United States Patent

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[33] **Germany**
[31] **S 107671**

[54] **METHOD FOR PRODUCING A MONOCRYSTALLINE ROD BY CRUCIBLE-FREE FLOATING ZONE MELTING**
7 Claims, 12 Drawing Figs.

[52] **U.S. Cl.** ..... **23/301 SP,** 23/273 SP, 219/10.79
[51] **Int. Cl.** **B01j 17/18,** B01j 17/10
[50] **Field of Search** ..... 23/273 SP, 301 SP; 219/10.79

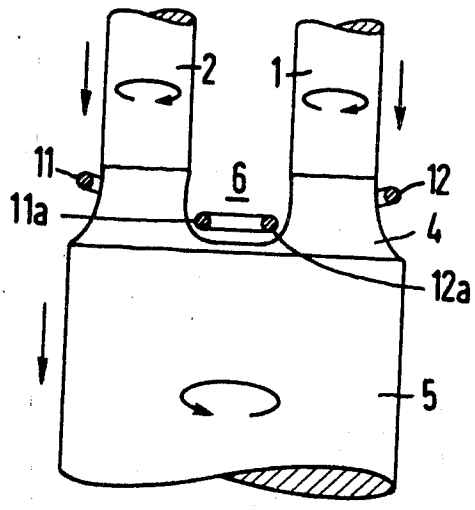
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ABSTRACT: Method for producing a monocrystalline rod, such as a semiconductor rod, by crucible-free zone melting includes heating to melting temperature and end of at least two supply rod portions of given material supplied at spaced locations to a melting zone of the material, and additionally heating the melting zone with at least one additional heat source disposed within the space between the supply rod portions being supplied to the melting zone.



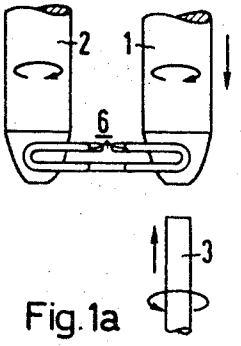


Fig. 1a

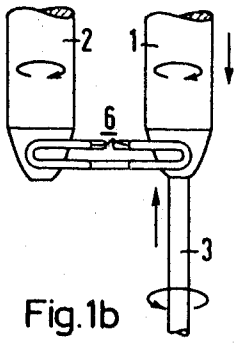


Fig. 1b

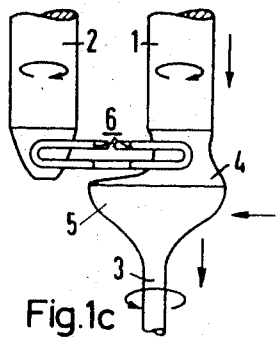


Fig. 1c

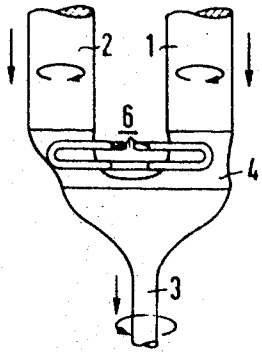


Fig. 1d

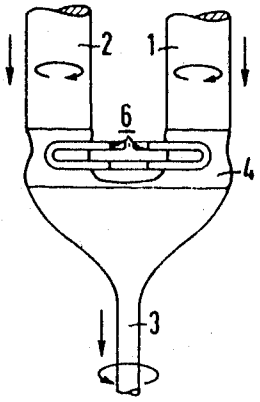


Fig. 1e

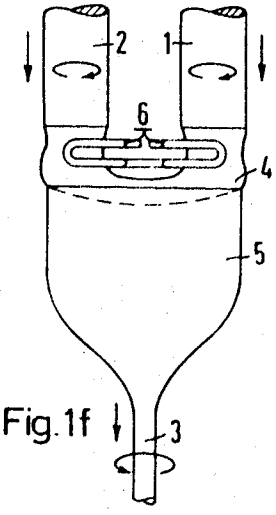


Fig. 1f

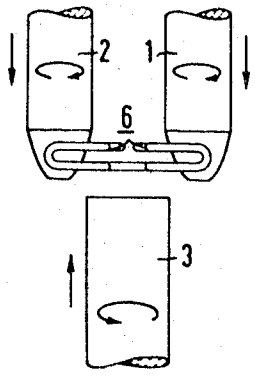


Fig. 2a

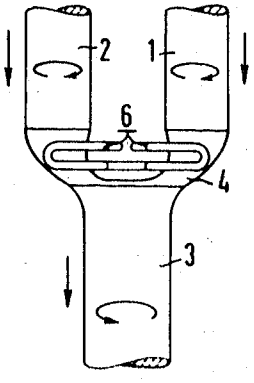


Fig. 2b

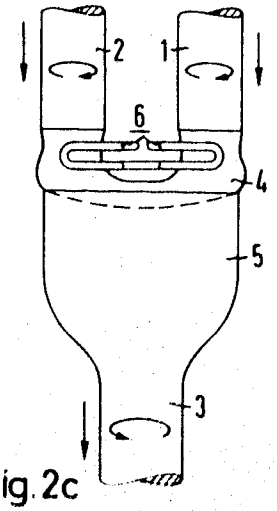


Fig. 2c

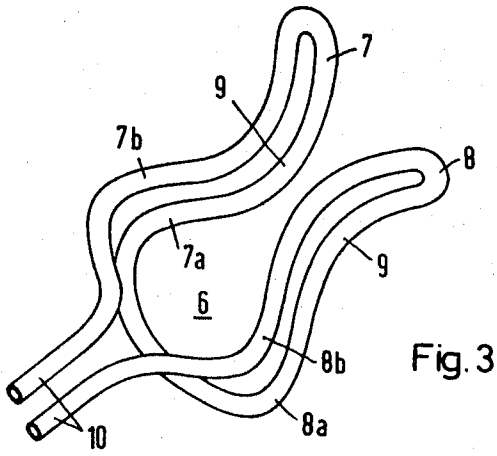


Fig. 3

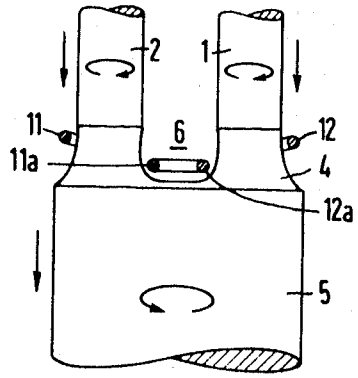


Fig. 4a

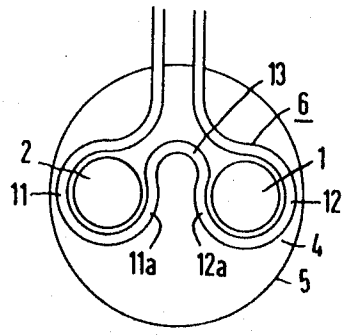


Fig. 4b

METHOD FOR PRODUCING A MONOCRYSTALLINE ROD BY CRUCIBLE-FREE FLOATING ZONE MELTING

My invention relates to method for producing a monocrystalline rod by crucible-free floating zone melting.

For a relatively long time now, the crucible-free floating zone melting process has proved successful in growing rod-shaped semiconductor crystals from which electronic semiconductor components are to be produced. In such a process, a monocrystalline seed crystal of relatively small diameter when compared to the diameter of a given rod-shaped polycrystalline semiconductor member, is fused for example by means of an induction heating coil to an end of the rod shaped semiconductor member. Thereafter, the melting zone formed when the seed crystal is joined to the rod-shaped member is passed in a direction from the fused junction at least once through the rod-shaped semiconductor member. The polycrystalline semiconductor member is thereby transformed into a monocrystal. Moreover, impurities are separated from the semiconductor member or deposited at one end of the semiconductor member. Semiconductor material employed for producing electronic semiconductor components must not only be monocrystalline and free of impurities forming recombination centers to a very high degree but also must have as few dislocations as possible which might impair the homogeneity of a semiconductor member and therewith the electrical characteristics of the semiconductor component produced therefrom. The dislocations especially reduce the durability or effective longevity of the minority carriers in the semiconductor material. The last mentioned requirement, namely the zone-melting production of dislocation-free monocrystals, is able to be fulfilled particularly for semiconductor rods or relatively large radial dimensions such as for example a diameter of more than 35 mm. and more specifically for diameters of 50 mm., only after overcoming great difficulties. More particularly, great care has to be taken that thermal stresses in the semiconductor rod be widely avoided when cooling the semiconductor rod.

It has been proposed heretofore to produce semiconductor rods with relatively large radial dimensions, for example, with rod diameters of 50 mm. and more, of good crystal quality, mainly with a relatively small dislocation density, and with a doping over the rod cross section and accordingly with a radial resistance distribution which are as uniform as possible, by supplying two or more rods to the melting zone, the rods extending in a direction eccentric to the axis of the resolidifying rod portion.

It has been found that even if there is a uniform temperature with the entire melting zone, optimal results will not always be obtained when zone-melting crystalline rods, particularly those having relatively large radial dimensions. It is rather more desirable in many cases to heat the central region of the melting zone to a somewhat higher temperature than the marginal region thereof. This requirement cannot be fulfilled with conventional heating devices, chiefly inductive heating devices with one or more heating coils annularly surrounding the crystalline or semiconductor rod.

It is accordingly an object of my invention to provide method for crucible-free or floating zone melting a crystalline rod which avoids the aforementioned disadvantages of the heretofore known devices and which moreover particularly avoids the production of thermal stresses causing imperfections in the quality of the monocrystal which is produced.

With the foregoing and other objects in view, I accordingly provide such a method comprising inductive heating means having at least one conductive heating loop extending into a space between at least two supply rod portions, which supply semiconductor material at spaced locations to the melting zone, for heating the end of the supply rod portions to melting temperature.

In accordance with a further feature of my invention, the conductive loop is disposed in a plane parallel to the plane of the melting zone for additionally heating the central region of the melting zone located between the supply rod portions.

In accordance with yet another feature of the invention, means are provided for adjusting the intensity of the additional heating of the melting zone so that the solid-liquid interface or phase boundary of the recrystallizing rod portion is matched or conformed to the course of isothermal lines formed in the part of the recrystallizing rod portion located adjacent the melting zone. Thereby, even for rods having relatively large radial dimensions, no significant thermal stresses will occur during recrystallization of the resolidifying rod portion.

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 device for producing a crystalline rod by crucible-free floating zone melting, 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*a* to 1*f* are diagrammatic views of assembled crystalline rod portions and an inductor heating device suitable for carrying out a floating zone-melting process, showing different phases of the process wherein the crystallizing rod portion is displaced laterally relative to the rod portion supplied to the melting zone and to the heating device;

FIGS. 2*a* to 2*c* are diagrammatic views of crystalline rod portions and an inductive heating device in different phases of another zone-melting process wherein a seed crystal is disposed coaxially to the heating device;

FIG. 3 is a perspective view of an inductive heating device suitable for carrying out the zone-melting processes depicted in the preceding figures; and

FIG. 4*a* and 4*b* are cross-sectional and plan views, respectively, of another embodiment of the heating device of FIG. 3 as shown in operation with the crystalline rod portions.

Referring now to the drawings and first particularly to FIG. 1*a* thereof there is shown a pair of supply rods 1 and 2, and a monocrystalline seed crystal 3 disposed coaxially to the supply rod 1. The rods 1 and 2 and the seed crystal 3 can suitably be formed of a semiconductor material such as silicon for example. The end of the supply rod 1 located adjacent an end of the seed crystal 3 is heated by a heating device 6 to the melting temperature of the crystalline material. As the end of the supply rod is being melted, the supply rod 1 and the seed crystal 3 are displaced relatively toward one another and fused to one another, a condition illustrated in FIG. 1*b*. The supply rod 1 and the seed crystal 3, as shown in FIG. 1*b*, are moreover rotated about their axes preferably in opposite rotary directions as indicated by the arrows associated therewith. When the heating device 6 is maintained stationary, the supply rod 1 and the seed crystal 3 are both displaced in the same vertical direction as indicated by the straight arrows in FIG. 1*c*, but at different speeds, however. The vertical displacement speeds of the supply rod 1 and of the seed crystal 3 are adjusted so that the radial dimensions of the rod portion 5 recrystallizing from the melting zone 4 continually increase. In addition, the seed crystal 3 executes a lateral displacement in a direction toward the left-hand side of FIG. 1*c*, as shown by the arrow on the right-hand side of the figure so that the melting zone 4 gradually moves in a direction toward the previously melted end of the other supply rod 2. As soon as the melting zone 4 reaches the illustrated molten end of the supply rod 2, the rod 2 is also fused with the melting zone 4, is displaced in the direction of its own axis and is rotated about its axis as indicated by the associated arrows in FIG. 1*d*. The seed crystal 3 and the recrystallizing rod portion 5 continue to be laterally displaced in a direction toward the left-hand side of FIG. 1*d* until, as shown in FIG. 1*e*, the desired thickness of the

recrystallizing rod portion 5 is attained. From that moment on, the rod portions 1, 2, and 5, while maintaining their rotational movement, are displaced only in the axial direction thereof as shown in FIG. 1*f*, the portion 5 being no longer displaced laterally. An inductive heating coil is provided as the heating device 6 for heating to melting temperature the illustrated ends of the supply rod portions 1 and 2 which are supplied to the melting zone 4, the heating coil 6 having at least one additional loop extending into the space between the rod portions 1 and 2. A more detailed description of the configuration of the heating device 6 will be given hereinafter with regard to FIGS. 3 and 4*a* and 4*b*.

The hereinbefore described zone-melting process can be modified in accordance with the phases depicted in FIGS. 2*a* to 2*c* of the drawing, wherein both supply rods 1 and 2 are associated with a comparatively thick seed crystal 3. The thickness of the seed crystal 3, as shown in FIG. 2*a*, is at least as great as the spacing between the rod portions 1 and 2, and is disposed substantially symmetrically to the rod portions 1 and 2. After the ends of the rod portions 1 and 2 are melted as shown in FIG. 2*a*, they can be simultaneously or at least nearly simultaneously fused with the seed crystal 3 as shown in FIG. 2*b*. When the heating coil 6 is held stationary, the axial displacement speeds of the rod portions 1, 2 and 5 are adjusted to one another so that the thickness of the recrystallizing rod portion 5 attains a predetermined nominal value. The use of a seed crystal 3 which bridges the spacing between both rod portions 1 and 2, provides the advantage that the time required for beefing up the recrystallizing rod portion 5 to a predetermined nominal value is thereby reduced. The aforementioned heating device 6 which will be hereinafter described in greater detail can be employed for heating the ends of the rod portions 1 and 2 of FIGS. 2*a* to 2*c* as well as the central melting zone region lying between these rod portions 1 and 2.

In the enlarged perspective view of FIG. 3 there is shown an embodiment of the inductive heating device 6, which is formed of two needlelike or hairpin-shaped inductors 7 and 8 which are curved so that they are disposed in a substantially semicircular form respectively about the supply rods 1 and 2. The hairpin inductors 7 and 8 simultaneously form the additional inner conductive loop 9 for additionally heating the central melting zone region located between both rod portions 1 and 2. The portions 7*a* and 8*a* of the hairpin-shaped inductors 7 and 8, which are located adjacent the recrystallizing rod portion 5, are connected to one another to form the electrically conductive loop 9. The parts 7*b* and 8*b* of the needle-shaped or hairpin-shaped inductors 7 and 8 which are farther removed from the recrystallization rod portion 5 than the parts 7*a* and 8*a* are provided with current-connecting leads or terminals 10. In order to ensure sufficient heating of the rod portion 1 and 2, the spacing between the parts 7*a* and 7*b* as well as between the parts 8*a* and 8*b* of the hairpin-shaped inductors 7 and 8 is at least 1½ times the diameter of the current-conducting lead portions. To strengthen the heating effect, both the inductors 7 and 8 as well as the inner loop 9 can be provided with several windings. It is also possible to heat the central region of the melting zone 4 by means of an additional inductor heating device or also another type of energy source such as a heat-radiating device or an electron beam device for example. If an additional inductive heating device is provided, an advantage is afforded in that when a separate energy source is employed, the energy acting on the central melting zone region between the rod portions 1 and 2 is able to be better controlled. Accordingly it is possible for the solid-liquid interface or phase boundary of the melting zone 4 to assume the profile of a substantially flat plate as is shown by the dotted lines in FIGS. 1*f* and 2*c*. Such a course of the solid-liquid phase boundary of the melting zone 4 widely corresponds to the isothermal lines formed in the part of the recrystallizing rod portion 5 located adjacent the melting zone 4. Accordingly, during recrystallization of the rod portion 5, thermal stresses are greatly avoided even for rods with very large radial dimensions. This produces a recrystallizing rod portion 5 which is virtually free of dislocations and thereby has good crystal quality. In accordance with a further modification of the illustrated embodiment of the heating coil 6, both needlelike or hairpin-shaped inductors 7 and 8 can be connected in parallel. Accordingly, the parts 7*a* and 8*b* on the one hand and the parts 7*b* and 8*a* on the other hand can be connected respectively to a common current lead. It is also possible to provide both inductors 7 and 8 with separate current junctions or terminals and to also separately energize them. Thereby, the inner electrically conductive loop can be formed by a separately energizable flat coil having one or more windings.

In FIGS. 4*a* and 4*b*, there is shown a further advantageous embodiment of an inductive heating device in accordance with my invention.

The heating device 6 of FIGS. 4*a* and 4*b* has two electrically conductive loops 11 and 12 virtually encircling the rod portions 1 and 2 respectively. The conductive loops 11 and 12 form an inner conductive loop 13 from portions 11*a* and 12*a* respectively thereof, the inner loop 13 being located in the space behind the rod portions 1 and 2. The coupling of the conductive loop 13 and the melting zone 4 can be affected desirably so that the electrically conductive loops 11 and 12 on the one hand and loop 13 on the other hand are disposed in planes that are inclined relative to one another as shown more particularly in FIG. 4*a*. It is also desirable for the conducting loop 13 to be disposed in a plane parallel to the melting zone 4 and the conducting loops 11 and 12 to be disposed in planes inclined thereto whereby, when viewed in cross section as shown in FIG. 4*a*, the loops 11 and 12 and the inner loop portions 11*a* and 12*a* form a trapezoid, the smaller side of the two parallel sides of the trapezoid being defined by the conductive portions 11*a* 12*a*. The conductive loops 11, 12 and 13, as mentioned hereinbefore, can have a plurality of windings. It is also possible to provide the rod portions 1 and 2 with respective circular inductors, and to heat the central region of the melting zone between these rod portions 1 and 2 by means of an additional inductive heating device such as for example a flat coil.

As mentioned hereinbefore, further features of the herein described and illustrated embodiment can be modified within the scope of the invention. More particularly, the rod portions 1 and 2 supplied to the melting zone 4 can have various dimensions. They can also be supplied at different speeds to the melting zone 4. The rod portions 1 and 2 can also be disposed inclined to the axis of the recrystallizing rod portion 5 and, more specifically in such a way that the rod ends merge or intersect in the vicinity of the melting zone 4. Moreover, more than two rod portions can be supplied to the melting zone 4. The described heating devices can also be employed with advantage when the recrystallizing rod portion 5 is located above the heating device 6 and the melting zone 4 is passed in a direction from above downwardly through the rods 1 and 2.

I claim:

1. Method for producing monocrystalline rod, such as a semiconductor rod, by crucible-free zone melting which comprises heating to melting temperature an end of at least two supply rod portions of given crystalline material, contacting at least one of the molten ends of the supply rod portions with an end of a monocrystalline seed crystal of the given material so as to form a melting zone of the material at the end of the seed crystal, relatively displacing the seed crystal and the supply rod portions to as to add a replenishing supply of the given material at spaced location to the melting zone opposite the seed crystal and so as to pull from the melting zone a rod of monocrystalline material fused to the seed crystal, and additionally heating the melting zone with at least one additional heat source disposed within the space between the supply rod portions being supplied to the melting zone.

2. Method according to claim 1 wherein said heating to melting temperature is by induction, and said heat source comprises an electrically conductive heating loop.

3. Method according to claim 2 wherein the inductive heating is effected by a plurality of electrically conductive heating loops having axes inclined with respect to one another.

4. Method according to claim 1 which comprises placing at least two electrically conductive heating loops substantially around, respectively, each of the supply rod portions being supplied to the melting zone, said additional heat source disposed within the space between the supply rod portions being formed of parts of said two heating loops substantially surrounding the respective rod portions.

5. Method according to claim 2 wherein said inductive heating is effected by at least two elongated U-shaped inductive windings respectively disposed substantially in a semicircle about the rod portions being supplied to the melt, said heating loop disposed within the space between the supply rod portions being formed of parts of said two U-shaped inductive windings.

6. Method according to claim 1 wherein said first-mentioned heating step comprises heating the ends of the supply rod portions with at least one conductive heating loop, and said additional heating step comprises heating the melting zone between the supply rod portions with an additional conductive heating loop, separately energizable from said one heating loop, disposed within the space between the supply rod portions.

7. Method according to claim 1 wherein said first-mentioned heating step comprises heating the ends of the supply rod portions with conductive heating loops, and said additional heating step comprises heating the melting zone between the supply rod portions with an additional conductive heating loop disposed within the space between the supply rod portions at a location closer to the melting zone than said first mentioned conductive heating loops.

* * * * *