

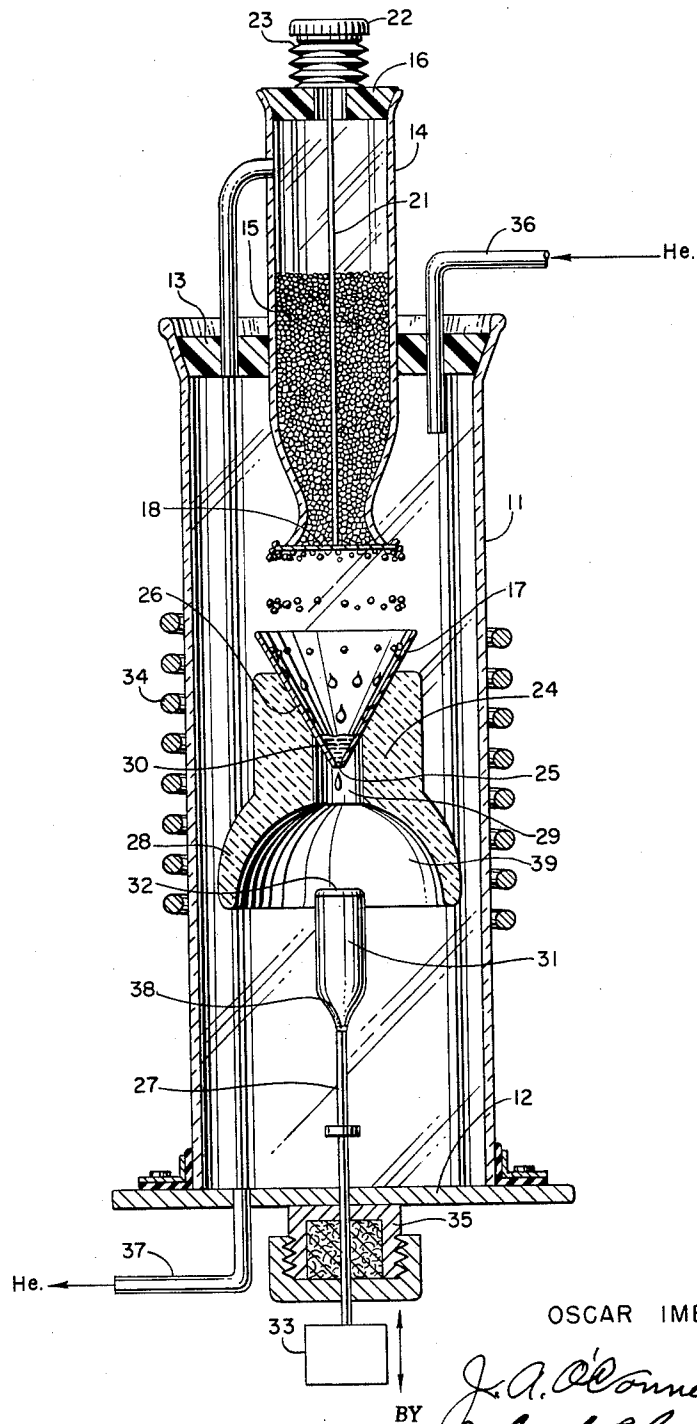
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CRYSTAL GROWTH APPARATUS

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CRYSTAL GROWTH APPARATUS

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

This application is a continuation-in-part of application Serial No. 693,496, filed October 30, 1957, and now abandoned, for Crystal Growth Apparatus.

The present invention relates generally to a crystal growing apparatus and more particularly to an improved apparatus for flameless fusion growth of single crystals wherein the molten material is maintained in minimum amounts during the crystal growth.

The flameless fusion technique has been found most useful in growing crystals, which, because of their chemical properties, cannot meet the rigorous requirements of flame fusion and exposure to reducing atmospheres, hence crystal growths are produced by melting finely divided particles and successively adding molten droplets to a growing crystal boule in an inert atmosphere or in a vacuum. Although crystals such as germanium and silicon have been produced by this method it has been the general practice to provide a melt in a relatively large quantity, for prolonged periods, and often at temperatures which affect crucible materials. The introduction of undesirable impurities from the walls of a crucible and the drastic conditions of the molten state often affect the stability of the melt and lead to unpredictable results. Previous devices have been designed which maintain the crystallizable material as a powder and which meter said powder through a heated zone to the growing crystal, but the solid and semi-solid particles which reach the region of crystal growth require additional heating to establish a molten state on the crystal surface.

The general purpose of this invention is to provide an improved crystal growing apparatus to overcome the disadvantages described above by minimizing both the quantity and duration of the molten material within the crucible.

It is also an object of the invention to provide an improved crystal growing apparatus which supplies a molten nutrient that is free of solid particles or impurities.

It is another object of the invention to provide an improvement in a crystal growing apparatus wherein the crucible is readily interchangeable to meet various conditions of crystal growth and to increase the versatility of the apparatus for growing various types of crystals.

A further object is to provide an improved apparatus for the flameless fusion technique which is easier to operate resulting in improved yields of usable products.

Other objects and advantages of the invention will become more fully apparent in the following detailed description with reference to the accompanying drawing, wherein:

The figure is a vertical cross-section view of the apparatus illustrative of the invention.

Broadly stated the invention refers to an improvement in an apparatus for growing crystals which provides a conical crucible supported within a cylindrical chamber, and a powder dispensing means, or hopper suspended in the upper part of the chamber directly above the conically-shaped crucible. A discharge opening in the apex of the conically-shaped crucible communicates with the lower part of the chamber where a seed crystal is supported directly beneath the opening. The conical member is read-

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ily interchangeable thus providing crucible structures of various degrees of inclination, composition and discharge opening. In operating the device, powder spills from the hopper onto the hot conical crucible where the powder melts and collects in a molten pool over the discharge opening of the cone. Drops form and fall through the opening to the crystal grown below.

An important feature of the present device resides in the crucible structure; conical structures in the past have been merely funneling devices. In the present structure the powder which falls into the conical crucible is melted and retained therein in a relatively small amount. The crucible opening at the apex has a diameter size which prevents the flow of melt until a sufficient amount has accumulated therein to allow droplets to form and fall through said opening. It has been found that a discharge opening of from about 0.5 mm. to about 1.1 mm. is sufficient to prevent initial flow of melt in the absence of sufficient hydrostatic pressure.

Referring now to the drawing, a cylindrical quartz jacket 11 is mounted on a base plate 12 which provides a closure means at the base of the cylinder and supporting means for the entire apparatus. A detachable cover plate 13 provides sealing means at the top of the cylinder and also a support for the powder dispensing means or hopper 14 which is adapted to receive a powder 15 through cover plate 16. A taper arrangement in the hopper is designed to spill powder into a conically shaped crucible 17. The taper arrangement includes a movable plate 18 positioned across a relatively wide mouth opening at the base of the hopper and connected by a rod 21 to an anvil plate 22. A spring type bellows positioned between the anvil plate 22 and cover plate 16 urges the movable plate 18 tightly against the mouth of the hopper by means of rod 21. The powder 15 spills from the mouth around the outer edge of the movable plate 18 whenever the anvil plate 22 is tapped or vibrated while bellows 23 acts to stop the downward movement of the taper by restoring plate 18 to its original position after each tap. The conical crucible 17 is positioned within the jacket directly below the hopper and rests upon an annular carbon ring 24. The ring has a center opening 29 and a slanting surface 26 on which the conical crucible rests and forms a close alignment therewith while the apex portion of the crucible descends into the central opening 29. The lower portion 28 of said annular ring is flared out radially forming a centrally disposed dome space 39 into which the central opening 29 emerges. A support rod 27 extends vertically through base plate 12 to provide supporting means for seed crystals 38 on which is grown the crystal boule 31. The support rod is positioned directly below and in line with opening 25 and is adjustably mounted to be raised and lowered by adjustable means 33 of any well known type. An inlet gas tube 36 at the upper end of the cylinder and an outlet gas tube 37 at the lower end thereof allow for an inert gas to circulate within the cylinder space. Sealing means 35 positioned around the movable support rod, below the base plate prevents escape of the enclosed gas.

An induction coil 34, surrounding the cylindrical jacket at the approximate location of the annular carbon ring within the cylinder provides for induction heating of the carbon ring.

In growing crystals of germanium, silicon or of alkali metal halides the, conditions of crystal growth may be varied depending upon the composition of the melt, the inert dry atmosphere which is employed, the particular opening at the apex of the cone and the material of which the cone is made. The conical member therefore, which is provided in the present apparatus, is made interchangeable and the crucible composition, opening at the apex and conical angle may be varied to meet the requirements

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of various conditions for crystal growth. For example, silicon crystal growth requires a cone made of silica composition having a discharge opening at the apex of the cone in the range of about 0.7 mm. to about 1.1 mm. in diameter. In utilizing the same apparatus for germanium crystal growth, it has been found necessary to employ a cone structure made of carbon; the opening at the apex is preferably in the range of about 0.5 mm. to about 0.8 mm. in diameter. Again when the apparatus is converted for alkali metal halides, the crystals are grown by providing a platinum crucible; the melt of the halides is considerably more fluid, thus, the conical crucibles have discharge openings of about 0.5 mm. in diameter.

In growing crystals by means of the present apparatus, the powder material must have a high degree of purity, although the particle size thereof is not very critical. Most satisfactory results are obtained from particles somewhere in the order of about 50 to 80 standard mesh. Variations in the particle size will not interrupt crystal growth, since the particles melt in the cone region and only droplets of melt of uniform size will fall through opening 25 in uniform droplet size. The opening, therefore, in the apex of the conical crucible is of a certain dimension, such that the molten substance will be retained within the cone, the surface tension of the liquid preventing any initial flow. When the melt has built up sufficiently above the discharge opening, the hydrostatic pressure of the melt increases to the point where it exceeds the surface tension of the melt, a droplet forms and falls through the opening to the crystal surface below. Discharge openings in the range of from about 0.5 millimeter to about 1.1 millimeters have been found to operate essentially in this manner.

In addition to controlling and minimizing the melt present in the cone, the conical hearth is especially useful for retaining any impurities that may be present in the melt. Impurities appear in the form of "slag" which float in the melt and are thus retained in the conical vessel near the surface of the melt.

In operating the apparatus, a seed crystal 38 is positioned initially on the tip of support rod 27, and powder material suitable for crystal growth is placed in the hopper. The induction coil 34 is connected to an electric source and the annular ring 24 becomes inductively heated, and the conical crucible 17 is, in turn, raised in temperature sufficient to melt the powder. Any suitable mechanical means may be rigged to vibrate or tap the anvil plate 22 whereby a powder, such as germanium or silicon, falls in small increments onto the heated crucible 17 when the anvil plate of the tapper arrangement is vibrated or tapped. The mouth of the hopper is broad enough so that the powder that spills from movable plate 18 falls near the base of the conical surface, that is, away from the apex region of the cone. The powder, after it has contacted the heated surface, melts to form droplets that roll toward the apex to form a molten mass 30 above opening 25. The molten mass forms droplets at the opening when the melt has sufficient weight to overcome its surface tension. Droplets will form and fall through opening 25 to the molten surface 32 on crystal boule 31.

The support rod on which the crystal is grown is moved vertically within the dome space near the heat zone, and it is initially adjusted to bring the molten portion of boule 32 to the desired heat zone in the dome space. Once the optimum temperature for crystal growth is obtained, the support rod is lowered slowly as the crystal boule is growing to keep the molten part 32 at approximately the same level within the dome. The heat surrounding the crystallizing surface remains constant during the entire operation.

Inlet 36 and outlet 37 at the top and bottom of the cylinder, respectively, provide means through which helium gas or some other inert gas is circulated about the heat zone and the cooling portion of the crystal. The presence of an inert gas prevents oxidation interference

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during the crystal growth and minimizes contamination of the growing crystal. Although the apparatus is shown operating with inert gas, it is of course understood that the apparatus can be readily converted to vacuum operation.

Obviously many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practised otherwise than as specifically described.

What is claimed:

1. A crystal growth apparatus comprising:

- (a) a chamber having a vertical axis,
- (b) a conical container having an open upper base, said base being in open communication with said chamber, an inner lateral surface and a suitable opening at the apex thereof,
- (c) said opening having a dimension approximately in the range of about 0.5 mm. to about 1.1 mm. to permit gravity flow under a hydrostatic head,
- (d) means arranged above said container for periodically discharging powder on said surface,
- (e) heating means provided at the periphery of said container, for melting the powder contacting said surface,
- (f) a support rod below said opening adapted to support a crystal growth element thereon,
- (g) said conical container, said means for discharging powder and said support rod being supported in said chamber and arranged in substantially coaxial relationship to said axis,
- (h) means coupled with said chamber for moving said rod downward away from said opening, and
- (i) means connected to said chamber for circulating an inert atmosphere therethrough.

2. A crystal growth apparatus comprising:

- (a) a chamber having a vertical axis,
- (b) a conical crucible having an open upper base, said base being in open communication with said chamber, an inner lateral surface and a suitable opening at the apex thereof,
- (c) said opening having a dimension approximately in the range of about 0.5 mm. to about 1.1 mm. to permit gravity flow under a hydrostatic head,
- (d) a hopper arranged above said crucible and adapted to discharge powder periodically on said surface,
- (e) a carbon annulus contacting said crucible over a considerable area thereof and adapted to be inductively heated to convert the powder contacting said surface into a melt,
- (f) a support rod below said opening adapted to support a crystal growth element thereon,
- (g) said crucible, said hopper and said support rod being supported in said chamber and arranged in substantially coaxial relationship to said axis,
- (h) means coupled with said chamber for moving said rod downward away from said opening, and
- (i) means connected to said chamber for circulating an inert atmosphere therethrough.

3. A crystal growth apparatus comprising:

- (a) a chamber having a vertical axis,
- (b) a conical crucible having an open upper base, said base being in open communication with said chamber, an inner lateral surface and a suitable opening at the apex thereof,
- (c) said opening having a dimension approximately in the range of about 0.5 mm. to about 1.1 mm. to permit gravity flow under a hydrostatic head,
- (d) a hopper arranged above said crucible, said hopper having a wide-mouth opening at the base thereof, a movable cover plate over said wide-mouth opening and means for moving said plate periodically to discharge powder circumferentially from said wide-mouth opening to said surface,

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- (e) a support rod below said opening adapted to support a crystal growth element thereon,
 (f) an inductively heated annulus having an upper surface in contact with said crucible over a considerable area thereof and a lower spherical cavity for heating the area of said rod,
 (g) said crucible, said hopper and said support rod being supported in said chamber and arranged in substantially coaxial relationship to said axis,
 (h) means coupled with said chamber for moving said rod downward away from said opening, and

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- (i) means connected to said chamber for circulating an inert atmosphere therethrough.

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