

Nov. 9, 1965

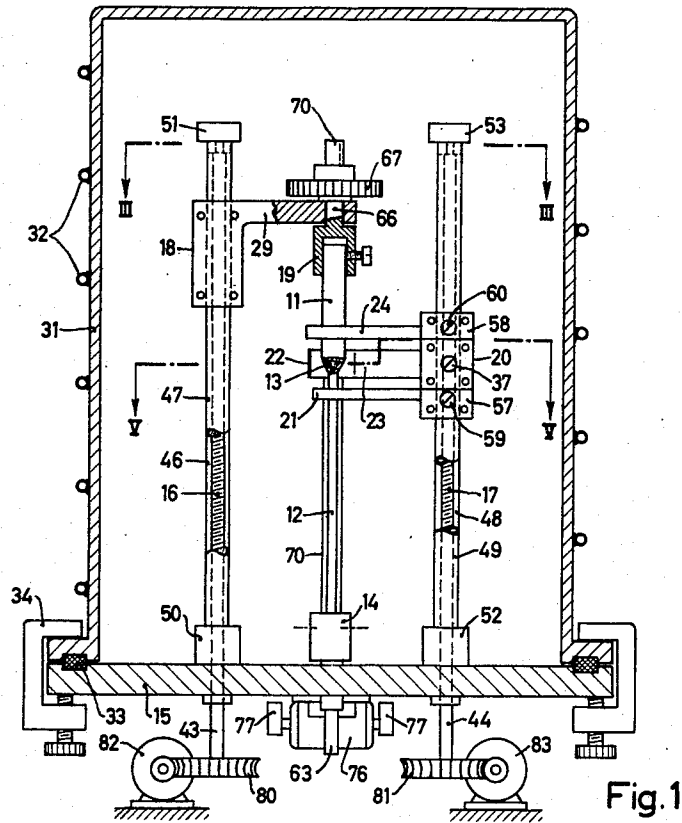
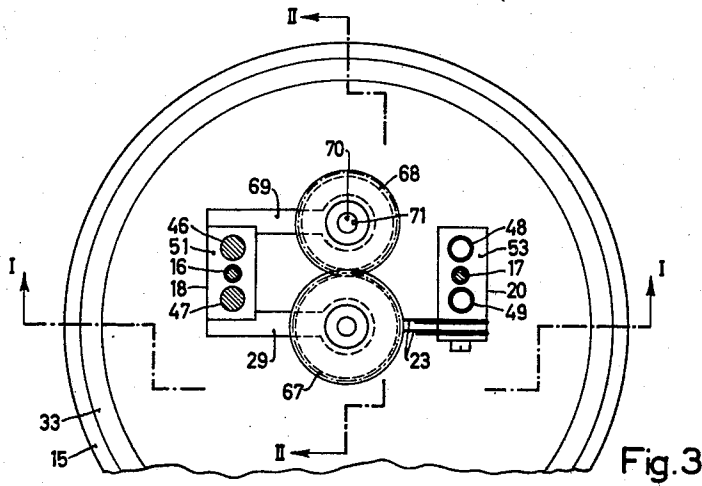
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3,216,805

DEVICE FOR CRUCIBLE-FREE ZONE MELTING

Filed Oct. 26, 1961

2 Sheets-Sheet 1



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DEVICE FOR CRUCIBLE-FREE ZONE MELTING

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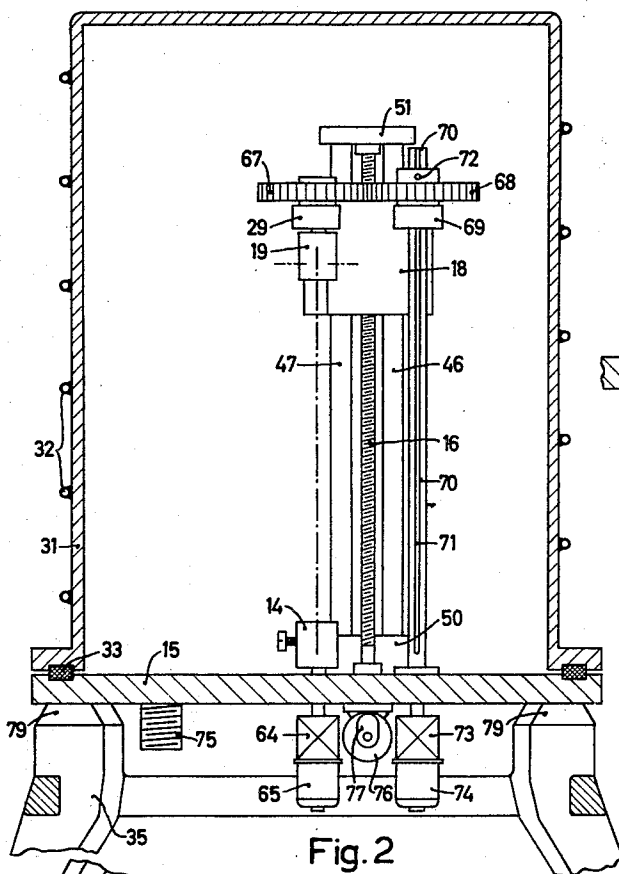


Fig. 2

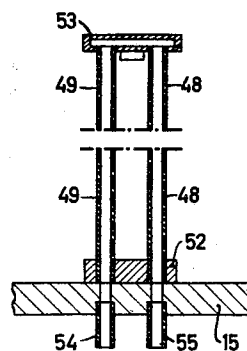


Fig. 6

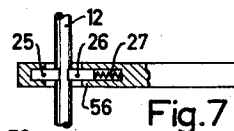


Fig. 7

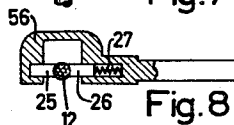


Fig. 8

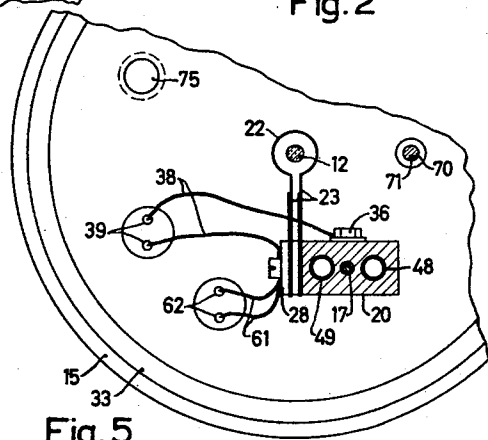


Fig. 5

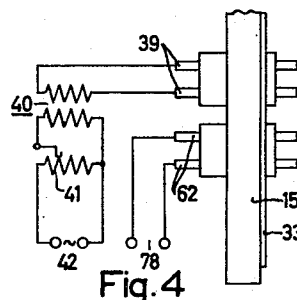


Fig. 4

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DEVICE FOR CRUCIBLE-FREE ZONE MELTING

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2 Claims. (Cl. 23—273)

This application is a continuation-in-part of application Serial No. 409, 610, filed February 11, 1954, now Patent No. 3,030,194 and relates to a device for the crucible-free zone melting of an elongated rod-shaped body of crystalline substance, preferably semiconductor substance. The crucible-free or "floating" zone melting is a known type of a zone-melting method and is preferably employed for substances having a very high melting point, such as silicon for example, which would cause difficulties when employing a melting crucible. However, the method is also suitable for a processing of germanium, aluminum antimonide and the like.

The crucible-free zone melting method consists in firmly clamping the two ends of the rod-shaped body and to liquefy a cross-sectional zone of slight axial thickness by heating without the use of a crucible or the like retaining means. The heating device required for this purpose, and the crystalline rod, are moved relative to each other in the longitudinal direction of the rod, so that the melting zone travels longitudinally through the rod.

In case the method is performed in a vacuum chamber, a considerably better result can be obtained by means of a device which, according to the invention, has a vacuum chamber whose width is very large relative to the diameter of the ring-shaped heating device, particularly if a multiple of that diameter. The vacuum chamber width should be at least 10 times the diameter of the rod to be processed. The advantages of such a spacious apparatus was recognized from observations made with a device for crucible-free zone melting employing a quartz tube closely surrounding the semiconductor rod and the heating device, the zone-melting method being performed with a silicon rod under high vacuum. It was found that a precipitation of silicon evaporated out of the melting zone was deposited on the inner wall of the quartz tube. After only a few zone passes this precipitation became so dense that further observation of the travelling melting zone became impossible. For reestablishing the possibility of observation, it would have been necessary to remove the precipitated coating. This, however, would have required to interrupt the processing operation and to open the vacuum chamber. After again closing and sealing the chamber, it would be necessary to first reestablish the vacuum before the processing could be continued.

After a greater number of zone passes, the precipitated coating reached such a thickness that portions thereof scaled off the wall of the quartz tube and dropped down, due to the continuous change of heating when the melting zone passed by and the immediately following cooling. When such scaling occurs at a moment in which the melting zone just passes through a rod location beneath the scaling-off point, the precipitation scales can then enter into the melting zone and contaminate the molten zone because the precipitation also comprises impurity substances, for example those that became evaporated together with the silicon out of the melting zone during the first few zone passes. The precipitation scales thus reaching the melting zone are again melted into the silicon material of the rod. As a result, new impurities enter into the zone as well as into the portion of the silicon rod through which the melting zone will subsequently

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travel. It follows that one of the prime purposes of the crucible-free zone melting, namely the production of extremely pure silicon, can be defeated in some cases when using a relatively narrow vacuum vessel.

The above-described disadvantages are eliminated by giving the vacuum chamber a comparatively large inner width, because then the thickness of the precipitating silicon, for an equal quantity of material vaporized out of a melting zone, is considerably smaller because of the larger area of the inner wall surface. Consequently, the precipitant need be removed only after a long period of operation and can be undertaken when the vacuum chamber is already opened, for example, when removing a completely processed silicon rod or when inserting a new rod. Furthermore, any portions of the precipitated coating cannot reach the melting zone, even if such portions scale off during the operation, because the radial spacing of the melting zone from the inner wall of the vacuum chamber is too large.

An example of a device according to the invention suitable for the pulling of monocrystals is illustrated on the drawing.

FIG. 1 shows a sectional front view of an embodiment of a complete processing device according to the invention, the section being taken along the line I—I indicated in FIG. 3;

FIG. 2 shows a side view thereof, also in section, the section being taken along the line II—II in FIG. 3;

FIG. 3 shows a cross section seen from the top along the line III—III in FIG. 1, the bell-shaped housing of the device being removed;

FIGS. 4 and 5 show details relating to the electrical connections and circuits of the device; and

FIGS. 6, 7 and 8 show separately a number of individual parts of the device.

Referring to FIGS. 1 to 8, the rod of semiconductive material to be treated is shown in an intermediate stage of the processing procedure in which this rod has a relatively thick upper portion 11 and a relatively thin lower portion 12 (FIG. 1). That is, the portion 12 has already passed through the zone-melting process so that its cross section has become reduced in comparison with the original cross section of the upper rod portion 11. The material in the melting zone 13 is liquid and forms a drop. A holder 14 for the lower rod portion is revolvably mounted on a base plate 15 and is vacuum-tightly sealed at the base plate. The plate 15 forms the bottom of a recipient within which the process is carried out in vacuum or in an atmosphere of inert gas. To this end, the base plate 15 is provided with a nipple 75 (FIG. 2) for connection to a high-vacuum pump or to a gas tank. The bell 31 of the recipient consists of steel. A cooling coil 32 of copper tubing forms several turns around the bell and is soldered thereto. The cooling coil 32 may be connected to a water supply pipe. The bell is sealed by means of a gasket ring 33 and is held pressed against the base plate 15 by screw clamps 34 (FIG. 1). The base plate 15 has rubber feet 79 resting upon a three-legged support 35 so that the base plate is readily accessible from below. It may be seen from the drawing that the internal diameter of the bell is at least ten times the diameter of heating ring 22.

Two screw spindles 16 and 17 pass revolvably and vacuum-tightly sealed through respective openings of the base plate 15. An internally threaded support 18 of brass, in threaded engagement with the spindle 16, carries a holder 19 for the upper end of rod portion 11. Holder 19 is joined with support 18 by means of an arm 29 consisting preferably of insulating material. Another support 20, also of brass, is mounted on the spindle 17 in threaded engagement therewith. Support 20 carries the heating device. The supports 18 and 20 slide along guid-

ing frames each consisting of two steel rods 46, 47 and 48, 49 respectively, of circular cross section. Two cross bars 50 and 51 interconnect the rods 46 and 47 at both ends respectively. Corresponding cross bars 52 and 53 interconnect the ends of the pair of rods 48 and 49. The rods 48 and 49 that guide the support 20 for the heating device are preferably designed as tubes according to FIG. 6. The upper openings of the tubular rods communicate with each other through a duct in the interior of the cross bar 53. The lower openings are connected through respective bores in base plate 15 with nipples 54 and 55 through which cooling water is circulated through the rods.

In the illustrated embodiment, a ring-shaped heating device, consisting of an annularly bent strip 22 of tungsten, is energized by electric current in the order of magnitude of 100 amps. It will be noted that the heating is by heat radiation, since, as stated above, contact of the molten material with apparatus elements is to be avoided. The temperature of the heater is controlled depending upon the melting point of the semiconductor material. This temperature, for instance, is about 2000° C. for silicon, corresponding to a melting point of approximately 1400° C. The melting points for germanium and the semiconductive compounds are lower, in part below 1000° C. so that the temperature of the ring-shaped heater may be correspondingly lower. The liquid zone of the semiconductor rod, therefore, has the melting temperature of the particular substance being processed. The time of the heating treatment results from the velocity at which the heating device is moved along the rod as specified in this disclosure.

Current is supplied to the heater ring 22 by means of terminal lugs 23 integral with the heater ring. The lugs 23 are clamped to the support 20 by means of a copper plate 28 with an intermediate layer of mica. The plate 28 is fastened to support 20 by means of insulated screws. The current-conducting cross section of the terminal lugs 23 can be doubled by back-folding the clamped ends as is apparent from FIG. 5. The support 20 and the plate 28 carry respective terminals 36, 37 for the connection of flexible current supply cables 38 leading to conductor pins 39 (FIG. 5) that extend vacuum-tightly through the base plate 15. The current supply cables are only schematically represented in the drawing (FIG. 5). They consist preferably of uncovered Litz wire with short tubular pieces or beads of glass strung upon them to provide the necessary insulation as well as a sufficient flexibility. The lead-in pins 39 are connected with the high-current output winding of an auxiliary transformer 40 (FIG. 4) having a primary winding energized through a variable autotransformer 41 from the terminals or buses 42 of an alternating current line so that the output voltage can be adjusted between zero and, for instance, 10 volts. The temperature of the heater is thus controlled by the variable autotransformer.

The respective shaft ends 43 and 44 of the threaded spindles 16 and 17 are driven through suitable transmissions, for instance the illustrated worm gears 80 and 81, from small direct-current shunt motors 82, 83. These motors may be mounted on the support 35 in any suitable manner. The ratio of the revolving speeds of motors 82 and 83 is so adjusted that the support 20 moves upwardly at a greater velocity than the support 18. For instance, the heating device 22 and its support 20 may move upwardly at a speed in the order of 0.5 to 5 mm. per minute. When the upper holder 19 and its support 18 are moved upwardly at half the speed of support 20, then the cross section of the lower rod portion 12 becomes about half as large as that of the upper rod portion 11. By a corresponding selection of the speeds, the rod-shaped body being zone-melted can be drawn to a cross section of any desired thinness. The ratio of the revolving speeds may also be adjusted by a mechanical transmission or by an automatic electric control and, if desired, this speed

ratio may be varied at will or may be automatically regulated during the zone-melting operation.

Also mounted on support 20, by means of a clamping plate 57, is a guiding device 21 for the free end of the lower rod portion 12. According to FIGS. 7 and 8, the clamping device 21 comprises a holder 56 that forms a semi-annulus about the rod portion 12 and carries two guide fingers 25 and 26 of which one is resiliently displaceable and biased by a pressure spring 27. The guide fingers 25 and 26 may consist of carbon or aluminum oxide. They are preferably cylindrically recessed where they come into slidable engagement with the rod portion 12. The guide fingers maintain the free end of rod portion 12 axially aligned with the heating device and with the upper rod portion 11. However, if the cross section of the treated body is larger than 5 square-millimeters, it is, as a rule, unnecessary to provide additional means for guiding the rod during the processing operations.

Another guiding device 24 is provided for the upper rod portion 11. The guiding device 24 is similar to device 21 and is mounted on support 20 by means of another clamping plate 58. The guiding devices 21 and 24 may also serve as means for supplying an electrical current in the order of a few amperes to be additionally passed through the melting zone 13. To this end, the holders 56, consisting of a conductive material such as brass, are in conductive contact with the clamping plates 57 and 58 also consisting of conductive material, but are insulated from the support 20 and the fastening screws by an intermediate insulating layer of heat-resistant material such as mica. The clamping plates 57 and 58 are provided with terminal screws 59, 60 that are connected by leads 61 to vacuum-tightly sealed lead-in terminals 62 traversing the base plate 15 (FIG. 5). The lead-in terminals 62 are connected to a direct-current or alternating-current source.

The device is further equipped with means for revolving the rod portions 11 and 12. To impart such a revolution to the rod ends, the shaft end 63 of the holder 14, vacuum-tightly journaled to the base plate 15, is coupled by a gear transmission 64 with a drive motor 65. The upper holder 19 is also provided with a shaft end 66 (FIG. 1) which is journaled in the bore of the supporting arm and carries a spur gear 67. Another spur gear 68 is journaled on a supporting arm 69 (FIGS. 2, 3) mounted on support 18. Gears 67 and 68 are continuously in meshing engagement with each other. A shaft 70 with a longitudinal groove 71 (FIGS. 1, 2, 3) passes through a central bore in the hub portion of spur gear 69. A pin 72 screwed into the hub portion of spur gear 68 engages the groove 71, so that spur gear 68 is entrained by the revolving shaft 70 while being capable to slide upwardly and downwardly along the shaft. Shaft 70 has its lower end vacuum-tightly journaled in the base plate 15. The shaft end, extending through the base plate, is connected by a gear transmission 73 with a drive motor 74. The apparatus for rotating the rod ends just described makes it possible to drive the upper rod portion 11 alone or the lower rod portion alone or both rod portions simultaneously, either in the same direction or in opposite directions of revolution, and at any desired speed between 0 and 1000 or more revolutions per minute. This permits modifying the shape and consistency of the melted zone in various ways. It is also possible, for instance, to cause impurities, that may be included in undissolved condition within the melted zone, to migrate due to centrifugal force to the rod surface, from which subsequently such impurities may readily be removed. That is, any undissolved foreign substances in the interior of the melted zone, for instance particles of silicon carbide, having a higher specific gravity than the melted material, when subjected to a sufficiently high speed of revolution, are moved by centrifugal force to the exterior surface of the body. By subsequent etching of the processed

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body, such inclusions can be laid open and can easily be removed mechanically, for instance, by scraping.

In certain cases, the texture of the zone-melted body to be produced can be improved by imparting vibration to the liquid zone during the processing period. Such vibrations are produced by a shaker motor 76 whose shaft ends carry eccentric unbalance masses 77. The shaker motor 76 is mounted on the base plate 15.

It will be obvious to those skilled in the art, upon study of this disclosure, that my invention permits of various modifications other than those specifically illustrated and described, without departing from the essence of my invention and within the scope of the claims annexed hereto.

I claim:

1. Apparatus for crucible-free zone melting of rod-shaped crystalline material, comprising a gas-tight vacuum enclosure member having means for connection with a vacuum pump, axially spaced holders in said vessel for securing the rod material between them, annular electric heating means in said vessel axially surrounding the rod-shaped material, means for displacing said heating means along said axis for zone melting the rod-shaped material, means for supplying current to said electric heating means, the improvement which comprises the enclosure member providing an enclosure chamber having an inner width which is a multiple of the diameter of the annular heating means and the distance between the inner wall of the enclosure member and the annular heating means is a multiple of the inner diameter of the annular heating means.

2. Apparatus for crucible-free zone melting of rod-

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shaped crystalline material, comprising a gas-tight vacuum enclosure member having means for connection with a vacuum pump, axially spaced holders in said vessel for securing the rod material between them, annular electric heating means in said vessel axially surrounding the rod-shaped material, means for displacing said heating means along said axis for zone melting the rod shaped material, means for supplying current to said electric heating means, the improvement which comprises the enclosure member providing an enclosure chamber being of steel and having a minimum inner width which is a multiple of the diameter of the annular heating means and the distance between the inner wall of the steel chamber and the annular heating means is a multiple of the inner diameter of the annular heating means, and cooling means for cooling the walls of the enclosure member.

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