United States Patent

[11] 3,607,109

[72] Inventor Emil R. Capita
7020 Kennedy Blvd., North Bergen, N.J. 07047

[21] Appl. No. 696,560
[22] Filed Jan. 9, 1968
[45] Patented Sept. 21, 1971

[54] **METHOD AND MEANS OF PRODUCING A LARGE DIAMETER SINGLE-CRYSTAL ROD FROM A POLYCRYSTAL BAR**
2 Claims, 2 Drawing Figs.

[52] U.S. Cl. .... 23/273 SP, 23/301 SP
[51] Int. Cl. B01j 17/10
[50] Field of Search .... 23/301 SP, 273 SP; 29/25.3; 252/62.9

[56] **References Cited**

UNITED STATES PATENTS

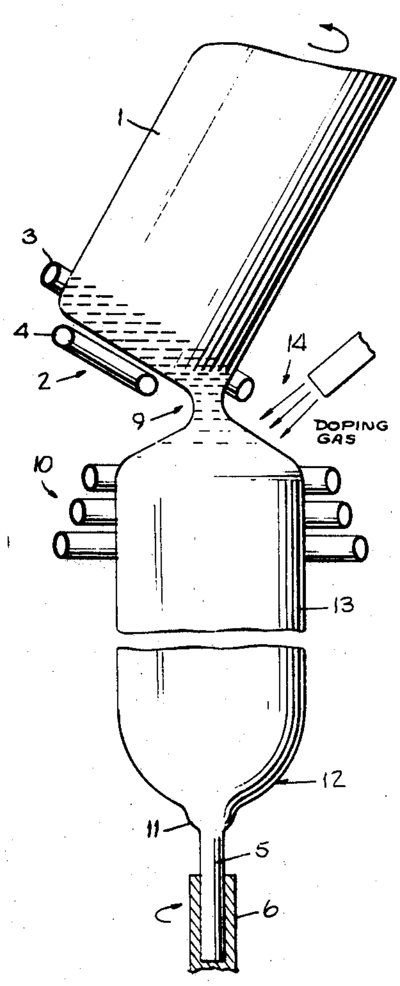
2,962,363	11/1960	Martin	23/273 SP
2,972,525	2/1961	Emeis	23/273 SP
3,002,824	10/1961	Francois	23/301 SP
3,036,892	5/1962	Siebertz	23/301 SP
3,477,811	11/1969	Keller	23/301 SP

FOREIGN PATENTS

1,061,745	11/1957	Germany	23/273
1,482,659	6/1966	France	23/301

Primary Examiner—Norman Yudkoff
Assistant Examiner—S. Silverberg
Attorney—Norman N. Holland

ABSTRACT: A method and means for producing a large diameter monocrystalline or single-crystal rod of semiconducting material from a polycrystalline bar comprising the supporting of the bar at an angle with the vertical and the heating of the lower end to a molten state by means of a high-frequency induction coil disposed thereabout. The rod may be formed by passing a monocrystalline seed through a space in the induction coil to contact the molten end of the bar forming a fused junction and retracting the seed downwardly while rotating it to draw the molten bar material between the coils out of the heated area to form a recrystallized rod. An additional heating coil may be positioned beneath the angularly disposed bar and heating coil to provide a slow and controlled heat recession during the solidification of the rod and the rod may be gas-doped by introducing the gas into the area of the molten zone.



PATENTED SEP 21 1971

3,607,109

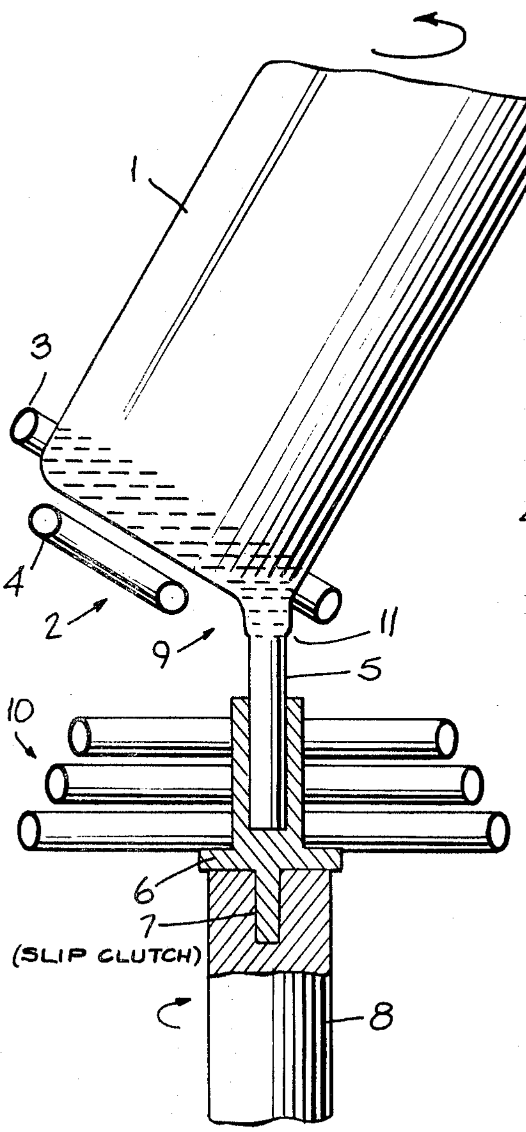


Fig. 1.

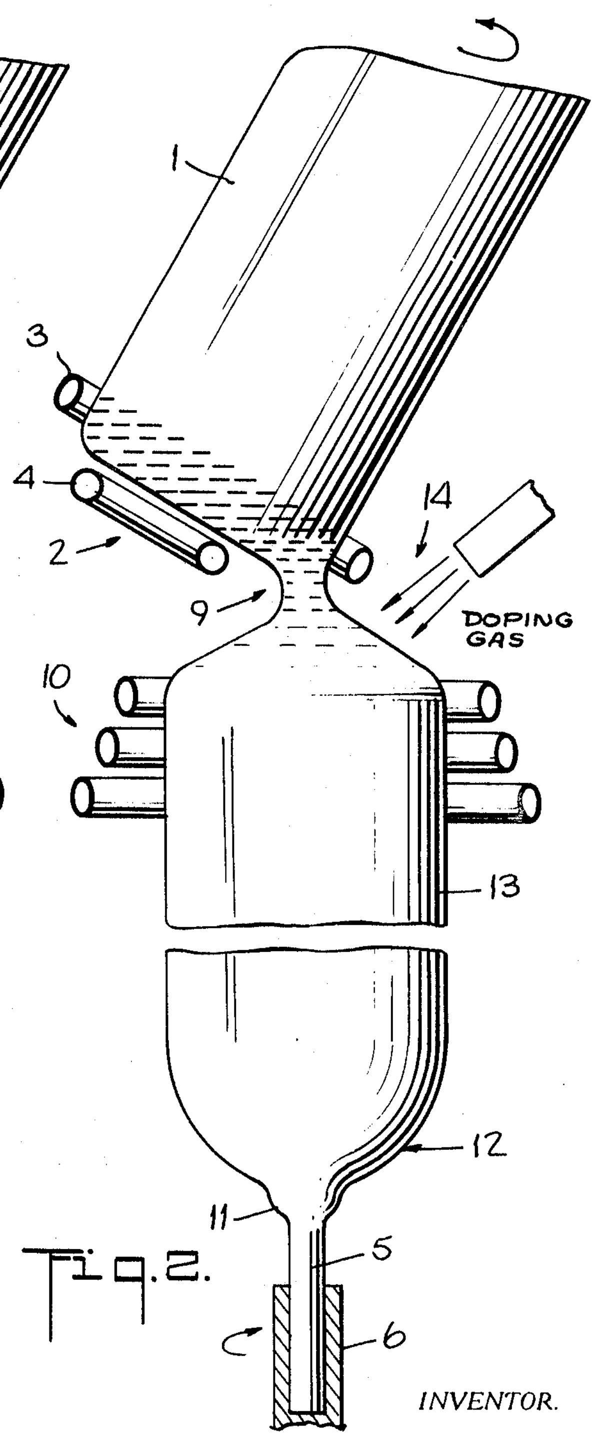


Fig. 2.

INVENTOR.

EMIL R. CAPITA

BY

Norman N. Holland

ATTORNEY

METHOD AND MEANS OF PRODUCING A LARGE DIAMETER SINGLE-CRYSTAL ROD FROM A POLYCRYSTAL BAR

BACKGROUND OF THE INVENTION

The present invention relates to an improved method and apparatus for manufacturing semiconductor materials and more particularly for manufacturing a large diameter monocrystalline or single-crystal rod from a bar of polycrystalline material.

The term "bar" herein shall be used exclusively in connection with the supply of unmelted polycrystalline material and the term "rod" shall be used in connection with the supply of resolidified monocrystalline material merely in an attempt to avoid confusion between the two, and in no way should these terms be interpreted to indicate the relative diameters or other dimensional features of these members.

Two well-known methods of manufacturing semiconductors of materials such as silicon and germanium involve the melting of a supply of the material in polycrystalline form, contacting the molten material with a monocrystalline particle or seed and retracting the seed to draw or pull the molten material with it, thereby resolidifying the melted material and producing a semiconductor material in monocrystalline form. The earliest and a still popular method consists in the melting of the material in a heated crucible, and the dipping of the seed therein. However, an alternate method was subsequently developed wherein a bar of the material is held only at its ends preferably in a vertical position and a molten zone produced in the bar extending through its cross section. This molten zone is gradually passed along the bar by relatively moving the energy source, which produces the heat to melt the zone, and the bar parallel to the bar axis. The molten zone is freely supported by the adjacent nonmelted material of the bar. This operation is commonly performed to purify the material causing foreign substances to become dissolved and collect at the ends of the bar material.

This zone melting method is adapted to the production of monocrystals if the end of the polycrystalline bar is placed into contact with a monocrystalline rod, or merely a piece or seed of the same material arranged in a suitable crystal orientation, and the molten zone is produced first at the junction point and then gradually and successively carried through the polycrystalline bar. The bar material crystallizes at the rear side of the molten zone in monocrystalline condition.

The zone melting method is also particularly conducive to the doping of semiconductor rods with the desired impurities. The doping substances are introduced into the molten zone and then become part of the recrystalizing semiconductor material at the rear side of the molten zone. This method produces a material of a higher degree of purity and an undisturbed monocrystal formation as there is little possibility of undesirable impurities diffusing into the molten zone such as from the walls of a vessel which may occur during the crucible melting operation. However, the zone melting method has been technologically more difficult to carry out as the freely floating liquid material may easily drop off.

A further disadvantage of the zone method is the limitation on the size of the monocrystalline rod which can be produced. In present practice the bar of material to be melted is generally maintained in the vertical position and the heating means such as a high-frequency induction coil surrounding the bar is moved up and down along its length to produce the molten zone; or, alternatively, the coil is fixed and the bar moved through it. In either event, the use of this coil prevents significant enlarging of the diameter of the resulting monocrystalline rod as the possible diametrical enlargement of the recrystallized rod over that of the bar is limited by the diameter of the heating coil. In addition, with this method a high degree of heat is required to melt the bar completely through to the core to insure that the entire zone of material becomes molten and subsequently recrystallized. The present invention is designed to overcome these various problems presented by the prior method of zone melting and is adapted to permit the resulting monocrystalline rod to be of a much larger diameter than the bar to be melted while requiring less heat to be used in the process.

SUMMARY OF THE INVENTION

The improved method and means of the present invention comprises the arranging of the axis of the polycrystalline bar to be melted, at an angle with respect to the axis of the monocrystalline seed. This permits the end of the bar to be heated by an induction coil disposed thereabout, insuring melting of the entire cross section of the bar. Also, the coil elements may be spaced, permitting the molten portion of the bar end to pass through the space in the induction coil before resolidifying. With this arrangement it is possible to construct the monocrystalline cord of any desired diameter, as the limiting dimensions of the heating coil no longer interfere with the formation of the resolidified monocrystalline material.

It is therefore an object of the present invention to provide a method and means for producing a monocrystalline rod from a polycrystalline bar without restricting the relative diameters of the members.

It is another object of the present invention to provide a zone method of producing purified semiconducting material, which requires the use of less heat than the prior art methods.

It is another object of the present invention to provide an improved method and means for producing purified monocrystalline semiconducting material, permitting more rapid and improved melting than achieved by the prior art methods.

Other and further objects of the invention will be obvious upon an understanding of the illustrative embodiment about to be described, or will be indicated in the appended claims, and various advantages not referred to herein will occur to one skilled in the art upon employment of the invention in practice.

BRIEF DESCRIPTION OF DRAWINGS

A preferred embodiment of the invention has been chosen for purposes of illustration and description and is shown in the accompanying drawings, forming a part of the specification, wherein:

FIG. 1 shows the molten end of the polycrystalline bar being contacted by a monocrystalline seed in accordance with the present invention.

FIG. 2 is a showing as in FIG. 1, after the monocrystalline seed has been retracted to form a portion of the resolidified monocrystalline rod.

DESCRIPTION OF THE PREFERRED EMBODIMENT

FIG. 1 shows a bar of polycrystalline semiconducting material **1** having a high frequency induction heating coil **2** disposed at its end. One or more of the coil elements **3** may be arranged to surround the bar **1** and at least one of the elements **4** is disposed directly beneath the end. This arrangement insures heating of the bar material throughout the entire cross section at the end, resulting in the melting of a zone covering the entire tip of the bar. It will be seen that less heat is required with this arrangement to melt the bar through its entire cross section than is required by the prior art method of heating about the lateral circumference of the bar to produce the molten zone.

A monocrystalline seed **5** of the same material as the polycrystalline bar **1** is held in a crystal holder chuck **6**, which is mounted by means of a slip clutch **7** on a shaft **8** that may be rotated and controlled in up and down movement. A space **9** is provided between the coil elements **3** surrounding the bar **1** and the elements **4** disposed beneath its end to permit the monocrystalline seed **5** to pass therethrough and contact the end of the bar **1**. It will be noted this operation is performed while arranging the axis of the polycrystalline bar **1** at an angle with respect to the axis of the seed **5** and shaft **8**. It has been found to be preferable to maintain the seed and shaft axis in a vertical direction while tilting the bar axis from the vertical, as the flow of the molten material is assisted by gravity in this orientation. However, it is within the purview of the present invention to orient the particular elements in various other angular arrangements to essentially accomplish the same results.

An additional heating coil **10** is disposed beneath the end of the bar **1** and coil **2** to provide a slow and controlled heat recession during the resolidification process as the molten material passes therethrough, as shown in FIG. **2**.

The method of the present invention is performed in the following manner. The end of the polycrystalline rod **1** is preheated to produce the initial molten condition. This may be accomplished by inserting a tantalum disc in the area so that the radiated heat will cause the polycrystalline material to become conductive to a point where the high frequency power will take over an induce into the material directly.

When the end of the bar **1** is completely melted the monocrystalline seed **5** is passed up through the space **9** in the coil elements **3** and **4** and contacts the molten lower edge of the bar **1**. This contact results in the formation of a fused junction **11** between the two materials. During this operation the shaft **8**, including the seed **5** and clutch **7** are rotating. After a suitable junction has been achieved, additional molten material will be caused to flow into this melted area. The seed **5** is then slightly retracted and the bar **1** is proportionately fed to the junction area so as to build up a cone-shaped portion of resolidified material, as shown at **12** toward the bottom of FIG. **2**. An up-and-down motion of the shaft **8** while feeding the bar material **1** is continued until the desired rod diameter is obtained. Thereafter the shaft **8** is lowered at a predetermined rate and the bar **1** is fed at a predetermined rate to produce a uniform diameter resolidified monocrystalline rod **13** on the end of the shaft **8**.

The bar **1** may also be rotated in the opposite direction from that of the shaft **8** while performing the method, to keep the molten zone in rapid rotation, thereby providing a thorough mixing of the material therein. This motion may also contribute to increasing the diameter of the recrystallized rod **13** when desired. The seed-holding chuck **6** is mounted on the shaft **8** by means of the slip clutch **7** to provide a release in the event of a freezeup.

While the above operation can be performed in a high vacuum or an inert gas atmosphere, this method is particularly adapted to achieve improved gas doping of the semiconducting material. As shown in FIG. **2**, the doping gas **14** may be introduced into the area between improved heating coils **2** and **10** and the reduced cross section of the molten material at this point permits the gas to be more evenly diffused through the material in the resolidified rod **13**. The gas may also be directed against the lower surface of the molten end of the bar with similar results. The lower heating coil **10**, which as previously stated, provides a slow and controlled heat recession during the solidification cycle also assists in the doping operation by preventing too rapid a solidification of the molten material in this area.

It will thus be seen that an improved method and apparatus is provided for producing a monocrystalline rod of semiconducting material from a polycrystalline bar, wherein by arranging the cooperating elements and apparatus at a suitable angle less heat is required and more rapid and improved melting is achieved, while preventing the diameter of the recrystallized rod from being limited by the dimensions of the heating apparatus. In addition, improved gas doping may be achieved with the arrangement.

As various changes may be made in the form, construction and arrangement of the parts herein and in the steps of the method herein without departing from the spirit and scope of the invention and without sacrificing any of its advantages it is to be understood that all matter herein is to be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim:

1. An apparatus for producing a monocrystalline rod of semiconducting material from a polycrystalline bar comprising:

a. means for supporting the bar at an acute angle with the vertical with at least one end free;

b. spaced high frequency induction coils about and directly beneath the free end respectively to heat the free end to a molten state; the respective coils being in planes parallel to each other;

c. means for placing a vertically oriented monocrystalline seed between the respectively spaced induction coils to contact a molten edge of the bar at its lowest extremity; and

d. means for retracting the seed along a vertical axis thereby drawing the molten bar material out of the heated area to form a recrystallized rod.

2. An apparatus for producing a monocrystalline rod of semiconducting material from a polycrystalline bar comprising:

a. means for supporting the bar at an acute angle with the vertical with at least one end free;

b. spaced high frequency induction coils about and directly beneath the free end respectively to heat the free end to a molten state;

c. means for placing a vertically oriented monocrystalline seed between the respectively spaced induction coils to contact a molten edge of the bar at its lowest extremity;

d. means for retracting the seed along a vertical axis thereby drawing the molten bar material out of the heated area to form a recrystallized rod; and

e. additional high-frequency coils adjacent to the molten zone to provide a slow and controlled heat recession during the solidification of the rod.