March 3, 1970 W. KELLER 3,498,847

METHOD AND APPARATUS FOR PRODUCING A MONOCRYSTALLINE ROD, PARTICULARLY OF SEMICONDUCTOR MATERIAL

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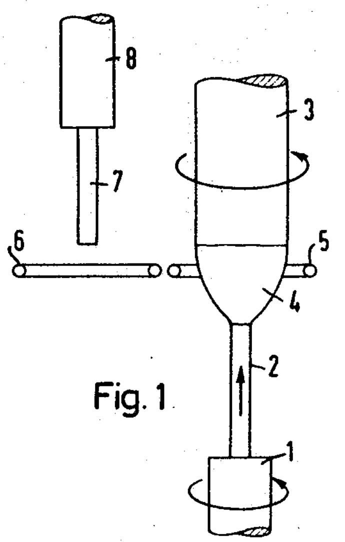

Fig. 1

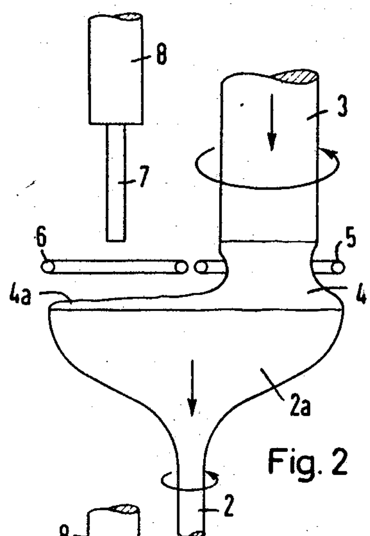

Fig. 2

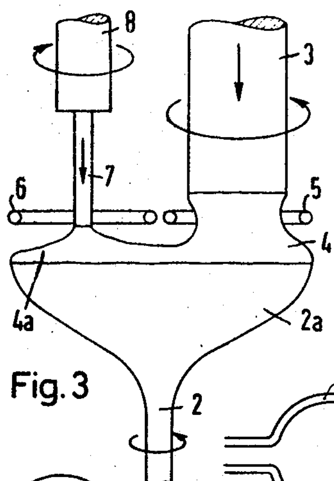

Fig. 3

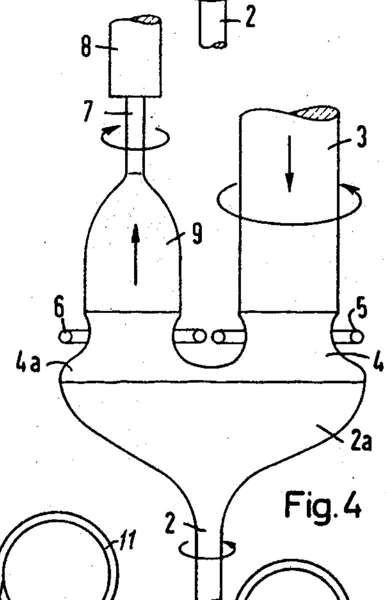

Fig. 4

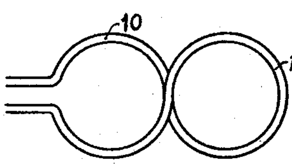

Fig. 5

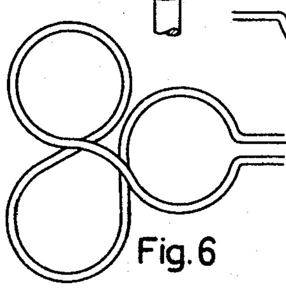
Fig. 6

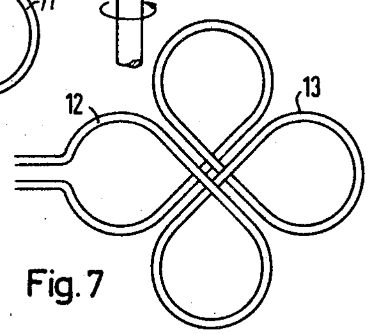

Fig. 7

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METHOD AND APPARATUS FOR PRODUCING A MONOCRYSTALLINE ROD, PARTICULARLY OF SEMICONDUCTOR MATERIAL

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Int. Cl. H01l 7/00
U.S. Cl. 148—1.6 12 Claims

ABSTRACT OF THE DISCLOSURE

Method of producing a rod of monocrystalline material includes melting an end of a crystalline supply rod of the material that is brought in contact with one end of a substantially vertically disposed crystalline rod-shaped starting body of the material supported at the other end thereof, rotating the starting body about the axis thereof and thickening the one end thereof with material melted from the supply rod into a platform surmounted by a melt of the material, dipping a monocrystalline seed crystal of the material into the melt and pulling therewith a rod of monocrystalline material crystallizing from the melt substantially vertically out of the melt while gradually melting the supply rod further to replenish the melt on the platform.

My invention relates to method of producing a monocrystalline rod, particularly of semiconductor material, by pulling the rod from a melt located on a platform, and apparatus for carrying out the method.

It has been known to produce monocrystalline rods by crucible-free floating-zone melting process. In such known processes, a polycrystalline rod, for example, is supported in a vertical position, and a monocrystalline seed crystal which may have a smaller cross section than the polycrystalline rod, is fused to an end of the polycrystalline rod by being heated with an induction heating coil which coaxially surrounds the rod and the seed crystal. The molten zone formed at the fused junction of the polycrystalline rod and the seed crystal is then passed from the junction along and through the polycrystalline rod by relative motion of the polycrystalline rod and the induction heating coil in the axial direction of the polycrystalline rod.

Furthermore, it has also heretofore been known to pull a monocrystalline rod out of a melt contained in a crucible. In such a crystal-pulling process the lower end of a vertically held, rod-shaped monocrystalline seed crystal, which may have a smaller diameter than that of the monocrystalline rod that is to be pulled from the melt, is dipped into the melt. The seed crystal is then rotated about its axis and is pulled from the melt, drawing with it the recrystallizing monocrystalline rod.

In the German published application 1,207,920 there is described a method of producing a rod-shaped monocrystal of semiconductor material, wherein a pill of semiconductor material is placed on the upper surface of a vertical supporting rod of the same semiconductor material, which is provided with longitudinally extending slots, and is melted with an induction heating coil. A monocrystalline rod is pulled from the melt by a vertically disposed seed crystal that has been dipped therein, and the depleted melt is replenished by gradually melting the supporting rod.

Only monocrystalline rods having a diameter not much greater than 25 mm. are capable of being produced by crucible-free zone melting. Moreover, the solidifying front of the rod recrystallizing from the melt is not completely even which can cause the formation of dislocations.

Rod-shaped monocrystals having diameters greater than 25 mm. can be produced, however, by crystal-pulling from a melt in a crucible and, moreover, the recrystallizing front of the rod being pulled is relatively even. Nevertheless, impurities, such as oxygen and atoms of heavy metals which form recombination centers in semiconductor crystals, can diffuse out of the wall of the crucible into the melt contained therein. Furthermore, if the melt is of material having a high melting point, such as silicon for example, the wall of the crucible can become plastically deformable. Consequently, from a practical standpoint, crystal-pulling of semiconductor rods from melts contained in crucibles is limited only to germanium and the so-called $A^{III}B^{V}$ and $A^{II}B^{VI}$ compounds.

With the so-called platform method of the aforementioned German published application 1,207,920, an even recrystallizing front of the monocrystalline rod pulled from the melt is obtained and rods of relatively large diameter can be pulled; however, in this known method there is also always danger of contaminating the pulled rods with atoms of heavy metals which may have been deposited in the longitudinal slots of the supporting rod, particularly when the slots are formed in the supporting rod.

It is accordingly an object of my invention to provide method and apparatus for producing a monocrystalline rod which may have a relatively large diameter and low dislocation density and does not have the concealed danger of undesired impurities in the pulled rods, such as from atoms of heavy metals.

With the foregoing and other objects in view, I provide in accordance with the invention, method for producing a rod of monocrystalline material, which comprises melting an end of a crystalline supply rod of a material that is connected to the upper end of a substantially vertically disposed crystalline rod-shaped starting body of the material supported at the lower end thereof, rotating the starting body about the axis thereof, thickening the upper end thereof with material melted from the supply rod into a platform surmounted by a melt of the material, dipping a monocrystalline seed crystal of the material into the melt and pulling therewith a rod of monocrystalline material crystallizing from the melt vertically or nearly vertically out of the melt, while gradually melting the supply rod further to replenish the melt on the platform.

In accordance with a further feature of the method of producing monocrystalline rods with relatively large diameters, the crystalline starting body is displaced in a direction parallel to its axis and in the axial directon thereof from the juncton thereof with the supply rod for the purpose of building up the platform, while the supply rod is displaced in the axial direction thereof toward the junction.

In accordance with the features of the apparatus of the invention I provide an induction heating coil having a plurality of turns disposed adjacent one another in a substantially common plane. More specifically, in accordance with a feature of the apparatus, the heating coil has the general shape of a clover.

Other features which are considered as characteristic for the invention are set forth in the appended claims.

Although the invention is illustrated and described herein as embodied in a method and apparatus for producing a monocrystalline rod, particularly of semiconductor material, it is nevertheless not intended to be limited to the details shown, since various modifications and structural changes may be made therein without departing from the spirit of the invention and within the scope and range of equivalents of the claims.

The construction and method of operation of the invention, however, together with additional objects and advantages thereof will be best understood from the following description of specific embodiments when read in connection with the accompanying drawings, in which:

FIGS. 1–4 are diagrammatic views of the apparatus showing individual steps of the method for producing a monocrystalline rod in accordance with my invention; and

FIGS. 5–7 are plan views of different embodiments of the induction heating coil of the apparatus employed in carrying out the method of the invention.

Referring now to the drawing and first particularly to FIG. 1 thereof, there is shown a vertically disposed seed crystal **2** which serves as crystalline starting body and which need not be of monocrystalline material, which is clamped in a holder **1** that is rotatable about its axis, as shown by the curved arrow in the figure, and is displaceable in the axial direction thereof, as shown by the associated straight arrow in the figure. A polycrystalline supply rod **3**, which is vertically disposed and rotatably clamped in a non-illustrated holder located at the upper end thereof and is displaceable in the axial direction thereof, is fused at the lower end thereof to the upper end of the seed crystal **2** by means of an induction heating coil **5** which surrounds the lower end of the supply rod **3** and forms a molten zone **4** therein. Adjacent the polycrystalline supply rod **3**, there is located vertically disposed monocrystalline seed crystal **7** which is clamped at its upper end in a holder **8** and is rotatable about the axis thereof and displaceable in the axial direction thereof. An induction heating coil **6** is located coaxially to the seed crystal **7** adjacent the induction heating coil **5** and is located substantially at the same level or in the same plane as the induction heating coil **5**. The induction heating coils **5** and **6** can be, for example, cylindrical coils having one to three windings and may be energized by a single or two separate high-frequency generators.

After the polycrystalline supply rod **3** has been fused to the seed crystal **2**, the supply rod **3** and the seed crystal **2** are set in rotation, and the holder **1** is displaced laterally toward the induction heating coil **6** for a distance which is substantially equal to half the distance between the center points of the interior space within both heating coils **5** and **6**. Simultaneously, the polycrystalline rod **3** is displaced in the axial direction thereof toward the heating coil **5** and the seed crystal **2** is disposed in the axial direction thereof away from the heating coil **5**, as shown in FIG. 2. Thus the platform **2a** is formed thereby and supports the melt from the molten zone **4** thereon. A portion **4a** of the molten zone is located beneath the opening or interior space of the induction heating coil **6** which also heats this portion **4a** of the molten zone. Then the monocrystalline seed crystal **1** clamped in the holder **8** is also set into rotation about its axis, as shown in FIG. 3, and is dipped with its free end into the portion **4a** of the melt located on the platform **2a**.

Thereafter the holder **8** with the monocrystalline seed crystal **7** is moved upwardly, as shown in FIG. 4 by the associated straight arrow, in the axial direction thereof away from the induction heating coil **6** and a monocrystalline rod **9** recrystallizing from the melt portion **4a** is pulled through the opening or interior space of the induction heating coil **6**. The molten zone **4** is continuously replenished by the material of the polycrystalline rod **3** which is displaced in the axial direction thereof toward the induction heating coil **5** and is accordingly melted thereby. The induction heating coil **6** can be energized with a weaker current than the current energizing the induction heating coil **5** because recrystallization heat is produced at the recrystallization front of the rod **9** growing or pulled from the melt portion **4a**.

The induction heating coils **5** and **6** can be replaced by a single coil of the type shown in FIG. 5, for example, which has a pair of adjacent windings together forming a figure eight. One loop or turn **10** of the induction heating coil having the shape of figure eight, as shown in FIG. 5, accordingly corresponds to the induction heating coil **6** of FIGS. 1–2, while the other loop or winding **11** corresponds to the induction heating coil **5** of FIGS. 1–4. The melt of the molten zone **4** located on the platform **2a** can be replenished also by gradual melting of one or more additional supply rods surrounded, if desired, by induction heating coils. One or more of these additional supply rods can have a dopant content or can consist of dopant material. In this regard, it is desirable also to heat the ends of the additional supply rods which are in contact with the molten zone **4** by means of induction heating coil, for example. The additional supply rods can also be rotated about the axis thereof. It is also advantageous, in accordance with my invention, to employ a single heating coil having the shape of a three-leaf clover according to FIG. 6, the loops or windings thereof respectively surrounding the end of a supply rod and the end of the rod being pulled from the melt.

It is also within the scope of my invention to clamp the rod-shaped crystalline starting body at the upper end thereof and to form the platform at the lower end thereof. In such case, the polycrystalline supply rod is end-supported at its lower end in a rod holder and is fused at its upper end to the crystalline rod-shaped starting body. The melt is supported or levitated by means of at least one induction heating coil located below the platform and energized by high frequency alternating current and is heated thereby. The windings of this induction heating coil are located in a plane substantially parallel to the surface of the melt. The monocrystalline rod is pulled vertically downwardly out of the melt. It is particularly desirable to employ an induction heating coil which has the shape of a 4-leaf clover, as shown in FIG. 7, and which has a loop or winding **12** surrounding the supply rod and another loop or winding **13** surrounding the monocrystalline rod which is being pulled out of the melt.

The advantages achieved by the invention of this application reside particularly in that the recrystallization front of the pulled rod **9** is substantially even at the molten zone **4** and therefore only few dislocations are formed in the pulled rod **9**. Furthermore, the melt in the molten zone **4** has a uniform temperature because it is also heated in the interior thereof by the induction heating coils **5** and **6**, and the rotations of the platform **2a** and polycrystalline rod **3** located eccentrically to the platform **2a** produce a thorough mixing of the melt. This not only results in a low dislocation density in the pulled rod **9**, but also produces a uniform distribution of dopants in the melt of the molten zone and thereby also in the pulled rod **9**. In addition, any mechanical treatment or processing of the platform supporting the melt of the molten zone **4** which produces a source of undesired impurities is thereby avoided.

I claim:

1. Method of producing a rod of monocrystalline material which comprises melting an end of a crystalline supply rod of the material that is brought in contact with one end of a substantially vertically disposed crystalline rod-shaped starting body of the material supported at the other end thereof, rotating the starting body about the axis and thickening the one end thereof with material melted from the supply rod into a platform surmounted by a melt of the material, dipping a monocrystalline seed crystal of the material into the melt and pulling therewith a rod of monocrystalline material crystallizing from the melt substantially vertically out of the melt while gradually melting the supply rod further to replenish the melt on the platform.

2. Method according to claim **1**, wherein the end of the crystalline supply rod is brought in contact with the upper end of the starting body, the starting body is rotated about the axis thereof, and the upper end of the starting body is thickened with material melted from the supply rod.

3. Method according to claim 1, wherein the starting body is supported at the upper end thereof, the end of the crystalline supply rod is brought in contact with the lower end of the starting body, the starting body is rotated about the axis thereof, and the lower end of the starting body is thickened with material melted from the supply rod so as to form the platform with the melt disposed thereon beneath the same, and which includes levitating the melt with at least one induction heating coil energized with high frequency alternating current and located beneath the platform, and pulling the monocrystalline rod crystallizing from the melt in a substantially vertically downward direction out of the melt.

4. Method according to claim 1, which includes forming a platform by displacing the starting body parallel to itself and in the axial direction thereof away from the junction thereof with the supply rod while displacing the supply rod in the axial direction thereof toward the junction.

5. Method according to claim 1, which includes rotating the supply rod about the axis thereof.

6. Method according to claim 1, wherein the supply rod contains dopant.

7. Method according to claim 1, which includes heating the rod pulled from the melt at the end thereof in contact with the melt.

8. Method according to claim 1, which includes rotating the rod pulled from the melt about the axis of the pulled rod.

9. Method according to claim 1, which includes gradually melting at least one additional supply rod in contact with the one end of the starting body so as to replenish the melt on the platform.

10. Method according to claim 9, wherein at least one of the additional supply rods consists of dopant.

11. Method according to claim 9, which includes heating the additional supply rod at the end thereof in contact with the melt.

12. Method according to claim 9, which includes rotating the additional supply rod about the axis thereof.

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U.S. Cl. X.R.

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