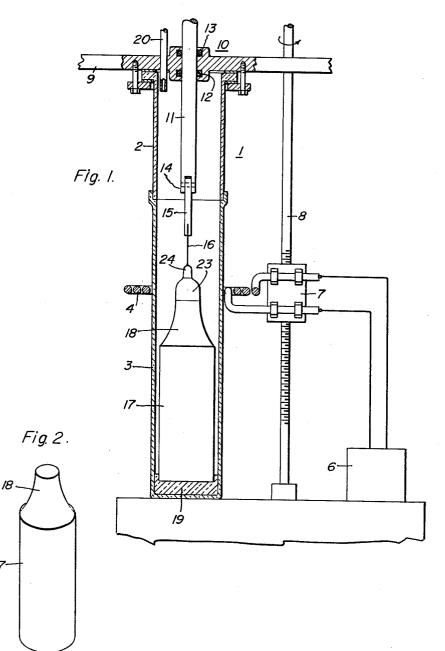
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METHOD OF GROWING DISLOCATION-FREE SEMICONDUCTOR CRYSTALS

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3,135,585 METHOD OF GROWING DISLOCATION-FREE SEMICONDUCTOR CRYSTALS William C. Dash, Ballston Lake, N.Y., assignor to General Electric Company, a corporation of New York Filed Mar. 1, 1960, Ser. No. 12,206 14 Claims. (Cl. 23-301)

The present invention relates to methods for growing semiconductor crystals having a high degree of crystal 10 perfection. More particularly, the invention relates to methods for growing high purity, oxygen-free single crystalline ingots of semiconductor materials.

Many different methods for the preparation of monocrystalline ingots of semiconductive materials such as 15 germanium, silicon, silicon carbide, and the compound semiconductors such as compounds of the elements of group IIIB and group VB of the periodic table, such as gallium arsenide, gallium antimonide, indium phosphide and the like have long been known in the semiconductor 20 arts. These methods are generally predicated upon the mechanism of seed crystal withdrawal of an ingot from a melt of semiconductive material contained in a reasonably nonreactive crucible. Such crucibles are most generally made from quartz, which is silicon dioxide. A1-25though quartz and other crucible materials suitable for crystal growth are reasonably non-reactive with semiconductor materials in the liquid state, they generally do cause some contamination of the melt by the introduction of oxygen from the oxide of which the crucible is 30 fabricated. This is particularly true in the case of silicon and the compound semiconductors such as those of the group III-group V type which have relatively high melting points. Although some oxygen contamination may be tolerated in certain instances, this contamination is 35 highly objectionable. Thus, for example, in silicon bodies, the presence of oxygen as an impurity is responsible for the phenomenon known as "heat treating." This phenomenon, for example, may result in a high resistivity P-type silicon crystal, grown with crystal rotation, reverting to N-type silicon when heated to a temperature of 450° C. or higher.

Certain methods have been devised for growing single crystals of semiconductive materials keeping the molten 45 semiconductor out of contact with the crucible and, hence, avoiding oxygen contamination. Some of these methods are the "floating zone" techniques, "caged zone refining" and like processes. All of these methods, however, cause the resultant crystals to be formed under conditions of 50 high strain and result in a high density of dislocations therein. As used herein, a crystal dislocation in a semiconductor body may be defined as a highly strained inner region within the crystal produced by a displacement of one large portion of the crystal lattice structure with re-55 spect to another such portion. Once a dislocation has been established, it tends to propagate through a crystal, resulting in a chain or string of dislocations. Semiconductor bodies possessing a high degree of dislocations exhibit poor minority charge carrier lifetime characteristics and are of limited utility in the preparation of semiconductor devices such as transistors.

Accordingly, an object of the present invention is to provide methods for growing single crystals of substantially oxygen-free dislocation-free semiconductive materials. 65

A further object of the present invention is to provide such methods having a minimum complexity and a maximum ease of operation.

Briefly stated, in accord with the present invention, 70 a charge ingot of the semiconductor material in substantially the form of a long cylinder is prepared. The charge 2

cylinder is then shaped to provide a symmetrical reduction in the diameter of one end of the cylinder. For example, the end of the cylinder is tapered with a taper which is long as compared with its width. The tapered charge ingot is then placed within a quartz tube which has a diameter only slightly larger than the diameter of the charge ingot and the tapered end thereof is melted by radio frequency induction caused by an induction coil having a planar configuration. A seed crystal of the proper crystallographic orientation having an extremely restricted diameter is lowered into the molten semiconductive mass and a monocrystalline ingot of semiconductor which is free of oxygen and of dislocation is grown from the molten bead atop the charge ingot by seed crystal withdrawal. As a monocrystalline ingot is grown from the molten portion of the charge the induction heating coil is lowered to continually supply a sufficient amount of heat to the molten bead atop the charge so that as semiconductive material is removed therefrom by the growing ingot, the amount of liquid is replenished by melting the adjacent region of the charge ingot by thermal conduction so that a sufficient amount of semiconductive material is always present atop the solid charge ingot.

The novel features believed characteristic of the invention are set forth in the appended claims. The invention itself, together with further objects and advantages thereof, may best be understood by reference to the following description, taken in connection with the appended drawing in which:

FIG. 1 is a schematic illustration of one apparatus with which the invention may be practiced, and

FIG. 2 is a perspective view of a semiconductive charge ingot used as a starting material in the present invention. It has long been desired in the semiconductor arts that methods for growing dislocation-free, oxygen-free ingots of semiconductive materials be available. This was because the prior available methods for growing semiconductor materials by the seed crystal withdrawal method usually relied upon the growth of a semiconductor crystal from a melt maintained in a crucible. The crucible material inevitably introduced some contamination into the growing ingot. Other methods such as the "floating zone" process and "caged zone refining" process, although producing materials of free from oxygen inevitably produced materials which have a high dislocation density. One difficulty in growing a semiconductor crystal from a molten body of semiconductive material which is not in contact with a contaminating crucible, but rather, is maintained upon a pedestal of the same semiconductive material lies in the difficulty of limiting the amount of charge which is coupled to the electromagnetic field which melts the charge so as to maintain only the desired amount of charge material in the molten state.

In my copending application Serial Number 705,685, filed December 27, 1957, now U.S. Patent No. 2,961,305, and assigned to the assignee of the present invention, I provided a method which overcame this disadvantage in that a starting charge of semiconductive material which was susceptible to electromagnetic coupling was suspended upon a slotted pedestal of the same semiconductive material which is incapable of sustaining eddy currents and thus is not susceptible to electromagnetic cou-While this method is satisfactory for the growth pling. of semiconductive material having freedom from oxygen and dislocations, it is not a simple method in that the slotted crystal must be carefully prepared and that certain problems of wetting between the molten and solid semiconductor materials do exist. In accord with the present invention I have found it possible, by carefully selecting and controlling the geometry of the charge ingot

and the shape and size of the electromagnetic induction coil utilized to control the amount of semiconductive material maintained molten. I then grow therefrom crystals which are essentially oxygen and dislocation-free without any disadvantages which may have been attendant 5 in my aforementioned method.

While the invention may be practiced with a number of semiconductor materials such as germanium, silicon and compound semiconductors such as the Group III6-Vb semiconductors including gallium arsenide, indi- 10 um antimonide and the like, it will, for sake of ease of explanation, be herein described with respect to the growth of silicon semiconductor ingots since it is of particular utility when practiced with silicon.

In FIG. 1 an apparatus with which the invention may 15 be practiced includes a small diameter transparent crystal furnace enclosure 1, preferably fabricated from quartz including an upper portion 2 and a lower portion 3, fitted together in non-sealing relationship to allow for the escape of a flushing gaseous atmosphere. An induction heating 20coil 4 composed of a plurality of concentric windings, all in the same lateral plane and having the diameter of the smallest winding thereof only slightly larger than the outside diameter of furnace enclosure 1 permitting free movement longitudinally therealong, is positioned about 25 furnace enclosure 1. Induction heating coil 4 is supplied with a suitable radio frequency alternating electric current from an alternating current source 6 and is adjustable to be moved vertically along furnace enclosure 1 by means of gear train 7, mounted upon threaded ver- 30 tical rod 8 and driven by rotation of rod 8.

Furnace enclosure 1 is supported from the lower surface of a plate 9 and may be secured thereto in vacuumtight relationship by means of a gasket assembly 10. Plate 9 is apertured centrally of the furnace enclosure to 35 receive a crystal pulling mandrel 11 which extends axially within the furnace. The mandrel 11 is maintained in vacuum-tight relationship with plate 9 by means of gaskets or O rings 12 received into receiver 13. The lower end of mandrel 11 terminates in a chuck 14 which contains 40 a split rod of quartz 15 which may receive a seed crystal 16 in the lower end thereof.

In practicing the invention, a seed crystal 16 of the semiconductive material of which the grown ingot is to be composed is inserted within the lower split end quartz 45 rod 15 after the rod has been heated and cooled to render the respective parts of the split end flexible. A charge ingot 17 having a tapered upper end 18 is mounted within furnace enclosure 1 and is supported upon a quartz jig 19 which locates it concentrically within furnace en-50closure 1, which is only slightly larger than the outside diameter of the charge ingot. A gaseous inlet pipe 20 passes through plate 9 at the upper end of furnace enclosure 1 to facilitate flushing furnace enclosure 1 with a desired gas.

In FIG. 2 of the drawing there is shown a typical configuration utilized for charge ingot 17 in accord with the invention. As may be seen from the drawing, charge ingot 17 includes a cylindrical lower portion and an upper tapered portion 18 which has a restricted diameter which 60 is approximately 0.3 to 0.4 of the larger diameter of the ingot.

The tapering of ingot 17 represents the heart of the present invention. The smaller diameter of the tapered end represents a diameter which is actually a balance be-65 tween the necessity of satisfying two conditions. Thus, the diameter must be small enough so that the end thereof may be initially rendered molten and maintained stably atop the charge ingot, held by surface tension alone. Additionally, this diameter must not be so small that the heat capacity of the molten drop formed is comparable to the heat capacity of the seed crystal, resulting in freezing of the droplet when contacted thereby. This critical choice of diameter for the smaller end of the tapered portion of the ingot is not required in the apparatus used in my aforementioned copending application, since in that 75 heating coil 4 causing the tip of tapered portion 18 of

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case, the slotted disc which was used to support the molten material did not couple with the electromagnetic field, which could, therefore, be of any high strength sufficient to maintain the ingot molten without the danger of melting the slotted charge.

The larger diameter of the charge ingot utilized in the present invention represents a satisfaction of the necessity of having a substantial amount of semiconductive material available in order that large monocrystalline ingots may be grown. The unique feature of the tapered charge ingot is that the smaller diameter of the tapered end satisfies the criteria for initiation of crystal growth and overcomes the dangers of freezing on one hand and instability on the other hand. At the same time, the larger diameter of the untapered portion of the ingot makes it possible to grow large, useful size ingots. There is no danger of instability or freezing once the larger diameter of the ingot has been reached, since once the seed crystal and growing ingot have by that time, come into thermal equilibrium with the molten material and the size of the droplet may be increased substantially without freezing thereof or loss of stability. Accordingly, it is evident that the use of the taper on the charge ingot utilized in the present invention satisfies both the criteria for stability of growth and at the same time permits the growth of monocrystalline ingots of semiconductive material free of dislocation and oxygen and having a size heretofore unobtainable from any other method.

In the practice of the invention, the charge ingot is first prepared. While the ingot may be made of high purity polycrystalline semiconductive silicon, it is preferably cast in monocrystalline form either by conventional seed withdrawal techniques or other suitable techniques for the fabrication of monocrystalline ingots, thus insuring maximum freedom from unwanted impurities. The upper tapered end 18 of charge ingot 17 is then machined as, for example, with a diamond saw to symmetrically reduce the area thereof as, for example, by providing a taper thereto. The taper is not critical, but the length of the taper should be greater than the diameter of the charge ingot. It has been found convenient when a silicon ingot having a diameter of 5%'' is utilized, to provide a taper approximately 0.810'' long with a tip having a diameter of approximately 0.280". After treatment, the charge ingot is etched and washed in distilled water to remove impurities and is placed in jig 19. Furnace enclosure 1 is placed in vacuum-tight relationship with plate 9. Induction heating coil 4 is placed in position adjacent the upper surface of tapered end 18 of charge ingot 17. A monocrystalline seed crystal of an extremely restricted diameter is prepared as, for example, by taking a monocrystalline wire approximately 0.020" in diameter and approximately 34" in length and etching the seed until the tip diameter is reduced to approximately 0.010" in diameter. The split end of quartz rod 15 is then heated until it becomes 55soft and allowed to cool. The respective portions of the quartz rod are then flexed apart and the monocrystalline seed crystal 15 is inserted between them and held resiliently thereby.

In accordance with the present invention it has been found that dislocation-free crystals may be grown if the seed crystal has a crystallographic orientation in which the (1, 1, 1) or (1, 0, 0) plane is perpendicular to the axis of the seed crystal. The mandrel is then inserted into the furnace and is positioned in close proximity to the tapered portion 18 of charge ingot 17. The volume of furnace enclosure 1 is flushed with a suitable non-reactive gas as, for example dry argon at approximately atmospheric pressure for approximately 15 minutes to 70 remove all reactive gases therefrom, the argon together with any gas removed therewith being allowed to "bleedoff" through the unsealed junction of parts 2 and 3 of furnace enclosure 1. A suitable current of radio frequency alternating current is then supplied to induction

charge ingot 17 to be melted and causing the formation of a droplet 23 of molten silicon to rest on the upper surface thereof. As soon as this droplet is formed, the current passed through the induction heating coil is decreased and/or regulated so that it is high enough to prevent the droplet from freezing but low enough to prevent the droplet from increasing to a volume which cannot be maintained in a stable position atop the tapered portion 18 of charge ingot 17. Droplet 23 is maintained in position primarily by surface tension. If the volume thereof becomes too great, the force of its weight will exceed the surface tension forces and the silicon will flow down over the charge ingot. If the current is too low, the droplet will refreeze. While the suitable range of current to maintain any particular size droplet will vary with the 15 exact configuration of the furnace utilized, with the semiconductor material being melted, and other factors, it is well within the knowledge of those well versed in the art to make such adjustments.

After the proper value of current has been obtained, 20 mandrel 11 is lowered until seed crystal 16 comes into contact with droplet 23. The seed crystal is inserted into the droplet sufficiently far to cause the tip thereof to become molten. It is left there for a few seconds in order that thermal equilibrium be attained and then slowly the mandrel is withdrawn, either manually or in accord with a predetermined withdrawal cycle which is established by a suitable mechanism controlling the mandrel, which mechanisms are also well known to those well versed in the art.

In accord with another feature of the present invention, the grown ingot is rendered disclocation-free by growth upon a seed crystal, the volume of which is extremely small as compared with the volume of molten droplet 23. In fact, the seed crystal is made in the form 35 of a thin needle having a thickness just sufficient to support the weight of the grown ingot. This is to reduce to an absolute minimum any thermal stresses introduced into the grown crystal by the difference in temperature between the seed crystal and the molten mass of semi-40 conductive material. In the case of silicon, dislocationfree crystals have been grown utilizing crystals having a minimum cross-sectional area of from 0.001 square inch for each three grams of crystal to be grown or to a maximum cross-sectional area of 0.0025 square inch. 45 Another way in which the dislocations are minimized in the grown crystals, in accord with the present invention, is by properly orienting the seed crystal as mentioned hereinbefore. When the seed crystal is cut with the (1, 1, 1) or (1, 0, 0) plane of the seed perpendicular to the 50 seed axis, in the growing ingot the preferential orientation of dislocations is at an angle to the axis of the axis of the crystal ingot. In this manner, any dislocations which may occur are rapidly propagated to the lateral edges of the growing crystalline ingot and are thus unable to fur- 55 ther propagate leaving the remainder of the ingot free of dislocations.

As the mandrel and the seed crystal are withdrawn, a monocrystalline ingot 24 of silicon is formed by nucleation upon the seed. Ingot 24 is grown from the molten 60 mass of semiconductive material 23, without rotation, at a constant rate. While I prefer that the seed crystal be withdrawn at a rate of approximately five millimeters per minute, pulling rates of one to ten millimeters per minute are suitable in the practice of the invention. As a mat-65 ter of practice, as long as the molten droplet of semiconductive material rests upon a portion of the charge ingot which is less than the largest diameter of the charge ingot itself (on the tapered portion) the speed of withdrawal may be faster than when the droplet is resting upon the 70remainder of the charge ingot. Thus, for example, with a 5/8" diameter silicon crystal tapered down to approximately 5/16" at the tip, for the first 34" of the charge ingot.

ever, the molten droplet is resting upon the larger dimension of the ingot, the growth rate is reduced to approximately two to three millimeters per minute.

In the practice of the present invention, as ingot 16 is withdrawn from molten droplet 23, the temperature thereof tends to increase. This is because the electromagnetic coupling between electromagnetic coil 4 and droplet 23 is much greater than any coupling between coil 14 and either growing crystal 24 or charge ingot 17 because the conductivity of liquid silicon is much greater than the conductivity of solid silicon. This results in substantially all of the electromagnetic energy being coupled into the liquid silicon preferentially. This permits close control over the amount of silicon which is molten so as to prevent droplet 23 of molten silicon becoming so large as to exceed the force of surface tension, thus causing it to flow over charge ingot 17. As, however, the volume of droplet 23 decreases due to nucleation on seed crystal 16, induction heating coil 4 is progressively lowered a small amount to compensate for the loss by melting a portion of the tapered end 18 of charge ingot 17. At the same time, power to the coil is increased to compensate for the increased size of the molten droplet as the diameter of the charge ingot increases. After the maxi-mum diameter of cylindrical portion 17 is reached, no further change need be made. The progressive lowering of coil 4, so as to be always adjacent droplet 23, may be accomplished either manually or automatically.

In the described fashion, a monocrystalline ingot of semiconductive material 16 is continually withdrawn from molten mass 23 of semiconductive material which is continually replenished from charge ingot 17. This process is continued until charge ingot 17 is substantially exhausted or is molten down to the point where further melting will cause oxygen or other impurities to be drawn into the molten mass 23 from jig 19. At this point, the process is discontinued and the monocrystalline ingot is pulled free of molten mass 23 while it still remains molten. Subsequent thereto, the power to induction heating coil 4 is discontinued.

Since semiconductive ingot 16 is grown from a mass of semiconductive material 23 which is supported by surface tension atop a high purity charge ingot 17 of the same semiconductive material, it is uncontaminated by contact with quartz or any other impurity-supplying bodies which may supply oxygen thereto and is, hence, free from any objectionable oxygen contamination. Likewise, since the crystal is grown by seed crystal withdrawal and is free of constraint and since the seed crystal is of small diameter to preclude strains and has the proper crystallographic orientation to produce optimum freedom from dislocations, the dislocation density within the ingot falls to zero shortly after growth is begun. Thus, for example, in crystals grown in accord with this process there are no detectable dislocations over a substantial portion of the crystal which comprises better than half of the length thereof. Conventional crystals grown by non-contaminating methods as, for example, the "floating zone," "caged zone refining" and like methods, on the other hand, contain a dislocation density of approximately 10^{-4} , 10^{-5} dislocations per cubic centimeter.

The oxygen-free, dislocation-free semiconductor crystals grown in accord with the present invention may be of high purity, substantially intrinsic semiconductive material for use in thermosensitive and photosensitive elements or may, on the other hand, contain a substantial quantity of significant acceptor or donor activator impurities, or both, and may be utilized in the fabrication of asymmetrically conductive devices such as P-N junction rectifiers and transistors. Thus, for example, both a significant donor and a significant acceptor activator impurity may be included in ingot 17 so that, upon withdrawal of inget 24 therefrom, a semiconductor ingot havthe growth rate may conveniently be kept at approxi-mately five to seven millimeters per minute. When, how-75 formed which ingot is suitable for the starting material for the local fusion technique of manufacturing semiconductor devices as disclosed and claimed in U.S. Patent Number 2,822,309, issued to R. N. Hall. Alternatively, the ingot may be utilized as a source of starting material for the fabrication of fused and diffused P-N junction 5 rectifiers and transistors as set forth in the copending application of R. N. Hall, Serial Number 596,943, filed July 10, 1956, now U.S. Patent No. 2,994,018, and assigned to the assignee of the present invention. In both of these instances, if the ingot is of germanium or silicon 10 or a like semiconductor and it is desired that it possess N-type conductivity characteristics, the significant activator impurities added to ingot 17 prior to the growth of ingot 24 therefrom may comprise phosphorus, arsenic or antimony. If, on the other hand, it is desired that the 15 no detectable oxygen content under infrared absorption grown ingot be of germanium or silicon or a like semiconductor and possess P-type conductivity characteristics, a significant impurity activator may comprise boron, aluminum, gallium, indium or mixtures thereof. If the semiconductor material utilized is a compound semi- 20 conductor such as the group III-group V type such as gallium arsenide, the usual donors such as sulphur, selenium or tellurium and acceptors such as zinc or cadmium therefor may be added to the charge ingot.

While the invention has been described with particu- 25 larity hereinbefore, the following specific examples are set forth for the further use of those skilled in the art. These examples are illustrative only and are not to be construed in a limiting sense.

Example 1

A monocrystalline cylindrical rod of silicon 5%" in diameter and approximately 3" in length is cut from a monocrystalline ingot grown by the "floating zone" tech-The upper end of the rod is tapered in a taper 35 nique. The upper end of the rod is tapered in a taper 0.750'' in length symmetrically down to a diameter of This charge ingot is placed within the enclosure 0.280". 1 in FIG. 1 in a suitable jig 19. A monocrystalline seed crystal having the (1, 1, 1) crystallographic plane perpendicular to the axis thereof approximately 34" long 40 and 0.020" in diameter is etched down to a diameter of 0.010". The split end of quartz chuck 15 as illustrated in FIG. 1 of the drawing is heated until soft and allowed to cool. The seed crystal is then inserted therein. The chuck is allowed to cool and to permanently fix the seed 45 crystal therein. The seed crystal and the chuck are inserted into chuck 14 of mandrel 11 in FIG. 1 and inserted into furnace envelope 1. An induction heating coil which comprises three concentric turns 3/16" O.D. copper tubing are wound to form a flat coil having an inside diameter of 1" and an outside diameter of 2". The coil is con-50 nected to a suitable water supply and water is pumped therethrough for cooling purposes. The coil is connected to a 450 kc. radio frequency power generator and is connected with a screw adjustment for linear movement vertically over furnace envelope 1. The coil is 55initially positioned immediately adjacent the upper portion of the tapered portion of the charge ingot. Dry argon at atmospheric pressure is admitted into the charge and the envelope is flushed for 15 minutes to remove all 60 impurities therefrom. The portion of furnace envelope 1 immediately adjacent the end of charge ingot 17 is heated with a concentric resistance heater to raise the temperature of charge ingot 17 so that it may be coupled electromagnetically with induction heating coil 4. When the 65silicon glows red, approximately 450 kc. radio frequency power at a current of 150 a. is connected to induction heating coil 4, causing the formation of a molten droplet approximately 1/4" in diameter on the top of the tapered portion of the charge ingot. Minor adjustments in the input power are made to prevent the droplet from becom- 70 ing too large or from freezing. The mandrel containing the seed crystal is lowered until the seed crystal extends into the droplet. After a period of approximately five minutes to allow thermal equilibrium to become established, the seed crystal is withdrawn without rotation 75

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at a rate of five millimeters per minute. As the droplet lowers, by melting the portion of the charge ingot immediately adjacent thereto, the induction coil is lowered manually so as always to be adjacent the molten droplet. After the molten droplet has passed the tapered portion of the charge ingot, the rate of growth is lowered to approximately three millimeters per minute and the crystal 16 is grown until the molten droplet is within $\frac{1}{2}$ " of the lower end of the charge ingot. At this point, the ingot is pulled free of the molten droplet and the power is turned off. The crystal resulting from this sequence is approximately one centimeter in diameter, twelve centimeters long and weighs approximately twenty grams. Wafers cut from the central portion of this crystal showed at 9 microns and no detectable dislocations.

Example 2

The procedure of Example 1 is followed using as a charge an ingot of germanium of zone refined quality $\frac{1}{2}$ " in diameter and 3" long. The upper end is tapered to a diameter of $\frac{1}{4}$ " over a length of $\frac{3}{4}$ ". The seed crystal used is of germanium with the (1, 1, 1) plane perpendicular to the seed axis and having a length of 1" and a diameter of 0.020" tapered to 0.010" at the tip. The apparatus used is the same. A molten droplet is formed by applying approximately 100 a. at 450 kc. to the coil. The same procedure is followed with an initial growth rate of 1 cm./min. for the first 34" and a rate of 3.5 mm./min. thereafter. The grown ingot has a diameter of 6 mm. and a length of $10^{\prime\prime}$ is grown. The ingot is free of measurable dislocations after the first inch.

While the invention has been described with respect to certain practices thereof, it will be appreciated that many variations and changes will immediately occur to those skilled in the art. Accordingly, I intend by the appended claims to cover all such modifications that fall within the true spirit and scope of the foregoing disclosure.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. The method of growing large oxygen-free dislocation-free single crystals of inorganic semiconductive material which method comprises: preparing an elongated cylindrical solid source ingot of inorganic semiconductive material having essentially the same composition desired in a single crystal, said solid source ingot having a diameter thereof sufficient to allow the growth therefrom of large crystals; providing a solid tapered portion in one end of said solid source ingot, said portion being tapered to a substantially restricted diameter at said end of the ingot, the tapered portion having a length greater than said diameter of the solid source ingot, said substantially restricted diameter being large enough to provide stable support primarily by surface tension for a molten globule atop said tapered portion, and said restricted diameter being small enough to permit a needle-like seed crystal to be brought into thermal equilibrium with a molten globule supported primarily by surface tension atop said tapered portion without freezing said globule; suspending said ingot within a transparent refractory tubulation which is only slightly larger in inside diameter than the largest outside diameter thereof with said tapered portion uppermost; establishing a concentrated radio frequency electromagnetic field sufficient to cause the melting of said semiconductive material at the upper restricted diameter end of the tapered portion of said ingot so as to cause said end to melt; regulating the strength of said electromagnetic field to cause a molten droplet of said semiconductive material to be suspended by surface tension upon the upper end of said body while inserting therein a very thin needle-like seed crystal of the same semiconductive material; and growing a dislocation-free oxygenfree monocrystalline ingot of semiconductive material having a very large diameter as compared with said seed crystal from said molten droplet by seed crystal with-

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drawal while replenishing said droplet by further melting said ingot.

2. The method of claim 1 wherein said inorganic semiconductive material is silicon.

3. The method of claim 1 wherein the cross-sectional $_5$ area of said needle-like seed crystal varies from .001 sq. inch for each 3 grams of crystal to be grown to 0.0025 sq. inch for each of 3 grams of crystal to be grown.

4. The method of claim 3 wherein said inorganic semiconductive material is silicon.

5. The method of claim 1 wherein the longitudinal axis of said needle-like seed crystal is perpendicular to a crystallographic axis thereof selected from the group consisting of the (1, 1, 1) and the (1, 0, 0) axes.

6. The method of claim 5 wherein said inorganic semi- 15 conductive material is silicon.

7. The method of claim 1 wherein said concentrated radio frequency electromagnetic field is provided by an electromagnetic heating coil composed of a plurality of concentric windings, having a generally planar configuration, lying substantially in the plane defined by the restricted diameter end of said solid source ingot.

8. The method of claim 7 wherein said inorganic semiconductive material is silicon.

9. The method of claim 1 wherein further melting of 25 said ingot while withdrawing said crystal is accomplished by simultaneously lowering said electromagnetic field relative to said ingot while simultaneously increasing the strength of said field until all of said tapered portion is melted and thereafter maintaining the field strength at a 30 steady value.

10. The method of claim 9 wherein said inorganic semiconductive material is silicon.

11. The method of growing large oxygen-free dislocation-free single crystals of inorganic semiconductive ma-35 terial which method comprises: preparing an elongated cylindrical source ingot of inorganic semiconductive material having essentially the same composition desired in a single crystal; providing a taper having a restricted diameter approximately 0.3 to 0.4 of the diameter of said 40 ingot to one end thereof, said taper having a length greater than the diameter of said ingot; suspending said ingot within a transparent refractory tubulation which is only slightly larger in inside diameter than the largest outside diameter thereof with said tapered end uppermost; estab- 45 lishing a concentrated radio frequency electromagnetic field sufficient to cause the melting of said semiconductive material at the upper restricted diameter end of said taper to said ingot so as to cause said end to melt; regulating the strength of said electromagnetic field to cause 50 a molten droplet to said semiconductive material to be suspended by surface tension upon the upper end of said body while inserting therein a very thin needle-like seed

crystal of the same semiconductive material; and growing a dislocation-free oxygen-free monocrystalline ingot of semiconductive material having a very large diameter as compared with said seed crystal from said molten droplet by seed crystal withdrawal while replenishing said molten droplet by further melting said ingot.

12. The method of claim 11 wherein said semiconductive material is silicon.

13. The method of growing large oxygen-free disloca-10 tion-free single crystals of inorganic semiconductive material which method comprises: preparing an elongated cylindrical source ingot of inorganic semiconductive material having essentially the same composition desired in a single crystal; providing a taper which is greater in length than the diameter of said ingot at one end thereof; said taper reducing the diameter of the ingot at said end to a value of from 0.3 to 0.4 of the diameter of said ingot; suspending said ingot within a transparent refractory tubulation which is only slightly larger in inside diameter than the largest outside diameter thereof with said taper uppermost; establishing a concentrated radio frequency electromagnetic field sufficient to cause the melting of said semiconductive material at the upper restricted diameter end of said taper to said ingot so as to cause said end to melt; regulating the strength of said electromagnetic field to cause a molten droplet of said semiconductive material to be suspended by surface tension upon the upper end of said body while inserting therein a very thin needle-like seed crystal of the same semiconductive material; growing a dislocation-free oxygen-free monocrystalline ingot of semiconductive material from said molten droplet at a first rate of growth while progressively replenishing said molten droplet by lowering said electromagnetic field to encompass unmelted portions of the tapered portion of said ingot; and when the tapered portion of said ingot has been entirely melted continuing to grow said monocrystalline ingot of semiconductive material at a second rate at a steady power input to said electromagnetic field while further lowering said electromagnetic field to replenish said molten droplet from the unmelted portion of said ingot.

14. The method of claim 13 wherein said semiconductive material is silicon.

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