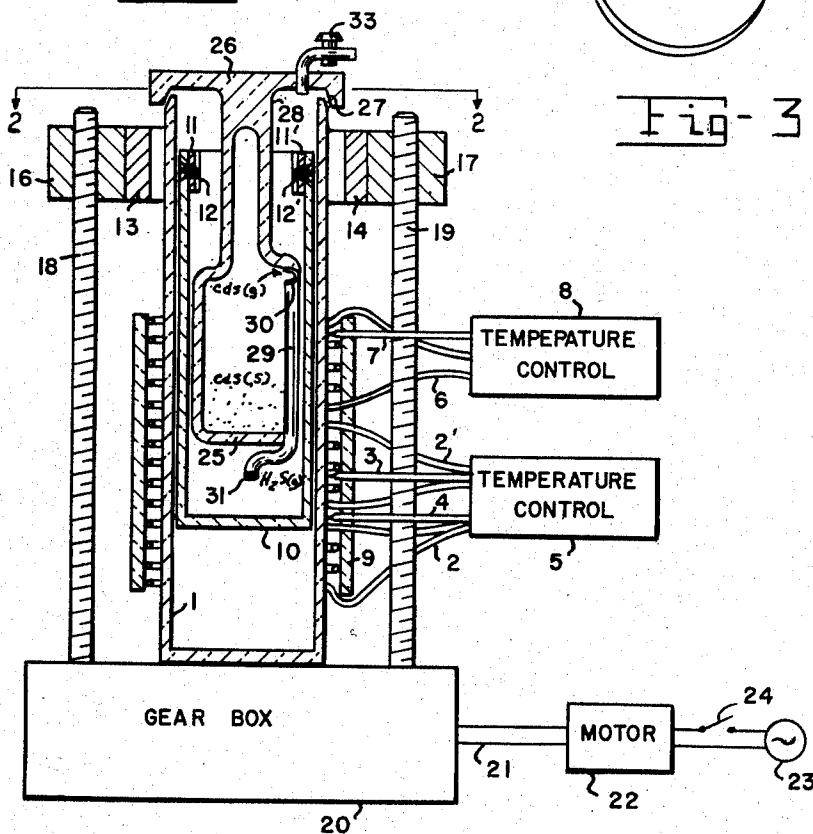
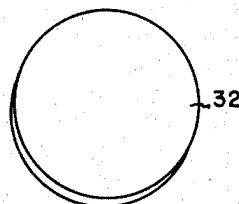
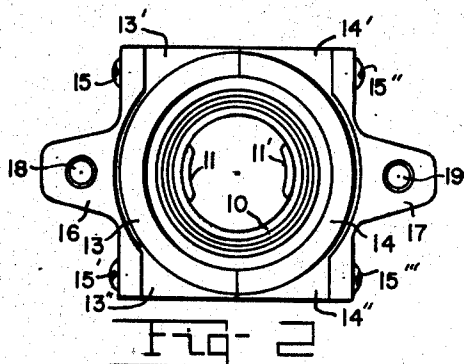


Fig-1

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## APPARATUS FOR GROWING CRYSTALS

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2 Claims. (Cl. 23—273)

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

The invention described herein may be manufactured and used by or for the United States Government for governmental purposes without the payment to us of any royalties thereon.

This invention relates to a means and a method for accomplishing crystal growth.

This invention was originally disclosed in application Serial Number 572,170, filed March 16, 1956, which is restricted to the disclosed method for accomplishing crystal growth. This application is a division of the above application and is restricted to the disclosed apparatus.

As a background for making the present invention as it is presented and claimed herein clearly understandable, a chemical element or a chemical compound has an energy content which commonly causes it to exist in three states. At low temperatures it may be solid, with the addition of heat it may liquefy and with the addition of more heat it may become vapor. Some materials sublime from the solid state directly to the gaseous phase under favorable pressure conditions. In an environment of unimpeded orientation an element or a compound may arrange itself in a crystalline form with a space lattice characteristic of that element or compound.

A general statement of the exact nature and of the substance or the subject matter of the present invention consistent with the claims which are appended hereto and together with the operation and the purpose of the invention, is that the exact nature or the essential character or constitution of the present invention is the production in the field of solid state physics of a crystal, such as cadmium sulfide, zinc sulfide and their equivalents. The substance or the subject matter of the present invention is an apparatus for making a crystal of a desired size and composition by its solidification from its gaseous state and a furnace comprising a plurality of temperature zones within which a closed bottom quartz cup receives the crystal as a solid from a retort tube orifice. The retort tube opens into the upper part of a quartz retort or tank containing the material from which a crystal is to be made. The material is in both its solid and gaseous states within the retort. The retort or tank is supported from the top of the quartz cup. An atmosphere of hydrogen sulfide at an illustrative pressure of from a one-fourth to a one-half atmosphere is maintained within the quartz cup within which the crystal forms.

A general object of the present invention is to provide a new and an improved apparatus for making a crystal of a desired size by a vaporization process.

A further object is to provide a new and an improved furnace for use in the field of solid state physics and the like.

Another object is to provide a crystal of cadmium sulfide, zinc sulfide or their equivalents.

In the accompanying drawing:

Fig. 1 is an elevational view partly in section, partly

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broken away and partly diagrammatic of a furnace embodying the present invention;

Fig. 2 is a plan view from above of the cup and floating cup with the retort removed and taken about from the line 2—2 of Fig. 1; and

Fig. 3 is a perspective enlarged view of a crystal made in the furnace shown in Fig. 1, in the practice of the method of the present invention.

The furnace shown in Fig. 1 of the accompanying drawing comprises illustratively an upwardly opening quartz cup 1, or hollow cylindrical tube with a closed bottom, which is positioned preferably within a multiple winding or a multiple heating element furnace. The furnace illustratively may comprise a first pair of windings 2 and 2', each of which is maintained at a temperature which is controlled by its own thermocouple 3 or 4 from a temperature control device 5, and a second winding 6, which is maintained at a temperature by operation of a thermocouple 7 and another temperature control device 8. The first pair of windings 2 and 2' illustratively may maintain a temperature slightly below the sublimation temperature of cadmium sulfide, whereas the third winding 6 maintains a temperature slightly above the sublimation temperature of the solid to be vaporized. The furnace is provided in the usual manner with an insulant 9 against objectionable heat loss with the insulant positioned outwardly of the windings 2 and 6.

A second upwardly opening quartz cup 10, which is a hollow cylinder with a closed bottom, is mounted to float axially and adjustably within the cup 1. Illustratively a soft iron ring, or a plurality of soft iron slugs 11, 11', etc., may be secured to the floating cup 10 of a desired size by screws 12, 12', etc. within a suitable magnetic field, such as that maintained by a pair of horseshoe magnets 13 and 14 with their like poles joined, as by being welded together or the like. The magnets 13 and 14 carry pairs of bosses 13', 13'' and 14', 14'' which are tapped and threaded to receive securing screws 15, 15', etc. which attach the horseshoe magnets to end members 16 and 17 through which yoke supporting screws 18 and 19 thread, respectively, throughout. The screws 18 and 19 are rotated simultaneously to raise or to lower the horseshoe yoke by operation of a gear train within a gear box 20. The gear train is actuated through a shaft 21 by a reversible motor 22 which may be energized from a potential source 23 upon the closing of a switch 24.

A retort 25, or tank, is positioned immovably within the floating cup 10. In the assembly shown, the retort 25 is supported by a cap 26 which makes substantially a gas tight seal with the upper edge of the outer cup 1 by means of a ground glass joint 27 or the like. Suitable means, such as a stop cock or valve 33 is provided for evacuating and then introducing hydrogen sulfide gas into the interior of the floating cup 10. The retort 25 may be charged with a material from which a crystal of a desired size is to be made, such as cadmium sulfide, zinc sulfide or an equivalent, preferably in powdered solid state. The retort may then be sealed by the use of heat and connected through a rod portion 28 with the cap 26 and installed in place.

The retort 25 is positioned to exhaust downwardly centrally of the bottom of the floating cup 10, as through a tube 29 or the like. The attached end 30 of the tube 29 opens into and receives its gaseous input from the upper part of the retort 25, as represented in the drawing. The orifice 31 in the unattached end of the tube 29, preferably is disposed centrally of the bottom of the floating cup 10.

The proximity of the output orifice 31 in the unattached end of the tube 29, to the inner surface of the bottom of the floating cup 10 is closely adjustable. This ad-

justment is accomplished by operation of the reversible motor 22 which rotates the screws 18 and 19 to adjust the yoke carrying the magnets 13 and 14. The iron slugs 12 and 12' follow the position of the magnets 13 and 14 and carry the floating cup 10.

An illustrative cadmium sulfide solid crystal 32 made by the disclosed method with the equipment shown in Figs. 1 and 2 of the drawing is represented in enlarged dimensions in Fig. 3 of the drawing.

A preferred method by which the crystals contemplated hereby are made comprises the use of the above described equipment. Prior to the assembly of the tank or retort 25 with the stem 28 of the cap 26, a desired quantity of solid, granular cadmium sulfide, or other material from which a crystal is to be made and of a desired degree of purity, is charged into the tank or retort 25 through its neck opening. The neck opening of the retort 25 is then closed by union with the cap stem 28, using heat to accomplish the seal. The assembly may be evacuated then charged with hydrogen sulfide through the stop cock 33, or the charged retort 25, together with cups 1 and 10 unassembled, may be placed in an outside container, not shown, where they may be evacuated, charged, where a sulfide crystal is in process of manufacture, with hydrogen sulfide gas of about 15 inches of mercury pressure and in that atmosphere the capsule 25 is inserted into the tube or cup 10 and the cap 26 caused to make a gas-tight union with the open top of the outer quartz cup 1. The cup 1, sealed by the cap 26, is then inserted through the yoke of the magnets 13 and 14 and is positioned within the furnace, consisting of the windings 2 and 6 or of comparable construction. The retort discharge orifice 31 is spaced a desired distance from the inner surface of the bottom of the cup 10, as determined by the position of the magnet yoke controlled by the pair of screws 18 and 19 and the motor 22.

The furnace is then brought to its operating temperature, with the first windings 2 and 2' slightly below the sublimation temperature of cadmium sulfide, or other desired material, and the second winding 6 slightly above the material sublimation temperature, which for cadmium sulfide is in the neighborhood of 1000° C. The furnace temperature will vary with both the material composition and with its degree of purity. Experimentally crystals have been grown in from 24 to 168 hours. The furnace illustratively may be maintained at its operating temperature for about a week or more in the building of crystals of usable size during which time the separation between the retort discharge orifice 31 and the bottom of the cup 10 may be increased to accommodate the increasing size of the growing crystal.

The crystals of cadmium sulfide grown by this disclosed method have been of a general hexagonal prismatic shape. Cadmium sulfide is an excellent photoconductor. Chemically pure cadmium sulfide has only one peak occurring at the absorption cutoff, with very rapid rise and decay of the photocurrent on either side of the peak.

It is to be understood that the method and the apparatus which are disclosed herein have been submitted for the purposes of explaining and illustrating an operative embodiment of the present invention and that similarly functioning method steps and apparatus arrangements may be made without departing from the spirit of the present invention.

What we claim is:

1. An apparatus for growing crystals comprising an upwardly open outer cup of quartz, an axially adjustably movable inner quartz cup positioned within the outer cup to be freely adjustable linearly thereof within limits and having a bottom surface upon which a crystal is to be grown and having an iron slug attached to the inner cup, a retort within the inner cup depending from a retort cup portion sealing the open upper end of the outer cup,

a quartz tube attached to the retort with a tube intake orifice opening into the retort and the quartz tube discharging downwardly through an orifice the proximity of which to the bottom of the inner cup may be adjustably regulated by the adjustment of the inner cup with respect to and axially of the outer cup, valve means for controlling the evacuation of the outer cup and the introduction of a gas atmosphere into the outer cup carried by the cup portion of the retort serving as a means supporting the retort in immovable relation with respect to the outer cup, magnet means having a magnetic field within which the iron slugs carried by the inner cup are positioned, yoke means carrying the magnetic means axially and externally of the outer cup, screw threaded means for adjusting the yoke means axially of the outer cup, and furnace means provided with a plurality of windings of a plurality of temperature gradients controlling the temperatures within the retort as distinct from the temperature within the bottom of the inner cup and the physical state of the contents thereof.

2. An apparatus for growing crystals comprising a quartz retort provided with a cap terminating laterally in a downwardly and outwardly flared ground glass joint face and the cap apertured for a gas connection and the retort provided with a quartz tube with an upper intake end opening into substantially the upper part of the retort and the quartz tube extending axially of the retort and having a discharge end below the lower end of the retort, a floating cup with a closed bottom and an open top with an iron slug secured adjacent to the floating cup top upper end and the floating cup containing the retort and the floating cup being adjustable axially of the retort for the purpose of adjusting the proximity of the inner surface of the bottom of the floating cup below the discharge end of the quartz tube that extends axially of the retort for performing the function of delivering gas conducted by the quartz tube out of the retort and against the inner surface of the bottom of the floating cup, an outer cup with a closed bottom and an open top providing at its lateral outer edge an upwardly and inwardly flared ground glass joint face for engaging in sealing relation the downwardly and outwardly flared ground glass joint face on the quartz retort cap and the outer cup so closed housing the floating cup and its contents, a multiple heating element furnace disposed outwardly of the outer cup and with a plurality of winding between the outer surface of the outer cup and an insulant that restrains heat energy to the proximity of the outer cup, a first temperature control maintaining a first furnace winding at a potential supplying heat for a vapor state at the upper intake end opening of the quartz tube in the upper part of the retort, a second temperature control maintaining a second furnace winding at a potential supplying a temperature for a solid state below the discharge end opening of the quartz tube below the closed bottom of the retort and inside of the floating cup, a magnet adjustably positioned outside the outer cup and providing a magnetic field around the iron slug at the top of the floating cup, a yoke supporting the magnet outside the outer cup for movement axially thereof in controlling the position of the floating cup relative to the retort, a yoke supporting screw that threads through the yoke and adjusts the position of the yoke and magnet axially of the outer cup, a gear box containing a mechanism for reversibly rotating the yoke supporting screw, and a motor operating the gear box.

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