Feb. 10, 1970 C. W. HANKS ETAL 3,494,804

METHOD FOR GROWING CRYSTALS

Filed July 15, 1968 3 Sheets-Sheet 1

FIG. 1.

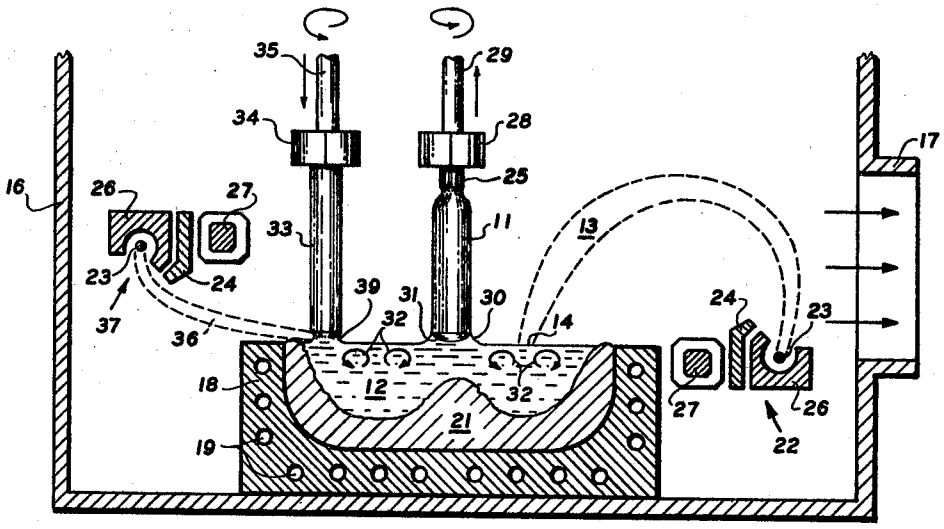
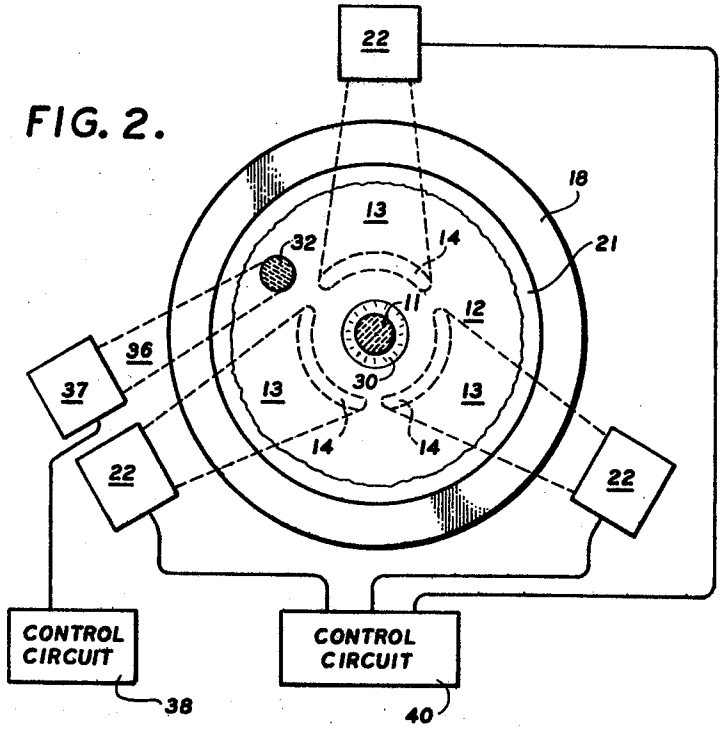


FIG. 2.



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FIG. 3.

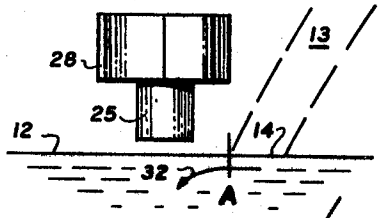


FIG. 4.

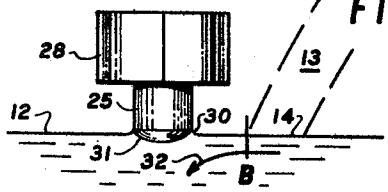


FIG. 5.

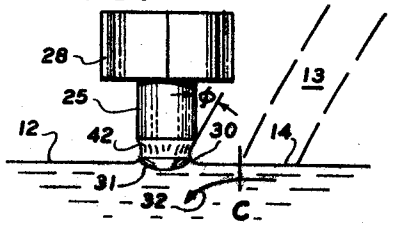


FIG. 6.

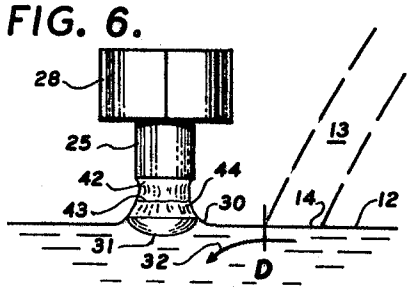


FIG. 8.

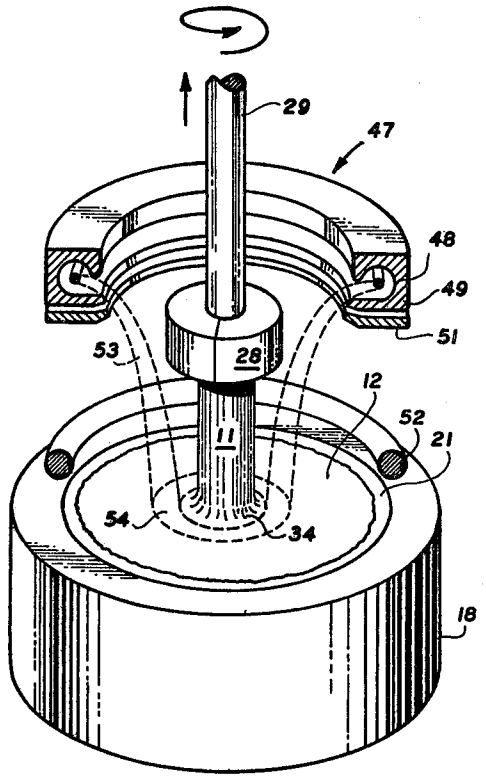
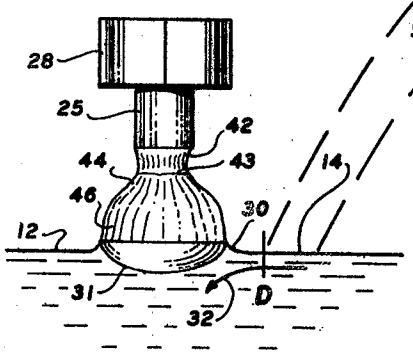


FIG. 7.



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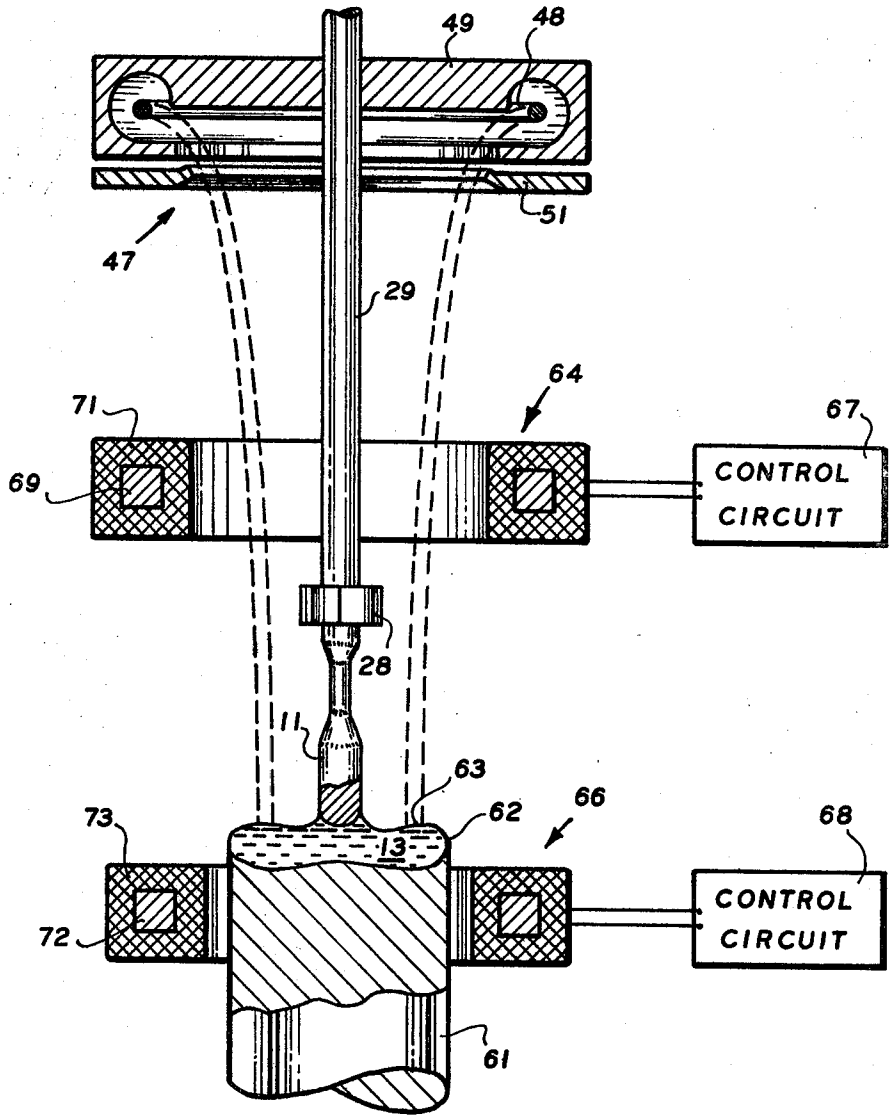


FIG. 9.

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METHOD FOR GROWING CRYSTALS

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Continuation-in-part of application Ser. No. 659,175, Aug. 8, 1967. This application July 15, 1968, Ser. No. 744,874

Int. Cl. B01j 17/06

U.S. Cl. 148—1.6

11 Claims

ABSTRACT OF THE DISCLOSURE

A method is described for growing a single crystal from a molten pool formed of crystalline material, such as silicon and super alloy metals. Growth of the single crystal is controlled by controlling the thermal pattern, in the molten pool from which the product is grown, through variation in the position of electron beams directed at the pool surface.

This invention relates to single crystals and, more particularly, to an improved method for growing such crystals from a molten pool, and is a continuation-in-part of application Ser. No. 659,175, now abandoned.

Certain articles of manufacture utilize material produced in single crystal form. For example, many types of electronic circuit elements, such as transistors and diodes, are manufactured from thin slices of semi-conductor material produced in single crystal form. In addition, some items which are subjected to high stresses and high temperatures, such as turbine blades, may be constructed of certain super alloy metals in single crystal form.

One general technique which has heretofore been developed for producing single crystals, from which various items mentioned above may be manufactured, involves the drawing or growing of a generally cylindrical single crystal from a molten pool. In the growing technique, a single crystal seed is dipped into a molten pool so that an interface is formed between the seed and the molten pool. The seed is then withdrawn from the pool in a manner which causes the molten material at the interface to solidify continuously as the seed is drawn upwardly. The precise way in which this is accomplished may vary considerably, frequently depending on environmental conditions and other factors.

Although satisfactory in some respects, heretofore known ways of accomplishing crystal growing often tend to be expensive and difficult to carry out. Coating flakes, condensate and dirt on the seed surface, etc., can cause nucleation and growth of other crystals, destroying the single crystal nature of the final product. Disturbing conditions at the original surface of the seed may result in poor surface quality in the crystal being grown. Surface imperfections, and surface nucleation agents can be caused to "grow out" of the surface by reducing the diameter of the crystal at an angle of fresh surface which is greater than about 30° from the crystal axis. To do this may be difficult, however, for the reason that it is usually necessary to precisely vary the temperature at the interface and, in most known systems, temperature has a very slow response.

Other difficulties may be encountered during crystal growing. Variation in environmental conditions may have

a deleterious effect on quality of the single crystal by producing non-symmetrical intracrystalline growth and consequent dislocations. Difficulties in achieving a desired crystal size, and diameter, and the introduction of wanted and unwanted impurities, may also present problems in heretofore known ways of crystal growing.

Accordingly, it is an object of this invention to provide an improved method for producing a single crystal.

Another object of the invention is to provide a method for producing a single crystal by which a high degree of consistency and quality in results may be obtained.

It is another object of the invention to provide a method for growing a single crystal from a seed, which minimizes the effect of crystal nucleation agents that may be residing on the surface of the seed, and which allows the reproduction of the subsurface quality of the seed.

A further object of the invention is to provide a method for growing a single crystal wherein the cross sectional size of the single crystal during growth may be readily varied.

Other objects of the invention will become apparent from the following description taken in connection with the accompanying drawings wherein:

FIGURE 1 is a schematic sectional view of apparatus for practising the method of the invention;

FIGURE 2 is a top plan schematic view of part of the apparatus of FIGURE 1;

FIGURES 3 through 7 are schematic side elevational views illustrating successive steps of one specific way of practicing the method of the invention;

FIGURE 8 is a schematic perspective view of an alternative type of apparatus for practicing the invention; and

FIGURE 9 is a schematic full section view of a further alternative type of apparatus for practicing the invention.

Very generally, the invention operates to grow a single crystal **11** from crystalline material formed into a molten pool **12**. The pool is heated by bombarding its surface with at least one electron beam **13**. A single crystal seed **25** is dipped into and drawn from the molten pool to form the single crystal and the cross sectional size of the single crystal being grown is controlled by controlling the position of the areas **14** of beam impact on the pool surface, relative to the single crystal.

Referring now more particularly to FIGURES 1 and 2, the apparatus used in practicing the method of the invention will be described in detail. The apparatus illustrated in FIGURES 1 and 2 is for growing a single silicon crystal of generally circular cross section, such as is used in the manufacture of certain types of transistors and diodes. The single silicon crystal **11** is produced in an electron beam furnace having a vacuum tight enclosure **16**. The region inside the enclosure is evacuated, through a duct **17** in a wall of the enclosure, by a suitable vacuum pump, not illustrated. The pressure inside the enclosure **16** is preferably reduced to less than one torr.

A crucible **18**, preferably comprised of stainless steel, is supported within the enclosure **16**. The crucible contains the molten pool **12** from which the single silicon crystal **11** is grown. Coolant passages **19** are provided in the walls of the crucible **18**, and a suitable coolant, such as water, is circulated therethrough. Accordingly, a skull **21** of solidified silicon forms between the walls of the crucible **18** and the molten pool **12**. This skull prevents any interaction between the crucible material and the molten silicon, and it insures that the molten silicon will have a high degree of purity.

The molten pool **12** is heated by bombarding its sur-

face with three electron beams **13**. The electron beams are produced in three electron beam guns **22**, respectively. Only one of the electron beam guns **22** is illustrated in FIGURE 1 in order to present a less confused appearance. The three electron beam guns **22** illustrated in FIGURE 2 are shown in block form. In the illustrated apparatus, the beams are shaped or swept to produce an almost continuous ring-shaped or annular impact area as shown in FIGURE 2. The inner diameter of the annular impact area is greater than that of the crystal being pulled, and the outer diameter is less than that of the pool. Preferably, the difference in outer and inner diameters is kept as small as possible consistent with providing sufficient energy to the pool.

The electron beam guns **22** may be of any type known in the art by which the beam position may be varied with respect to the single crystal **11**, however, the details of one type of electron gun which may be used are illustrated in FIGURE 1. The electron guns **22** are disposed within the enclosure **16** and each includes a directly heated cathode **23** and an accelerating anode **24**. The electrons emitted by the cathode **23** are directed into a beam by a shaping electrode **26** and the beam thus formed is accelerated by the accelerating anode **24**. To this end, the cathode **23** and the shaping electrode **26** are maintained at a negative potential with respect to the anode **24**. A transverse field, established by an appropriately positioned electromagnet **27**, is used to deflect the electron beam onto the surface of the material in the molten pool **12**. Suitable means, not shown, are provided for heating the cathode and for maintaining the desired potentials on the described elements.

The single crystal **11** is formed by drawing it upwardly out of the molten pool **12**. The growth of the crystal is initiated by immersing a suitable single crystal seed **25** in the pool and beginning a slow upward withdrawal. The seed is held in a suitable clamp **28**, and an actuating rod **29** is secured to the clamp **28**. A motor driven mechanism, not illustrated, is used to rotate the rod **29** and hence the single crystal **11** while it is being drawn upwardly. The crucible **18** may also be rotated in the opposite direction by suitable means, not illustrated. A meniscus **30** forms between the single crystal **11** and the surface of the molten pool **12** during the growing operation.

When the single crystal **11** is being drawn upwardly, the interface **31**, between the solid crystal and the molten pool **12**, assumes a generally spherical shape. As the crystal is drawn upwardly, the molten silicon freedes at the interface with the desired monocrystalline structure. A significant factor which affects the manner in which the silicon solidifies is the distribution of temperature in the molten material near the interface. Various techniques are known in the art for regulating the temperature distribution by regulating the temperature of the molten pool. Previous techniques, however, have often been hampered by a slow response time insofar as regulation of pool temperature is concerned. This makes it much more difficult to achieve satisfactory monocrystalline growth.

In practicing the method of the invention, the average temperature of the molten pool **12** is maintained somewhat above the melting point of the silicon. The temperature in and near the impact areas of the beams, however, is substantially higher than the average temperature of the pool. As a result, the electron beam **13** causes a region of turbulence adjacent its surface impact area because of the localized production of heat energy at the surface. This turbulence is characterized by an outward flow of superheated molten material at and near the pool surface from the regiion of highest heat toward regions of lower heat. A return flow of cooler material inwardly and then upwardly occurs at a lower depth in the molten pool. The flow of turbulence is indicated by the arrows **32** in FIGURE 1, and this turbulence region is generally annular, extending around the crystal being pulled in accordance with the shape of the corresponding beam impact pattern. The velocity of the molten material in the turbulence region generally decreases with increasing distance from the beam impact area.

When the electron beam **13** is moved closer to the crystal **11**, the velocity and temperature of the flow of molten material adjacent the interface **31** is correspondingly increased. This causes a reduction in the diameter of the crystal being pulled due to the increased temperature and washing action of the superheated molten material. Conversely, when the beam **13** is moved away from the crystal **11**, the velocity and temperature of the flow of molten material at the interface **31** is reduced, resulting in an increase in the diameter of the crystal being pulled. By regulating the position of the region of turbulence represented by the arrows **32** (i.e., by moving the beam toward or away from the crystal **11**) the flow of molten material at the interface **31** may be regulated as to velocity and temperature to achieve a desired crystal diameter.

The position of the beams as they are used in practicing the method of the invention is dependent upon several factors. Among these factors is the average pool temperature, the power of the electron beams, the rate at which the crystal is pulled, the size of the impact areas of the electron beams, and the rate at which heat is removed through the cooled crucible walls. The precise conditions required for successful operation are established empirically and may usually be determined after a few test runs. Some examples of satisfactory operating conditions are described subsequently.

Operation of the illustrated apparatus in accordance with the method of the invention, as above described, enables variation and control over the diameter of the crystal being grown by means which provide a very fast response. Such a fast temperature response is of significance in crystal growing. For example, variation in diameter of the crystal being pulled or grown may be accomplished almost immediately after movement of the electron beams in the described manner. This facilitates the "growing out" of crystal nucleation agents and surface imperfections, as described subsequently. Moreover, variation in environmental conditions may be very quickly compensated for by appropriate movement of the beam or beams. Care must be taken to avoid moving the beams too far away from the crystal being grown because, if the temperature of the molten pool drops sufficiently, a bridge of solid material may form between the crystal and the central hump in the skull **21**.

By using the position of the beams **13** as a primary and rapid means of temperature control, and by using control over the power level of the beams as a secondary and slowly changing variable, control over the crystal diameter may be achieved concurrent with the maintenance of constant molten pool conditions. Movement of the impact areas **14** of the beam results in a changing of the thermal pattern in the pool, thereby effecting the changes in growth conditions of the crystal as previously described. It has been found that the ratio of change in the distance of the beam impact areas **14** from the axis of the crystal to the change in crystal diameter caused thereby is about 3 or 4 to 1.

At the same time, the size and average temperature of the molten pool may be kept relatively constant, despite beam impact position changes and heat balance changes, by adjusting the total beam power level (i.e., adjusting beam current or emitter temperature or both). The constant conditions of purity, temperature, pool size, etc., result in production of a crystal which is symmetrical in its intracrystalline structure, being substantially free of dislocations, providing the subsurface structure of the seed crystal is also substantially free of dislocations.

In order to replenish the molten pool **12** for silicon removed therefrom during the growing of the single crystal **11**, silicon is fed into the pool. In the illustrated embodiment, the feed silicon is in the form of a solid bar **33**

in a commercially obtainable polycrystalline form. The bar **33** of feed silicon is held in a suitable clamp **34** and is lowered toward the molten pool by a rod **35** connected to the clamp. A suitable mechanism, not illustrated, is used to lower the rod **35** and to rotate it, thus rotating the silicon bar **33** as it is being lowered toward the pool.

The lower end of the feed silicon bar **33** is melted by means of an electron beam **36**. The electron beam **36** is produced by an electron beam gun **37** of a construction generally identical to that of the guns **22**. Accordingly, the various parts of the electron beam gun **37** illustrated in FIGURE 1 have been given reference characters identical with the corresponding parts in the electron beam gun **22** illustrated in the same figure. As may be seen in the drawing, the gun **37** is mounted in the enclosure in an attitude which is inverted with respect to the gun **22**. This is for the purpose of achieving a desired electron beam direction, as indicated. As has been described, the thermal pattern in the pool **12** has a substantial effect on the growth of the single crystal **11**. Accordingly, it is desirable to minimize the effect, on the thermal pattern, of the material being fed into the molten pool. The minimization of the effect of additional material is accomplished by directing the electron beam **36** such that it does not strike the surface of the molten pool **12**, thereby avoiding any thermal imbalance. Control over the power and direction of the electron beam **36** is provided by means of a suitable control circuit **38**, illustrated in FIGURE 2. The lower end of the feed bar **33** is melted by the electron beam **36** into a tapering shape and the bar is positioned sufficiently close to the surface of the molten pool **12** that a meniscus **39** forms between the lower tip of the feed bar **32** and the molten pool. The feed rate of the material being added is selected such that the surface of the pool is not disturbed by the flow of material thereinto from the melting lower tip of the bar **32**. The maintenance of a meniscus between the molten lower end of the bar and the molten pool facilitates the addition of material without unduly disturbing the surface of the pool.

Another factor of significance in minimizing the effect of material addition on the thermal pattern of the pool is the location of the region where the material is added. As may be seen in FIGURES 1 and 2, material addition is accomplished in a region outside of the annular region of thermal turbulence in the pool (i.e., the impact area of the electron beams). The thermal currents in the turbulent region act as a dam to insure that the added material is melted and mixed before it enters the central region. Thus, it is possible in many instances to add particulate material directly into the pool outside the turbulence annulus without deleterious effect. Dopant for effecting the semiconductor properties of the pulled crystal may also be added in this way to insure thorough mixing.

To accurately control the position of the impact areas **14**, a control circuit **40** (FIGURE 2) is provided suitably connected to each of the three electron beam guns **22**. The control circuit operates to control the strengths of the fields produced by the electromagnets **27** and hence the radius of curvature of the path which the electrons in the beam follow. By varying the strengths of the fields produced by the electromagnets **27**, therefore, the amount of deflection of the beams and hence the position of the impact areas **14** may be varied. The control circuit is preferably so constructed as to vary the strengths of the respective electromagnets **27** in the electron guns **22** to make the positional variation of the impact areas **14** with respect to each other and to the axis of the single crystal **11** equal and symmetrical. By means of the latter technique, the thermal pattern in the molten pool **12** may be varied generally symmetrically, as desired. A control circuit which may be adapted for use in the system illustrated in FIGURES 1 and 2 is disclosed in U.S. Patent No. 3,235,647, assigned to the present assignee. A similar control circuit may be adapted for use as the control circuit **38**.

In addition to controlling the positions of the beam impact areas, the control circuit **40** is also suitably constructed to control the power of the electron beams. This may be done by controlling the beam current through variation in the temperature of the emitters in the guns, or through variation in the current output of the power supply for the guns. In either case, the amount of energy transferred to the pool by the beams is controlled accordingly, facilitating the attainment of constant pool conditions (e.g., temperature and size).

Referring now to FIGURES 3 through 7, a specific manner of practicing the invention will be described, although the method of the invention is not limited to being practiced in this manner. FIGURES 3 through 7 schematically illustrate various steps in using the apparatus of FIGURES 1 and 2. For simplification, only one of the three beams is illustrated, but it is to be understood that all three beams are moved in the same way. The crystal seed **25**, held in the clamp **28**, is lowered to within about one-quarter inch of the surface of the molten pool **12**. This position is illustrated in FIGURE 3. The positions of the beams are moved in toward the axis of the seed **25** to a position "A," the beams being about ⅜" to 1" from the seed. With the beams **13** being this close to the seed, the seed increases in temperature due to bombardment by fringe electrons and an increased level of radiated heat. When the temperature at the seed tip reaches approximately 900° C., the seed is dipped into the pool to depth of about one sixteenth of an inch. This position is illustrated in FIGURE 4. At about the same time, the beams **13** are moved radially outward with respect to the axis of the seed, over a distance of approximately one-quarter inch, from position "A" to a position "B" indicated in FIGURE 4.

The rotating seed **25**, because of the high temperature of the molten pool, will melt slightly at the interface **31** and a meniscus **30** will form at the surface of the molten pool **12**. Once this meniscus is etsablished between the rotating seed and the pool, the seed is drawn upwardly by the connecting rod **29** (FIGURE 1) at a slow rate, for example, about two inches per hour. At about the same time, the electron beams **13** are moved so that their impact areas **14** are at position "C," illustrated in FIGURE 5. At position "C," the impact areas are approximately one-eighth of an inch closer to the axis of the seed than in position "B," and the turbulence represented by the arrow **32** is also closer.

At the position "C" of the beam, illustrated in FIGURE 5, the surface of the single crystal growing from the seed **25** in the region **42** tapers inwardly at angle ϕ which is greater than 30° from the vertical due to the washing action of the superheated melt in the turbulence region. With the growing crystal getting smaller in diameter than the seed at an angle of fresh surface greater than 30° from the vertical, crystal nucleation agents that may be residing on the surface of the seed grow out of the crystal because the maximum angle at which they can grow is about 30°. Such crystal nucleation agents may consist of coating flakes, condensate on the seed surface (which are all amorphous atoms) and dirt from faulty seed handling procedures. In addition to minimizing the effect of crystal nucleation agents residing on the surface of the seed, the growth condition maintained in the region **42** wipes out any influence of disturbing conditions at the original surface of the seed. Thus, the surface of the single crystal **11** being grown is of the sub-surface quality of the seed.

The growth condition indicated in FIGURE 5 at region **42** will continue until the diameter of the growing crystal **11** is about one-eighth of an inch smaller than the diameter of the seed **25**. After about a one-eighth of an inch reduction in diameter, a constant diameter growth will begin. As soon as constant diameter growth begins, the positions of the impact areas **14** of the beams **13** are moved radially outward about one-eighth to three-eigths of an inch to position "D" illustrated in FIGURE 6. This

reduces the washing action of the superheated melt and causes the growing crystal **11** to increase in diameter at a surface angle of about 20°, such as is indicated in FIGURE 6 at the region **43**.

As soon as the growth condition indicated at the region **43** begins, the rate of movement of the connecting rod **29** (FIGURE 1) is increased slowly, for example over a period of about 5 minutes, to increase the withdrawal rate to about four or five inches per hour. This causes the growth angle of the crystal **11** to change to about 40° from the vertical indicated in the region **44** of FIGURE 6.

Growth of the single crystal **11** under the conditions indicated in the region **44** is continued until the crystal reaches the nominal desired diameter. The proper selection of the beam position "D" will result in the crystal slowly changing the growth angle to 0° to enable the maintenance of a constant desired diameter during the remaining and major portion of the crystal growth. Some minor inward adjustment in beam position may be necessary in order to cause the crystal to "turn the corner" and change to a constant diameter growth. The constant diameter growth situation is indicated in the region **46** of FIGURE 7.

In growing single silicon crystals, successful results may be achieved in utilizing the method of the invention for the following examples:

EXAMPLE 1

Seed crystal diameter: 0.200 inch
Ultimate silicon crystal diameter: 1.50 inches
Furnace vacuum: 2×10^{-5} torr
Average electron beam power: 16 kw.
Pool diameter: 8 inches
Pool depth: 1 inch average
Pool volume: approx. 350 cc.
Seed crystal temperature at immersion: 900° C.
Average surface temperature: 1435° C.
Maximum surface temperature: approx. 1600° C.
Initial withdrawal rate: 2 inches per hr.
Ultimate withdrawal rate: 6 inches per hr.
Rate of crystal rotation: 15 r.p.m.
Rate of silicon feed: 390 grams per hr.
Feed stock cut-off beam power: 2.1 kw.
Rate of crucible rotation: 10 r.p.m. (opposite to crystal)

EXAMPLE 2

Seed crystal diameter: 4.5 mm.
Ultimate silicon crystal diameter: 20 mm.
Furnace vacuum: 4×10^{-5} torr
Average electron beam power: 4.2 kw.
Pool diameter: 90 mm.
Pool depth: 15 mm. avg.
Pool volume: approx. 75 cc.
Seed crystal temperature at immersion: 600° C.
Average surface temperature: 1435° C.
Maximum surface temperature: 1625° C. approx.
Initial withdrawal rate: 5 cm./hr.
Ultimate withdrawal rate: 15 cm./hr.
Rate of crystal rotation: 20 r.p.m.
Rate of silicon feed: 5 mm./hr.
Feed stock cut-off beam power: 1.5 kw.

By way of further illustration, the method of the invention may be practised for materials other than silicon as follows:

EXAMPLE 3

Material: B1900 nickel base alloy
Seed crystal diameter: ¼ inch
Ultimate grown crystal diameter: 2½ inches
Furnace vacuum: 8×10^{-5} torr
Average electron beam power: 23 kw.
Pool diameter: 8 inches
Pool depth: ¾ inch avg.
Seed crystal temperature at immersion: 300° C.
Average surface temperature: 1550° C. approx.
Maximum surface temperature: 1800° C. approx.
Initial withdrawal rate: 6 inches/hr.
Ultimate withdrawal rate: 12 inches/hr.

Since super alloy crystals or grains are usually of satisfactory quality when grown in the naturally preferred direction of crystal growth (as opposed to silicon crystals for semiconductors), crystal diameter may usually be increased directly to its ultimate value by moving the beam impact area outwardly an appropriate distance once growth is begun.

Referring now to FIGURE 8, an alternative embodiment of apparatus for performing the method of the invention is illustrated. Growth of a single crystal **11** from the molten pool **13** in the crucible **18** and skull **21**, is accomplished by utilizing the clamp **28** and the connecting rod **29** in accordance with the method as previously described. In the embodiment of FIGURE 8, however, the electron beam gun **47** is of generally annular configuration.

The gun **47** includes a ring-shaped cathode **48** and an annular shaping electrode **49** having a cross section generally similar to that of the shaping electrode **26** illustrated in FIGURE 1. The accelerating anode **51** is also of annular configuration and has a cross sectional shape generally similar to that of the anode **21** in FIGURE 1. A ring-shaped electromagnet **52** with multiple windings of different average diameters is utilized to achieve variation in the mean diameter of the annular electron beam **53** produced by the gun **47**. The resulting impact pattern or area **54** is of corresponding annular shape.

Thus, it will be appreciated that by varying the strength of the field established by the electromagnet **52** and by varying the effective diameter of the coil windings of the electromagnet by suitable control circuits (not illustrated), the mean diameter of the impact area **54** may be varied to produce corresponding variation in the thermal pattern of the pool **12**. This variation may be utilized to achieve growth of a single crystal as described in connection with FIGURES 3 through 7. Regulation of the beam power level may be utilized, as in the case of the guns **22**, to maintain constant molten pool conditions.

Referring now to FIGURE 9, a further alternative embodiment of apparatus for performing the method of the invention is illustrated. Growth of a single crystal **11** from the molten pool **13** is accomplished by utilizing the clamp **28** and the connecting rod **29** in accordance with the method as previously described. The apparatus is disposed within a vacuum enclosure, not illustrated, and the molten pool **11** is formed at the top of a vertical cylindrical pedestal of polycrystalline feed material, such as silicon. The pedestal **61** is preferably substantially greater in diameter than the ultimate diameter of the single crystal, and is moved upwardly at a rate selected to replenish material removed from the pool as the crystal is pulled. The thermal pattern in the molten pool is regulated so that the pool is contained, at its periphery **62**, by surface tension, the periphery of the pool thereby being about the same diameter as that of the pedestal **61**. Although a pedestal-type feeding arrangement is illustrated, it is to be understood that the molten pool may be contained in a cooled crucible as in the previously described embodiments.

As was the case in connection with the embodiment of FIGURE 8, an annular electron beam gun **47** is provided. The parts of the electron beam gun of FIGURE 9 are identical with those of the gun of FIGURE 8 and have been given identical reference numbers. The electrons are projected to the surface of the pool **13** to form an annular impact pattern **63** thereon, and the electrons comprising the beam define a beam which is generally hollow and frustoconical in configuration.

In the embodiment of FIGURE 9, the electron beam produced by the gun **47** is focused and controlled to regulate both the sharpness and the mean diameter of the annular impact pattern **63** on the surface of the pool **13**.

This is accomplished by means of a pair of electromagnetic coils **64** and **66**, and by a pair of control circuits **67** and **68** connected to the coils **64** and **66**, respectively. The general configuration of the apparatus is similar to that shown and described in U.S. Patent No. 3,105,275 assigned to the assignee of the present invention, and the method of the present invention includes a novel procedure for utilizing such apparatus. The electromagnetic coil **64** includes an annular core **69** of a material having low magnetic reluctance and is surrounded by electrically conductive windings **71**. The construction of the electromagnetic coil **66** is similar to that of the coil **64**, including an annular low reluctance core **72** surrounded by windings **73**. The windings **71** of the electromagnetic coil **64** are energized by the control circuit **67** which controls the magnitude of the current flowing therethrough. Similarly, the windings **73** of the electromagnetic coil **66** are energized by the control circuit **68**, which controls the magnitude of the electrical current flowing through the windings **73**.

The two electromagnetic coils **64** and **66** operate as a compound lens and, by way of analogy to glass lenses for focusing light rays, operate to change the effective focal length of the combination lens by changing the index of refraction of each lens. In terms of magnetic lenses, the magnetic induction of each electromagnetic coil is changed by appropriate changes in the current flowing therethrough. By appropriately regulating the induction of each of the electromagnetic coils **64** and **66**, the effective magnification of the combination lens can be changed to thereby regulate the mean diameter of the annular beam impact pattern **63** on the surface of the pool **13**. The regulation of the induction is selected in appropriate combinations to maintain sharp imaging of the beam on the surface and thereby provide a very narrow width of beam impact pattern, approximating the thickness of the filament or cathode **48**. By appropriate regulation of the two lenses, a wide range of variation in the mean diameter of the impact pattern may be achieved while maintaining sharpness in the image of the emitter (i.e., the impact pattern width).

By making the mean diameter of one or more of the electromagnetic lenses smaller, the image size, that is, the width of the annular impact pattern **63**, may be made even smaller than the thickness of the emitter **48**. Thus, a plurality of concentric coils may be provided in place of one or more of the electromagnetic coils **64** and **66**, and each of the concentric coils may be selectively energized for a desired image size.

Using an electron beam gun having an emitter mean diameter of 7", spaced 6¾" above the horizontal midplane of the coil **64**, and 14½" above the surface of the pool **13**, wherein the inner diameter of the coil **64** is 11" and the inner diameter of the coil **66** is 6½", and wherein the top of the coil **66** is spaced 1" below the surface of the pool **13**, the following combination of magnetic induction of the coils **64** and **66** give the following listed mean diameter of the impact pattern **63** while producing a sharp image of the emitter **48** on the pool surface:

Coil 64, ampere turns	Coil 66, ampere turns	Mean diameter of impact pattern, inches
6.6×10^3	4.5×10^2	1½
5×10^3	10.5×10^2	3
4.6×10^3	1.1×10^3	3½
4×10^3	1.3×10^3	4
2.4×10^3	1.6×10^3	5
1.5×10^3	1.7×10^3	5½
50	1.75×10^3	6

It may therefore be seen that the invention provides an improved method for producing a single crystal. Crystal growth conditions are readily controlled by the method of the invention, and the effect of nucleation agents residing on the surface of the seed is minimized. In accordance with the invention, a smooth surface single crystal of high quality is produced, the cross sectional size of which may be selected as desired. Replenishment of the molten pool from which the single crystal is grown is readily accomplished without deleterious effect on growth conditions.

Various modifications of the invention, in addition to those shown and described herein, will be apparent to those skilled in the art from the foregoing description and accompanying drawings. Such other modifications are intended to fall within the scope of the appended claims.

What is claimed is:

1. A method of growing a single crystal from a molten pool of a material from which a crystal is to be made comprising,

(a) contacting the surface of the molten pool with a single crystal seed to establish an interface between the seed and the molten pool,

(b) directing an electron beam onto the surface of the molten pool to create a region of turbulent flow of molten material within the molten pool,

(c) withdrawing the seed from the molten pool at a rate such that a single crystal of material is grown,

(d) and controlling the location of the impact area of the electron beam on the surface of the molten pool to control the distance of the region of turbulent flow from the interface between the crystal and the molten pool, thereby controlling the diameter of the single crystal being grown.

2. A method according to claim **1** wherein the electron beam is controlled so that the electron beam impact area is generally annular in shape and substantially surrounds the single crystal so that the region of turbulent flow is substantially annular and surrounds the single crystal, whereby an inward flow of superheated molten material is established substantially surrounding the single crystal.

3. A method according to claim **2** including adding material to the molten pool outside of the annular impact area.

4. A method according to claim **2** including adding a dopant to the molten pool outside of the annular impact area.

5. A method according to claim **1** wherein the power of the electron beam is varied to control the average temperature and size of the molten pool.

6. A method according to claim **2** wherein the electron beam is controlled so that the annular beam impact area is caused to move closer to the seed to cause a necking down of the single crystal at a surface angle greater than 30° from the axis of the single crystal.

7. A method according to claim **6** wherein, after necking down, the electron beam is controlled so that the annular beam impact is caused to move farther from the seed to produce an enlargement of the single crystal cross section at a surface angle of about 20° from the axis of the single crystal.

8. A method according to claim **7**, including substantially increasing the rate of withdrawal after enlargement of the single crystal cross section begins.

9. A method according to claim **8**, wherein the increase in the rate of withdrawal is approximately doubled over a period of about 5 minutes.

10. A method according to claim **2**, wherein the seed is positioned adjacent the pool surface, and wherein the electron beam is controlled to heat the seed by bombardment of fringe electrons and radiated heat.

11. A method according to claim **2**, wherein the mean diameter of the annular beam impact area, and the width thereof, is controlled by controlling the current through

a pair of annular electromagnetic lenses spaced axially along the path of the electron beam.

References Cited

UNITED STATES PATENTS

2,683,676	7/1954	Little et al.	148—1.5
2,858,199	10/1958	Larson	148—1.6
3,232,745	2/1966	Rummel et al.	148—1.6
3,278,274	10/1966	Liebmann et al.	

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,494,804 Dated February 10, 1970

Inventor(s) Charles W. Hanks; Charles d'A. Hunt

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 10, line 58, after "impact" insert --area--.

SIGNED AND
SEALED
JUL 14 1970

(SEAL)
Attest:

Edward M. Fletcher, Jr.
Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents