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METHOD OF CRUCIBLE-FREE ZONE MELTING

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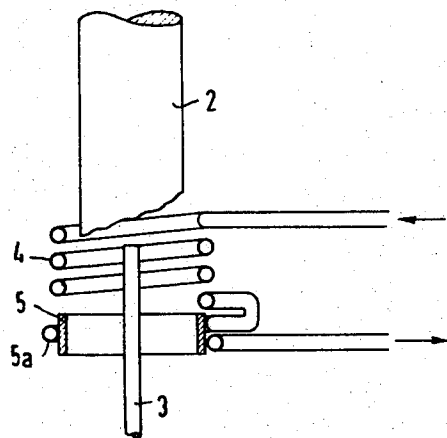


Fig. 1

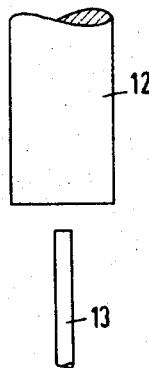


Fig. 2

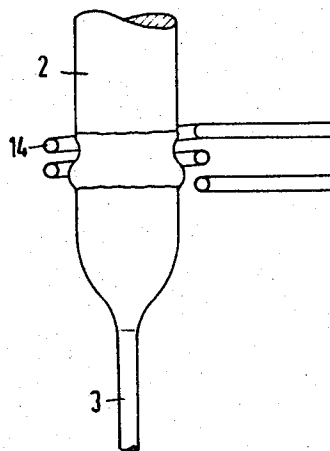


Fig. 3

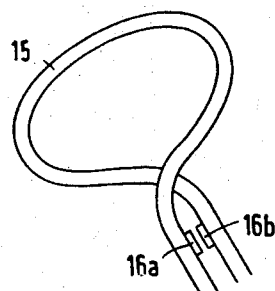


Fig. 4

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METHOD OF CRUCIBLE-FREE ZONE MELTING

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6 Claims. (Cl. 23—301)

My invention relates to a method of crucible-free zone melting of a rod-shaped, crystalline member held by its ends in a vertical position.

Various methods of crucible-free zone melting are known. As a rule, a crystalline rod is held at its ends in a vertical position within an evacuated vessel or one filled with a protective gas. A heating device surrounding the rod serves for melting a short length thereof, the so-called melting zone. By relative motion between the rod to be treated and the heating device, the melting zone is passed over the entire length of the rod. An induction heating coil has been found to be a particularly suitable heating device since, by means of such coil, the melting zone can be supplied with the necessary power for melting the rod without danger of contamination due to contact or the like.

It is already known, for example, from Patent No. 3,108,169, to place a closed ring of a material having good electrical conductivity, below the induction heating coil. The heating coil induces a current in this ring which produces an electromagnetic field opposing the field of the heating coil. The heating coil field is thereby balanced or compensated on the side where the short-circuiting ring is located. This makes it possible to limit the effect of the heating coil field, especially locally. The effect of the heating coil field is shortened, and it is accordingly possible to shorten the melting-zone length. This is particularly important when processing rod-shaped members with relatively large diameters, since the larger melting zones are more inclined to tear or overflow, and hence to cause an interruption in the process. When the melting zone, which fills out the entire width of the rod, is shortened in length and displaced upwardly relative to the heating coil, there is a greater likelihood of maintaining cohesion of the melting zone through cooperation between the surface tension forces of the melt and the supporting forces of the heating coil.

It is further known from German Patent No. 1,094,710 or Canadian Patent No. 642,713, for example, to attach to one end of a semiconductor rod, in a crucible-free zone melting process, a seed crystal whose cross section is much smaller than that of the treated rod. In this way, the quality of the resulting monocrystal is improved, especially with respect to the density of the dislocations. When treating semiconductor rods with relatively large cross sections, for example a cross-sectional diameter longer than 30 mm., difficulties occur especially when a narrow seed crystal is to be fused thereto. Experience has shown that a narrow seed crystal cannot be fused to a rod of such large diameter if no short-circuiting ring is placed under the coil, because the melt runs out or drips off in the transition or junction region between the seed crystal and the rod. This situation is remedied by the presence of the short-circuiting ring below the heating coil which permits the narrow seed crystal to be fused to the rod without causing the melt to leak. However, new problems are caused by the use of the short-circuiting ring. Due to the field distortion resulting from the use of a short-circuiting ring, the monocrystal formation is rendered more difficult of attainment for rods with larger diameters. While, normally, after a few melting-zone passes, such as three or four, for example, starting from the seed crystal, the entire rod of crystalline semiconductor material could be

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transformed to a monocrystal, when short-circuiting rings are utilized, disturbances occur occasionally which result in an undesirable prolongation of the process.

It is an object of my invention to provide a method of crucible-free zone melting which avoids these difficulties of the heretofore known methods.

With this and other objects in view, I provide a method of crucible-free zone melting of a rod-shaped member vertically held at its ends, the member being of crystalline semiconductor material, particularly silicon, and having a rod diameter exceeding 30 mm. An induction heating coil, which surrounds the rod and is energized with high frequency alternating current, is movable along the rod axis. A short-circuiting ring of good electric conductance material which compensates a portion of the heating coil field, is located below the coil, and, attached to the lower end of the rod-shaped body, is a crystal seed whose cross section is considerably smaller than the cross section of the rod-shaped body. In accordance with one feature of my invention, the melting zone produced with the aid of the induction heating coil is passed upwardly several times through the body, starting from the seed crystal. Thereby, at least the first pass of the melting zone is carried out with an effective partial compensation of the heating coil field, and thereafter at least one additional pass of the melting zone is performed without any partial compensation of the heating coil field.

Thus, for example, a semiconductor rod to be processed is inserted into a zone melting device wherein a short-circuiting ring is located below the heating coil. At the same time, a narrow seed crystal is inserted from below so that it extends up to the semiconductor rod. Thus, the seed crystal is stood up in the lower holder, while the semiconductor rod is hung from the upper holder. If the semiconductor rod is then melted at its lower end, for example by energizing the heating coil after first preheating the rod until it is capable of absorbing a current, a partial compensation of the heating coil field occurs due to the presence of the short-circuiting ring. The result is that the heating coil is effective only at its upper end. The semiconductor rod can be so placed that its lower end is somewhat above the heating coil or its lower end actually extends into the upper region of the heating coil. In both instances, a strong supporting effect of the heating coil is produced, since the heating coil field is so distorted by the partial compensation that it has an upward supporting effect. This prevents the drop, which has melted at the lower end of the semiconductor rod, from flowing downward, and by moving the seed crystal toward it, the seed crystal is dipped into the molten drop. Thereafter, the melting zone is moved upwardly due to relative motion between the heating coil and the semiconductor rod, so that the semiconductor material again becomes solidified starting at the seed crystal.

After the melting zone is passed along the semiconductor rod once or twice, the junction between the seed crystal and the treated rod has an outwardly curved shape, so that during further melting, no tearing or dripping off of the melting zone need be feared. The remaining portion of the semiconductor rod is made uniform in cross section and thickness, and especially when the semiconductor rod is rotated during zone melting, it is of circular, well centered shape.

The short-circuiting ring can then either be removed or the semiconductor rod may be withdrawn from the zone melting device and transferred to another zone melting device without a short-circuiting ring. As aforementioned, the semiconductor rod frequently has dislocations in the monocrystalline growth so that subsequent treatment, without the short-circuiting ring, is necessary. Thus, after dismantling the short-circuiting ring or after transfer of the semiconductor rod, the melting zone may be passed

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one or more times through the semiconductor rod. It has been found that a smaller number of passes of the melting zone, for example two or three, are required to transform the semiconductor rod into a monocrystal after removal of the short-circuiting ring.

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 method of crucible-free zone melting, it is nevertheless not intended to be limited to the details shown since various modifications may be made therein without departing from the spirit of the invention and within the scope and range of equivalents of the claims.

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

FIG. 1 is a fragmentary sectional view of a zone melting device for carrying out the method of this invention, showing a semiconductor rod with a broken end and a seed crystal in alignment prior to being joined together;

FIG. 2 is a fragmentary view of a semiconductor rod with a smooth end and a seed crystal in alignment prior to being joined together;

FIG. 3 shows the junction or transition region of the narrow seed crystal to the thick semiconductor rod, after the initial zone passes have been carried out; and

FIG. 4 shows a modified short-circuiting ring employed in carrying out my inventive method which is opened by opening a pair of switch contacts.

Referring now to the drawings and first particularly to FIG. 1, there is shown a semiconductor rod 2, which may have a diameter of 30 to 40 mm. for example, and more specifically 32 mm. in the example described herein. The upper end of the rod 2 is fixed in the upper holder (not shown) of a zone melting device, while its lower end hangs freely. As seen in FIG. 1 of the drawing, the lower end of the rod has an uneven broken surface. The semiconductor rod 2 is produced according to the described method of U.S. Patent 3,011,877, for example, through the deposition of semiconductor material from the gaseous phase. After adherent bridges or current-connecting components have been removed, the rod has the uneven fractured surface at the end thereof as shown in FIG. 1. A seed crystal 3 must then be attached to the lower fractured surface of the rod 2.

This crystal seed 3 has a much smaller diameter than the rod 2, for example a diameter of 5 to 6 mm. To produce the transition region between the semiconductor rod 2 and the narrow seed crystal, one might employ a mechanical operation, for instance, appropriately grinding down the lower end of the semiconductor rod 2, into the shape of a cone tapering downwardly, for example. Practice has shown that a very great contamination of the highly purified semiconductor material produced by precipitation from the gaseous phase results from such a mechanical operation, and it is, therefore, not recommended. One might also try to remove the impurities remaining at the surface following grinding, by means of etching. It has become apparent, however, that such impurities cannot be completely removed by etching solutions, and that, moreover, the etchant causes other impurities to form on the semiconductor material.

When in accordance with the invention, the first zone passes are performed with the aid of a heating coil, whose field effect is partly compensated by the opposing field induced in a short-circuiting ring, then an uneven, broken end surface of the rod is permissible. Since the lower region of the heating coil field is compensated by the counter-field of the short-circuiting ring 5, the heating effect of the heating coil field occurs only upwardly thereof and a predetermined supporting action takes place. The supporting action may be increased by supplying the heating coil with a current frequency of approximately 1 to 1.8 mHz., for example 1.5 mHz. instead of a frequency of

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about 4 mHz. which is normally employed for zone melting the semiconductor rod. FIG. 1 shows a cooling tube 5a placed around the short-circuiting ring 5, and connected with the cooling loop of heating coil 4 which consists of electrically conductive tubes, such as of silver-plated copper, traversed by water in the direction of the arrows. The cooling tube 5a may be soldered onto the ring 5 which can be made of silver or silver-plated copper.

FIG. 2 shows a semiconductor rod 12 which does not have an irregular end but which has a relatively smooth end surface, produced, for example, by cutting off the irregular end of the rod shown in FIG. 1, with a diamond saw. A narrow seed crystal 13 may also be attached according to my inventive method, to the smooth end of the rod 12 shown in FIG. 2.

FIG. 3 shows the result on the rod and seed crystal of one or more passes of the melting zone. The junction or transition region of the semiconductor rod 2 and the crystal seed 3 fused thereto has an approximately conical shape which reliably prevents dripping of the melting zone, even when the melting zone passes through the junction or transition region. Consequently, a heating coil 14 can then be used, whose field has no partial compensation. The heating coil 14 can be the same coil as heating coil 4 with the short-circuiting ring 5 removed or it can be a heating coil of another zone melting device. In the latter case, the semiconductor rod with the attached seed crystal must be transferred.

FIG. 4 shows a perspective view of a short circuiting ring 15 having another embodiment from that of FIG. 1. The short-circuiting ring 15 is substantially of the same construction as the customarily used induction heating coils, i.e. it is in the form of a silver tube or a copper tube, silver-plated on the outside thereof. The tube is traversed, while the zone melting device is in operation, by a liquid coolant, such as cooling water, for example. The ring 15 is not completely closed. The ring can be fastened to the heating coil by its end leads which are provided with two contacts 16a and 16b, which effect the short-circuiting of the ring 15 when in contact. If a ring, thus short-circuited, is used in a zone melting device for carrying out the method of this invention, the partial compensation of the heating coil field can be eliminated without removing the ring 15 from the apparatus by opening the contacts, since no current can consequently flow in the thus open ring 15, so that there is then no compensation of the heating coil field in this region.

The opening of contacts 16a and 16b, which can also be made of silver, for example, can be directly effected mechanically or by electromagnets, with the aid of a lever system or a relay.

The closed ring 15 can be opened simply by direct mechanical means, whereby in most cases, a corresponding interruption of the zone melting process takes place. However, remote operation may be provided for example by using an electromagnet which opens the contact pieces 16a and 16b without requiring the vacuum or protective gas-vessel (not shown) in which the zone melting process is taking place, to be opened. Preferably, the connecting and disconnecting of the contact pieces can be effected only by a change in the water pressure within the ring 15. Thus, if the water pressure is increased, for example, centrifugal force will cause the ring 15 to expand and the contacts 16a and 16b will engage. It is important that the contacts be arranged in such a way that, as viewed from the melting zone, they lie behind the parts of the ring 15. This prevents them from being subjected to vaporizing semiconductor material, which would disturb the contact making.

The protective vessel, the two opposite rod and seed crystal holders, means for relative displacement of the holders with respect to each other, means for relative displacement of the heater coil with or without the compensating ring with respect to the rod, and means for

rotating one or both holders, if desired, have not been shown in the drawings. Should such illustration be necessary, I reserve the right to import into the specification and drawings such means as is described in U.S. Patent No. 2,904,663, to Emeis et al., and in U.S. Patent No. 3,030,189, to Schweikert et al., for example.

I claim:

1. In a method of zone melting a polycrystalline rod having a diameter greater than 30 mm. so as to form a monocrystal thereof wherein the rod and a seed crystal of relatively small diameter are supported at the upper and lower end thereof respectively in vertical alignment with each other, the rod overlying the seed crystal and a molten zone is formed in the rod at a junction of the rod, and the seed crystal by a surrounding inductive heater coil energized by current from a high-frequency generator having a flux field, the molten zone being prevented from dripping by a short circuiting ring of electrically conducting material surrounding the rod and located beneath the heater coil at a distance therefrom adapted to induce in the ring a counter current with a flux opposing the flux of the heater coil in the region encompassed by the ring for compensating at least part of the heater coil flux so as to thereby support the molten zone, the improvement which comprises passing the molten zone at least once upwardly along the rod from the junction by relatively moving the rod and the heater coil and thereby simultaneously partly compensating the heater coil flux with the ring, to support the upwardly passing molten zone, and thereafter passing the molten zone upwardly along the rod from the junction at least once with the inductive high frequency heating coil and in the absence of the short circuiting ring thereby without compensating the heater coil flux.

2. In a method of zone melting a polycrystalline rod having a diameter greater than 30 mm. so as to form a monocrystal thereof wherein the rod and a seed crystal of relatively small diameter are supported at the upper and lower end thereof respectively in vertical alignment with each other the rod overlying the seed crystal and a molten zone is formed in the rod at a junction of the rod, and the seed crystal by a surrounding inductive heater coil energized by current from a high-frequency generator having a flux field, the molten zone being prevented from dripping by a short-circuiting ring of electrically conducting material surrounding the rod and located beneath the heater coil at a distance therefrom adapted to induce in the ring a counter current with a flux opposing the flux of the heater coil in the region encompassed by the ring for compensating at least part of the heater coil flux so as to thereby support the molten zone, the improvement which comprises relatively moving the rod on the one hand and the heater coil and the ring on the other hand in the direction of the rod axis so as to pass the molten zone at least once upwardly along the rod from the junction and thereby simultaneously partly compensating the heater coil flux with the ring to support the upwardly passing molten zone, then removing the ring and thereafter relatively moving the rod and the heater coil in the direction of the rod axis so as to pass the molten zone at least once upwardly along the rod from the junction with the inductive high frequency heating coil and in the absence of the short circuiting ring, thereby without compensating the heater coil flux.

3. In a method of zone melting a polycrystalline rod having a diameter greater than 30 mm. so as to form a monocrystal thereof wherein the rod and a seed crystal of relatively small diameter are supported at the upper and lower end thereof respectively in vertical alignment with each other the rod overlying the seed crystal and a molten zone is formed in the rod at a junction of the rod, and the seed crystal by a surrounding inductive heater coil energized by current from a high-frequency

generator having a flux field, the molten zone being prevented from dripping by a short circuiting ring of electrically conducting material surrounding the rod and located beneath the heater coil at a distance therefrom adapted to induce in the ring a counter current with a flux opposing the flux of the heater coil in the region encompassed by the ring for compensating at least part of the heater coil flux so as to thereby support the molten zone, the improvement which comprises relatively moving the rod on the one hand and the heater coil and the ring on the other hand in the direction of the rod axis so as to pass the molten zone at least once upwardly along the rod from the junction and thereby simultaneously partly compensating the heater coil flux with the ring to support the upwardly passing molten zone, then opening the ring so as to prevent induction of the counter current therein and thereafter relatively moving the rod on the one hand and the heater coil and the open ring in the direction of the rod axis so as to pass the molten zone at least once upwardly along the rod from the junction with the inductive high frequency heating coil and in the absence of the short circuiting ring, thereby without compensating the heater coil flux.

4. Method according to claim 3 wherein the ring comprises a flexible tube in the form of a loop having engageable contacts respectively on the end portions thereof, the tubular ring being traversible by fluid coolant, and being opened so as to disengage the contacts by varying the pressure of the fluid coolant in the tube.

5. In a method of zone melting a polycrystalline rod having a diameter greater than 30 mm. so as to form a monocrystal thereof wherein the rod and a seed crystal of relatively small diameter are supported at the upper and lower end thereof respectively in vertical alignment with each other the rod overlying the seed crystal in a first zone melting apparatus and a molten zone is formed in the rod at a junction of the rod, and the seed crystal by a surrounding inductive heater coil energized by current from a high-frequency generator having a flux field, the molten zone being prevented from dripping by a short circuiting ring of electrically conducting material surrounding the rod and located beneath the heater coil at a distance therefrom adapted to induce in the ring a counter current with a flux opposing the flux of the heater coil in the region encompassed by the ring for compensating at least part of the heater coil flux so as to thereby support the molten zone, the improvement which comprises relatively moving the rod on the one hand and the heater coil and the ring on the other hand in the direction of the rod axis so as to pass the molten zone at least once upwardly along the rod from the junction and thereby simultaneously partly compensating the heater coil flux with the ring to support the upwardly passing molten zone, then removing the joined rod and seed crystal and supporting the same vertically at the ends thereof in a second zone melting apparatus, forming a molten zone in the rod at the junction of the rod and the seed crystal by a surrounding inductive heater coil of the second apparatus energized by current from a high-frequency generator, and relatively moving the rod and the heater coil in the direction of the rod axis so as to pass the molten zone at least once upwardly along the rod from the junction with the short circuiting ring removed without compensating the heater coil flux.

6. Method according to claim 1 wherein the heater coil is energized with an alternating current having a frequency of substantially 1 to 1.8 mc.

No references cited.

NORMAN YUDKOFF, *Primary Examiner*.

G. HINES, *Assistant Examiner*.