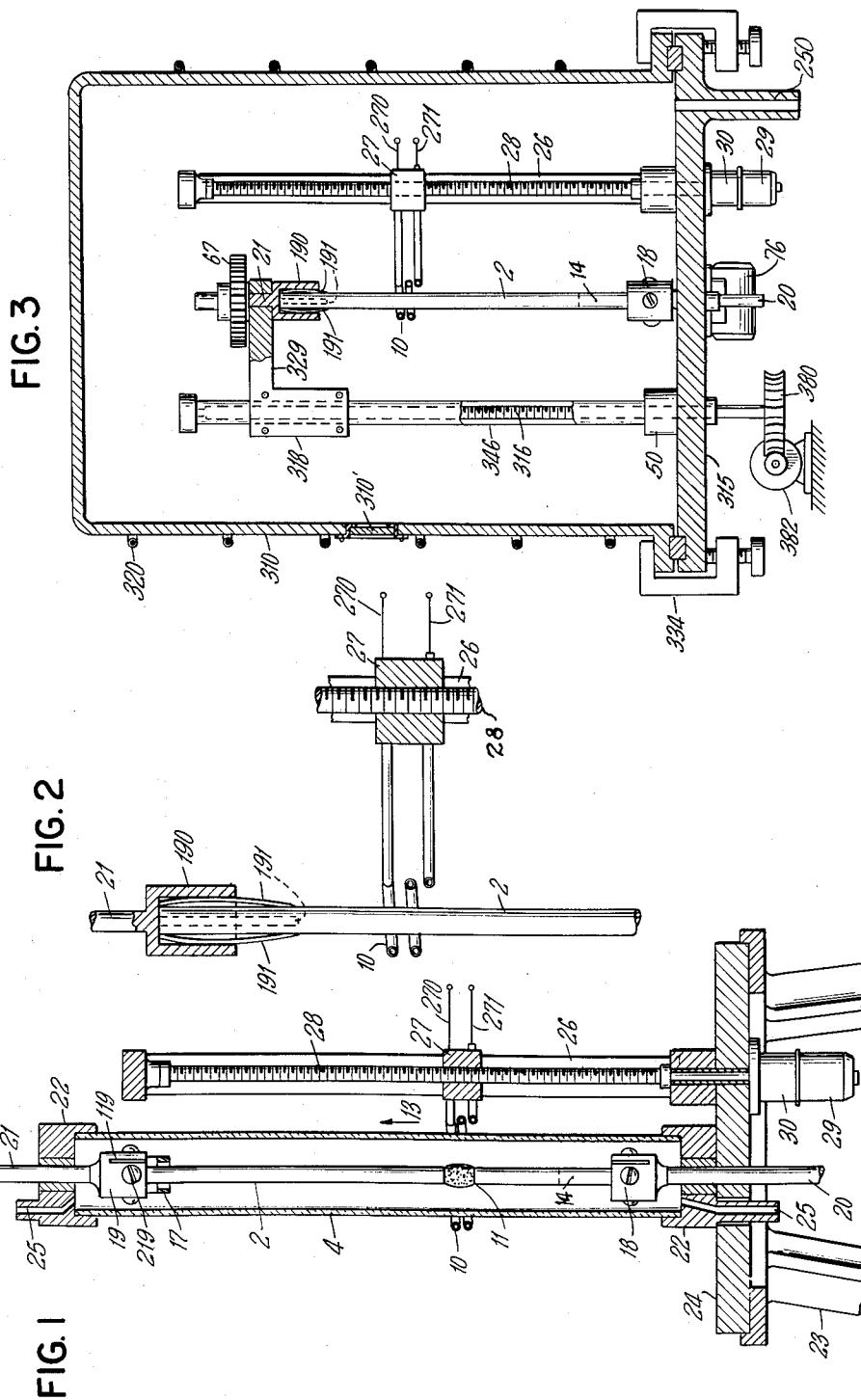


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CRUCIBLE-FREE ZONE MELTING METHOD AND APPARATUS FOR
PRODUCING AND PROCESSING A ROD-SHAPED BODY OF
CRYSTALLINE SUBSTANCE, PARTICULARLY
SEMICONDUCTOR SUBSTANCE
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CRUCIBLE-FREE ZONE MELTING METHOD AND APPARATUS FOR PRODUCING AND PROCESSING A ROD-SHAPED BODY OF CRYSTALLINE SUBSTANCE, PARTICULARLY SEMICONDUCTOR SUBSTANCE

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This invention relates to the treatment of rods of crystalline substance, particularly of semiconductor substance such as germanium or silicon, by means of a crucible free zone melting procedure, to increase the degree of purity, or to obtain a monocrystalline structure.

In my copending application Serial No. 409,610, filed February 11, 1954, of which the present application is, in part, a continuation, I propose the use of inductive heating to melt the semiconductor material in such a process. Since the conductivity of the semiconducting substances at normal or ordinary room temperature does not suffice to permit generating therein the induction currents required for heating, it is advantageous to first pre-heat the semiconducting material by means of an auxiliary heater. This may be accomplished, for example, by means of a ring shaped heater of metal or carbon which is heated to glowing temperature by resistance heating due to flow of electric current, and which acts upon a limited zone of the semiconductor by heat radiation, so as to heat this zone to such a high temperature that the resistance of the semiconducting material declines to a fraction of the original value, and is of a smaller order of magnitude, but the material is not melted. Subsequently, the pre-heated zone is further heated by means of an induction coil, and thus is melted.

According to a more specific aspect of the invention, this method is further improved by having the auxiliary heater itself inductively heated. The auxiliary heater then does not require any leads for the supply of current. The auxiliary heater is preferably heated with the aid of the same induction coil which also serves to subject the semiconductor body itself to zone melting.

The invention will be further explained with reference to the drawing, in which:

Fig. 1 is a vertical section of an apparatus for crucible free zone purification and crystal pulling; and

Fig. 2 is a partial view, partly in vertical section of a modified form, illustrating an improved auxiliary heater device and holder;

Fig. 3 illustrates the form shown in Fig. 2 in assembled relation inside a preferably steel housing; here, in contradistinction to Fig. 1, the induction coil is inside the housing.

The apparatus illustrated in Fig. 1 is similar in many respects to one previously proposed in my copending application Serial No. 409,610, for the crucible-free zone melting of a semiconductor rod, particularly silicon. The semiconductor rod 2 in the device is enclosed within a quartz tube 4, preferably in vertical position.

The rod 2 which, for example, may have a diameter between 3 and 10 mm. is clamped between holders 18 and 19 mounted at the ends of respective separately or conjointly rotatable shafts 20 and 21. The rotatable shaft 20 is vacuum tightly sealed thru the lower socket 22 for the quartz tube 4 and is displaceable independently

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of the rotation. The shaft 21 is rotatable as well as independently displaceable in the axial direction and passes thru the upper socket 22. The sockets 22 are provided with stub pipes 25 thru which the interior of the device can be evacuated and then filled with protective gas. However, pipes 25 can be used to introduce or supply minute, small, or controlled amounts of protective, or of doping, gas or vapor, or other substance. The entire assembly rests upon a base plate 24 and a support 23. Also mounted on the plate and support is a guiding device 26 on which an internally threaded slider 27 is displaceable. The slider can be moved up and down by turning screw spindle 28. The shaft of spindle 28 passes thru the plate 24 and is driven by auxiliary motor 29 thru gear transmission 30, for example, so that the slider travels upwardly or downwardly at a speed of the order of 0.5 to 5 mm. per minute. Fastened to the slider 27 is the induction heater coil 10 which consists, for example, of copper tubing connected to and energized by high frequency current of several thousand cycles per second, and traversed by cooling water. Electrical connectors 270, 271 to the brass slider 27 and to the coil 10 respectively are schematically indicated.

The radiation auxiliary heater 17 in the present case is located in the quartz tube 4. This radiator is mounted near the upper end of the rod 2 and is firmly joined with its holder 19 (in Fig. 1). At the beginning of a pulling operation, the heater coil 10 is moved upwardly, and coaxially of the rod, to its uppermost position so that the ring 17 is located in the field range of the coil. Although the preferred procedure is that described later, the zone melting can commence at the same location and can be carried out in the downward direction. During this latter operation the radiating heater 17 automatically stops operating. Obviously, this entire device of Fig. 1 (and also Fig. 2) can be inverted. Or the radiator 17 can be transferred to lower holder 18. However, two radiators 17 can be provided, each at the respective holders 18, 19, in each of the figures of the drawing.

The above mentioned pre-heating of the semiconductor rod is thus effected by means of the auxiliary heating device 17. This device 17 comprises, for example, a closed ring of sheet metal, of tungsten, molybdenum, nickel or the like. When the ring 17 is located in the field range of the coil 10, an electric current is induced in the ring which heats it up to incandescence. The semiconductor body 2, which in cold condition possesses high resistance, is preheated by the heat radiating from the glowing ring shaped heater 17. The conductivity of the semiconductor body is thus increased so that currents can also be induced in the interior of the rod by means of the coil 10, such currents occurring at the location within the magnetic field of this coil, with the result that the temperature is further increased. At the same time the temperature of the ring heater 17 declines due to the weakening of the magnetic field caused by the currents increasingly generated within the semiconductor.

Such a device also facilitates the production of a monocrystal. A monocrystalline seed 14 of silicon for example, is affixed to the end of rod 2 and the zone melting is commenced at this end location.

In order that the first pass in the zone melting take place in the upward direction the glowing zone produced by the ring shaped radiator is made to travel through the rod nearly to the other or lower end, by displacing the induction heating coil 10 downwardly and, or while, supplying a reduced heating power. When the lower end is reached, the high frequency power supplied to the heater is increased in order to liquefy the zone. Instead, the radiating heater 17 may also be mounted at the lower end on the holder 18, or each of the two ends may be provided with a separate radiating heater as stated above.

A preferred mode of operation is as follows. The silicon rod 2 is seeded at 14, with a small silicon single, or monocrystal. The molten zone is caused to move upwardly through and from the seeded location while the lower seeded end is rotated at about 20 to 120 revolutions per minute.

The rotation has a number of functions. It is necessary in order to obtain coaxial, neatly shaped monocrystal rods. It assists in determining, by visual observation, whether the rod is melted completely across. The rotation also increases the distance between the two solid liquid interfaces in the molten zone. The latter effect is advantageous because it decreases the danger of solidification and spoiling of the crystal. The advantageous effect is probably caused by centrifugal action in the small zone or globule 11 of molten silicon. As described in my parent application, this globule is entirely, or in major part sustained by surface tension effects at the two solid-liquid interfaces.

At the end of a melting-zone pass the rotation is terminated and the electric power supplied to the induction coil is reduced to such an extent that the melt solidifies, but the temperature remains sufficiently high for this zone of the rod to maintain its good electric conductivity. Preferably this zone of the rod remains glowing. The induction coil, and consequently also the glowing or good-conducting zone, is again moved toward the other rod end. After a predetermined location is reached, preferably in the vicinity of the other rod end, the supply of electric heating power is again increased, so that the glowing or good-conducting zone is melted, and another travel pass of the melting zone in the same direction as during the preceding pass is initiated. It is advantageous that the glowing or good-conducting zone be pulled through the rod at greater speed than the melted zone of the subsequent melting-zone pass. It is also advantageous that the melting zone be pulled through a semiconductor rod in the direction from below to above, and the glowing or good-conducting zone be pulled through in the direction from top to bottom.

In further explanation, and recapitulating in part: For initiating the melting operation a location of the rod near one of its two ends, preferably near the upper end, is preheated by heat transfer from an auxiliary heater. Subsequently, electric power is supplied for inductive heating of the pre-heated location, but the power is limited to such an amount that this location remains in solid condition but is given the elevated temperature required for good electric conductance, preferably glowing temperature. Thereafter the induction coil, and therefor together therewith the glowing or good-conducting zone, is shifted toward the other rod end. After a given location is reached, preferably near the other end, the supplied electric power is increased and thereby the glowing or good-conducting zone liquefied. Thereafter the melting zone is pulled through the semiconductor rod by reversing the travel motion of the inductance coil.

It is understood that relative displacement of the rod 2 and the heating coil 10 is sufficient. Either one can be moved relative to the other.

Fig. 2 illustrates a modified form of auxiliary heater. The latter is simplified and also serves as means for fastening silicon or germanium rod 2 in the holder 190. The holder 190 is not slitted and has no screws, in contradistinction to holder 19 of Fig. 1, which is slit at 119 and is fastened to the rod by means of screws 219. The rod 2, of silicon for example, is clamped fast in holder 190 with the aid of three sheet metal strips 191 of molybdenum, each of which may be approximately 0.2 mm. thick, 40 mm. long, and 5 mm. wide. Before inserting these strips, they are slightly bent to a wave shaped configuration, so that they serve as three clamping springs. They are placed against the rod end in the longitudinal direction and about 120° displaced from each other, being pushed into the holder together with the end of the rod.

The holder is advantageously made of aluminum oxide. The lower ends of the molybdenum strips protrude downwardly out of the holder about 10 to 15 mm. When the heating coil 10 is moved upwardly to such an extent or position that the protruding ends of the molybdenum strips enter into the high frequency field the said ends start flowing and transfer the heat to the adjacent zone of the silicon rod touched by the molybdenum strips. The heat, in contrast to the radiant heater ring 17 shown in Fig. 1, is mainly transmitted to the silicon by conduction.

Fig. 3 illustrates the device of Fig. 2 inside an enclosure member including a steel dome 310, the outside of which is cooled by water coil 320. The width of the dome is considerably larger than the width of the quartz tube 4 shown in Fig. 1. The inner diameter of dome 310 may be at least ten times the diameter of coil 10. Thus when the process is operated at high vacuum a more extensive internal surface area is provided for deposition of impurities volatilized out of the molten zone of the silicon rod. Preferably, an observation window 310' of refractory glass for example, is mounted in the wall of the dome 310. Where the total internal surface area of the dome 310 is relatively large, the observation window is less likely to be obscured by internal deposits. Clamps 334 fasten the dome on steel base plate 315. The latter is provided with a stub pipe 250 arranged to be connected to a pumping device (not shown) to produce high vacuum within the enclosure member. The support 26, spindle 28, and induction coil 10 are inside dome 310. The lower end of silicon rod 2 is rotated by motor 76 to the rotor of which lower holder 18 may be coupled by gear means not shown. The upper end of shaft 21 and rod 2 may be rotated by turning gear 67 by, or independently of, motor 76. The upper end of rod 2 can be pulled upwardly by means of bracket 329 on slider 318 moving along rotary spindle 316, which is carried by support 346 and turned by motor 382 through gear 380.

The pertinent parts of the disclosure of my copending application Serial No. 409,610 are incorporated herein by reference. This especially refers to the use of p- and n-doping substances, seeding procedures, and procedures for moving the two holders apart concomitantly or independently of the independent rotation of each of the holders. It is desirable or necessary in the ordinary zone pulling, for purification and production of monocrystals, that the holders be displaceable relative to each other in the axial direction. Particularly at the beginning of a process, when the monocrystalline seed is to be united with the lower rod end, the provision for axial displacement either of the lower holder in which the crystal seed is located, or the upper holder carrying the polycrystal, is practically indispensable. In many cases a readjustment is also necessary during the entire pass of the zone, such adjustment being made from time to time in order to obtain a uniform cross section over the length of the rod. In certain cases there may occur sputtering of gas, with the result that the cross section is deformed and in some cases there may occur a squirting away of some of the melting liquid. In such cases, too, the uniformity of the cross section can be reestablished by subsequent regulation of the spacing between the holders. The regulation comprises reducing or widening the space between the holders, as required.

The monocrystalline bodies of semiconductor material made according to the described method are used for the manufacture of rectifiers, transistors and like semiconductor devices.

I claim:

1. A crucible-free zone melting process for treating a semiconductor material, comprising supporting a rod of said material vertically, melting a zone of said rod by high frequency electromagnetic induction, the molten zone forming an electric conductor in the high frequency field, carrying out a melting-zone pass operation by relatively displacing the high frequency field with

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respect to the rod in its longitudinal direction, to cause the molten zone to travel along the rod, the material of the traveling molten zone being heated by electric current inductively generated within the rod, the melting operation being initiated by preheating by heat transfer from an electro-conductive body at a location off-center with respect to the longitudinal extent of the melting-zone pass, the high frequency electric field being juxtaposed with respect to said body and the supplied electric power being limited to effect inductive heating of the body and of the rod sufficient to provide a zone of good electric conductivity of the rod but not sufficient to melt the body, and displacing said electric field and said zone, which is solid, but not displacing said electro-conductive body, with respect to and through the rod in the direction of the opposite end, thereafter increasing the electric power to melt a zone of the rod and displacing the high frequency field, and consequently the molten zone, along the rod in the opposite direction.

2. The process of claim 1, the said preheating heat transfer being at least partially by heat conduction from said body to the rod.

3. A crucible-free zone melting process for treating a semiconductor material, comprising supporting a rod of said material vertically, melting a zone of said rod by high frequency electric induction, the molten zone forming an electric conductor in the high frequency field, carrying out a melting-zone pass operation by relatively displacing the high frequency field with respect to the rod in its longitudinal direction, to cause the molten zone to travel along the rod, the material of the traveling molten zone being heated by electric current inductively generated within the rod, the melting operation being initiated by preheating by heat transfer from an electro-conductive body at a location off-center with respect to the longitudinal extent of the melting-zone pass, the high frequency electric field being juxtaposed with respect to said body and the supplied electric power being limited to effect inductive heating of the body and of the rod sufficient to provide a zone of good electric conductance of the rod but not sufficient to melt the body, and displacing said electric field and said zone, which is solid, but not displacing said electro-conductive body, with respect to and through the rod in the direction of the opposite end, thereafter increasing the electric power to melt a zone of the rod and displacing the high frequency field, and consequently the molten zone, along the rod in the opposite direction, a monocrystalline seed being fused into an end region of the rod opposite the location of preheating, the seeded end region of the rod being rotated about the longitudinal axis of the rod during the melting-zone pass operation, the opposite end not being rotated

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during said pass operation, the opposite ends of the rod being displaced apart with respect to each other to determine the cross-sectional diameter of the processed rod.

4. A crucible-free zone melting process for treating a semiconductor material, comprising supporting a rod of said material vertically, melting a lengthwise limited zone of said rod by high frequency electromagnetic induction, the molten zone forming an electric conductor in the high frequency field, which field is of limited lengthwise extent, carrying out a melting-zone pass operation by relatively displacing the high frequency field with respect to the rod in its longitudinal direction, to cause the molten zone to travel along the rod, the material of the travelling molten zone being heated in at least major part by electric current inductively generated within the rod, the melting operation being initiated by preheating by heat transfer from an auxiliary heating means, the preheating being at a location relatively small with respect to the longitudinal extent of the melting zone pass, a monocrystalline seed being fused into an end region of the rod spaced lengthwise of the location of preheating, the high frequency electric field being juxtaposed with respect to said small location and the supplied electric power being limited to effect inductive heating of the rod sufficient to provide a zone of good electric conductivity of the rod but not sufficient to melt the rod, and displacing said high frequency field and said zone, which is solid, with respect to and through the rod in a direction away from said location of preheating whilst the auxiliary heating means stops operating, thereafter increasing the electric power to melt a zone of the rod and displacing the high frequency field, and consequently the molten zone, along the rod in the opposite direction, the seeded end region of the rod being rotated about the longitudinal axis of the rod during the melting-zone pass operation, the opposite end not being rotated during said pass operation.

References Cited in the file of this patent

UNITED STATES PATENTS

2,318,468	Denneen	May 4, 1943
2,384,982	Walton	Sept. 18, 1945
2,739,088	Pfann	Mar. 20, 1956
2,743,199	Hull	Apr. 24, 1956
2,809,905	Davis	Oct. 15, 1957

OTHER REFERENCES

P.H. Keck; W. Van Horn, J. Soled, and A. MacDonald, the Review of Scientific Instruments, vol. 25, No. 4, pp. 331-334, April 1954.

P. H. Keck and M. J. E. Golay, Physical Rev. 89, 1297 (1953).