

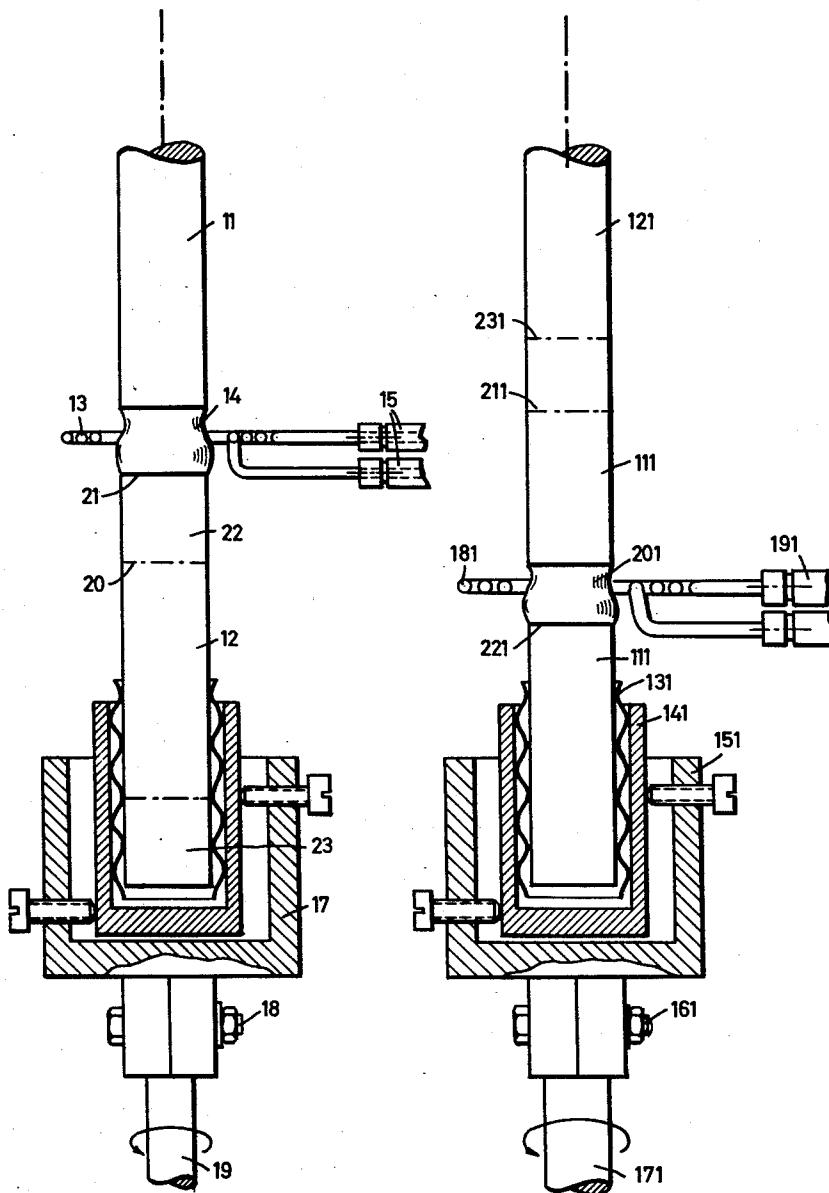
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L. SPORRER

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DISLOCATION REMOVAL BY A LAST PASS STARTING AT A
LOCATION DISPLACED FROM THE ORIGINAL
SEED INTO THE GROWN CRYSTAL

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DISLOCATION REMOVAL BY A LAST PASS STARTING AT A LOCATION DISPLACED FROM THE ORIGINAL SEED INTO THE GROWN CRYSTAL
Ludwig Sporrer, Erlangen, Germany, assignor to Siemens-Schuckertwerke Aktiengesellschaft, Berlin-Siemensstadt, Germany, a corporation of Germany
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My invention relates to the production of monocrystalline semiconductor material, such as silicon and germanium, for example, for electronic purposes by floating zone melting. Such a method, as a rule, is carried out by fusing a monocrystalline seed to one end of a polycrystalline rod of the same semiconductor material. The rod is then subjected to zone melting by heating a narrow cross-sectional zone of the rod and moving the molten zone longitudinally along the rod. When the zone melting operation is started at the junction point of the seed crystal and the molten zone is passed through the rod, the semiconductor material recrystallizes out of the molten zone in monocrystalline condition. By repeating the zone passes, a hyper-pure monocrystalline rod can be obtained. The seed crystal can thereafter be severed from the rod and can be used for similar processing of other polycrystalline rods.

In the known method, the seed crystal can be used for processing only a limited number of semiconductor rods because further use of the same seed crystal would greatly impair the electric properties of the additionally processed rods. This is because when the rod is heated up at the rod end adjacent to the crystal seed prior to each individual zone pass, the temperature of the then stationary heated and molten zone may cause crystal-lattice dislocations which, with an increasing number of zone passes, migrate from the end of the seed into a portion of the rod being processed. Such lattice dislocations can be made microscopically visible by etching of the crystal surface, and then manifest themselves by etch pits. It has been found that semiconductor rods thus converted from polycrystalline to monocrystalline constitution exhibit a greatly decreased lifetime of the minority charge carriers in the rod portion that was adjacent to the seed during processing. This is a serious defect because the lifetime of the charge carriers in semiconductor rods for the production of electronic semiconductor elements or wafers must be as high as possible and should be uniform over the entire length of the rod.

The mentioned detrimental effect can be minimized by replacing the crystal seed by a new seed prior to performing the ultimate pass of zone melting operation. This, however, is extremely intricate, particularly when performing the zone melting process in vacuum.

It is an object of my invention to avoid the above-mentioned deficiencies in a considerable simpler, more economical, and less time consuming manner.

Relating to a zone melting operation for the production of a monocrystalline hyper-pure semiconductor rod for electronic purposes from a polycrystalline rod by crucible-free zone melting with the aid of a crystal seed, where in the molten zone is repeatedly passed in a given direction lengthwise through the semiconductor rod, each time commencing from the fusion junction of the seed, it is a feature of my invention that the commencement of the ultimate pass of zone melting is shifted from the fusion junction of the seed away to a location of the rod that possesses a lesser degree of crystal lattice dislocations than obtaining near the junction.

For further explaining the invention, reference will be had to the accompanying drawing comprising two figures,

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each showing a portion of a zone melting device, in front view and partly in section.

Although the methods illustrated in FIGS. 1 and 2 have certain features in common, they will be individually described below.

Referring to FIG. 1 a semiconductor rod 11 of polycrystalline semiconductor material, such as silicon, is coaxially fused together with a longitudinally aligned monocrystalline seed 12, the (111)-crystal axis of which coincides with the travelling direction of the molten zone 14, which generally has a mass supportable by adhesion to the adjacent solid ends. The narrow floating zone 14 of the rod 11 is melted in a high-frequency alternating field produced by a water-cooled high-frequency coil 13. The turns of coil 13 consist of silver tubing and are traversed by cooling water supplied through bores of the current supply terminals 15 to which the coil 13 is mechanically and electrically attached. The coil 13 is displaceable in the longitudinal direction of the rod 11 to permit the passing of the molten zone 14 lengthwise through the rod. The crystal seed 12 is fastened in a holding and centering device 17 firmly joined by means of a clamping screw 18 with a drive shaft 19 which permits maintaining the seed 12 and the lower portion of the rod 11 in rotation during progress of the zone melting operation. The upper end (not illustrated) of the rod 11 is likewise fastened to a centering holder and may be likewise rotated as indicated by the arrow.

As a rule, a given number of passes, for example ten, are required for completely purifying the polycrystalline rod and converting it into a monocrystal. Each individual pass is commenced at the end of the crystal seed 12, this location being identified by a dot-and-dash line 20. Nine zone passes, all starting at line 20 and progressing to the upper rod end, are then completed. However, the tenth and last pass of the zone melting operation is commenced at a somewhat higher location 21 of the rod, which location may be spaced from line 20 a distance approximately equal to the diameter of the rod 11, and is located above the end of the crystal seed. At location 21, the completely processed rod is sawed off the seed end, and the rod next to be processed is fused to the crystal seed. Consequently, after processing of the first rod, the seed crystal is extended by a piece denoted by 22. A piece 23 of the same length can be cut from the lower end of the seed if necessary.

With the above method, each subsequent polycrystalline rod to be processed is always fused together with a crystal seed at a seed end consisting of hyper-pure monocrystalline material. As a result, the semiconductor rods processed have uniformly good electric properties regardless of the number of times one and the same seed is used.

The method has the further advantage that the molten zone does not pass through a locality where the cross section of the rod changes appreciably. This can be utilized in cases where the zone melting equipment is automated and the zone melting operation is controlled in dependence upon the electric current passing through the heater coil. The zone melting operation can then be regulated for constancy of the just mentioned current.

For the production of extremely pure monocrystalline, semiconductor rods from undoped semiconductor material up to about 20 zone passages lengthwise through the rod may be required. It has been found that the great number of zone passages may result in impairing the lifetime of the minority charge carriers because the recombination possibility becomes increased by crystal dislocations. Such dislocations occur in the seed crystal when fusing it together with the polycrystalline rod. The result of such dislocations is to unfavorably modify the crystal structure at the end of the seed down to approximately 2

cm. into the seed body. During the subsequent zone melting starting from the fusion bond, crystal displacements are dragged from the end of the seed into the rod being processed. The dislocations thus caused to migrate into the rod increase with an increasing number of zone passages and thus penetrate more and more into the semiconductor rod. It is a further object of my invention to eliminate such shortcomings and to thus render the production method more economical by reducing the quantity of unsuitable products. This object is achieved by substantially eliminating the crystal dislocations resulting from the fusion attachment of the seed to the rod at an earliest possible processing step. According to a more specific feature of the invention, the commencement of one of the first few zone passages effected after the seed is fused together with the polycrystalline rod, is located away from the fusion bond into the seed crystal. Preferably, this is done not later than in the fourth zone passage. This aspect of the invention will be further explained with reference to FIG. 2.

A monocrystalline seed 111 coaxially fused together with a polycrystalline semiconductor rod 121 to be converted to monocrystalline constitution is held in a holder comprising a cylindrical member 141 of ceramic material, a centering cup 151, a member of molybdenum strips 13 uniformly distributed about the periphery of the seed and acting as springs. The ceramic member 141 is held in proper position by a number of set screws in threaded engagement with the cup 151. The cup, and hence the seed 111, can be placed in rotation by means of a shaft 171 fastened by a screw bolt 161 to the centering cup 151. The upper portion of the rod 121 and its upper holder are not illustrated. A heating device permits melting a narrow zone 201 of the rod 111. The heater comprises an inductance coil 181 consisting preferably of copper tubing which, during operation, is traversed by cooling liquid. The coil 181 has its ends fastened to holders 191 which operates as current supply terminals for high-frequency alternating current.

During operation of the device the heater winding 181 is gradually moved upward from the seed 111 toward the opposite end of the rod, and this process is repeated. If for the production of a hyper-pure monocrystalline rod of semiconductor material, for example silicon, a predetermined number, for example 20, zone passages are necessary, then the first zone passage is preferably commenced immediately after fusing the seed 111 together with the rod 121 at the fusion point indicated by a dot-and-dash line 211. The next following zone passage is then commenced within the seed crystal at the location denoted by 221, so that the floating melting zone is pulled through the fusion point 211. The space of location 221 from the fusion bond 211 is preferably made 2 cm. or more. The next following zone passages again commence at the location 211. The commencement of the last zone passage can be placed at a location 231 away from the fusion bond into the rod being processed, thus being spaced from the fusion point 211 where crystal dislocations can be caused by the repeated melting. The location 231 is so chosen that the crystal structure is practically not affected and exhibits a greater degree of perfection.

Aside from improving the lifetime of the minority carrier in the rod portion adjacent to the seed, the methods of the invention have the further advantage that the entire process of converting a polycrystalline rod into a hyper-pure monocrystal can be completely performed automatically with automatic devices regulating the performance for constant current.

I claim:

1. A crucible-free, floating molten zone method of making a monocrystalline silicon semiconductor solid body from a polycrystalline silicon body, comprising supporting the polycrystalline solid body vertically, fusing a monocrystalline silicon seed crystal to the polycrystalline

silicon body, heating a transverse zone to produce a molten zone extending over the cross section of the body, repeatedly passing said molten zone along said initially polycrystalline body in one and the same direction, the passes starting at a point to include the location of fusion of the seed crystal to the polycrystalline body, the passes converting polycrystalline to monocrystalline silicon, and thereafter passing said molten zone in said direction but starting at another location removed from said location of fusion of the seed crystal but within the originally polycrystalline silicon body which is now monocrystalline silicon.

2. A crucible-free floating molten zone method of making a monocrystalline semiconductor body from a polycrystalline body, comprising supporting the polycrystalline body vertically, fusing a monocrystalline seed crystal to the polycrystalline body, heating a transverse zone to produce a molten zone extending over the cross section of the body, repeatedly passing said molten zone along said initially polycrystalline body in one and the same direction, the passes starting at a point to include the location of fusion of the seed crystal to the polycrystalline body, the passes converting polycrystalline to monocrystalline, and thereafter passing said molten zone in said direction but starting at another location removed, at least a distance equal to the diameter of the rod, from said location of fusion of the seed crystal but within the originally polycrystalline body which is now monocrystalline.

3. A crucible-free, floating molten zone method of making a monocrystalline germanium semiconductor body from a polycrystalline germanium body, comprising supporting the polycrystalline body vertically, fusing a monocrystalline germanium seed crystal to the polycrystalline germanium body, heating a transverse zone to produce a molten zone extending over the cross section of the body, repeatedly passing said molten zone along said initially polycrystalline body in one and the same direction, the passes starting at a point to include the location of fusion of the seed crystal to the polycrystalline body, the passes converting polycrystalline to monocrystalline germanium, and thereafter passing said molten zone in said direction but starting at another location removed from said location of fusion of the seed crystal but within the originally polycrystalline germanium body which is now monocrystalline germanium, severing the so produced monocrystalline rod at a location that is outside of the seed crystal and is within the originally polycrystalline rod, but which is now monocrystalline, removing the severed monocrystalline rod portion so produced, fusing a second polycrystalline rod to the other portion, at the location of severing, and repeating the passes defined above.

4. A crucible-free, floating molten zone method of making a monocrystalline silicon semiconductor body from a polycrystalline silicon body, comprising supporting the polycrystalline body vertically, fusing a monocrystalline silicon seed crystal to the polycrystalline silicon body, heating a transverse zone to produce a molten zone extending over the cross section of the body, repeatedly passing said molten zone along said initially polycrystalline body in one and the same direction, the passes starting at the location of fusion of the seed crystal to the polycrystalline body, the passes converting polycrystalline silicon to monocrystalline silicon, and thereafter passing said molten zone in said direction but starting at another location removed from said location of fusion of the seed crystal but within the originally polycrystalline silicon body which is now monocrystalline silicon, severing the so produced monocrystalline body at a location therein that is outside of the seed crystal and is within the originally polycrystalline silicon rod, but which is now monocrystalline silicon, and is removed from said location of fusion of the seed crystal, removing the severed monocrystalline body portion so produced, fusing a second polycrystalline silicon body to the other portion, at the loca-

tion of severing, and repeating the passes defined above.

5. A crucible-free, floating molten zone method of making a monocrystalline silicon semiconductor rod from a polycrystalline silicon rod, comprising supporting the polycrystalline rod vertically, fusing a monocrystalline silicon seed crystal to the polycrystalline silicon rod, heating a transverse zone to produce a molten zone extending over the cross section of the rod, repeatedly passing said molten zone along said initially polycrystalline rod in one and the same direction, the passes starting at a point to include the location of fusion of the seed crystal to the polycrystalline rod, the passes converting the polycrystalline silicon rod to a monocrystalline silicon rod, and thereafter passing said molten zone in said direction but starting at another location removed from said location of fusion of the seed crystal but within the originally polycrystalline silicon rod which is now monocrystalline silicon, severing the so produced monocrystalline body at a location therein that is outside of the seed crystal and is within the originally polycrystalline silicon rod, but which is now monocrystalline silicon, and is removed from said location of fusion of the seed crystal at least a distance substantially equal to the diameter of the rod being processed, removing the severed monocrystalline rod portion so produced, fusing a second polycrystalline silicon rod to the other portion, at the location of severing, and repeating the passes defined above.

6. A crucible-free, floating molten zone method of making a monocrystalline semiconductor rod from a polycrystalline rod, comprising supporting the polycrystalline rod vertically, fusing a monocrystalline seed crystal to the polycrystalline rod, heating a transverse zone to produce a molten zone extending over the cross section of the rod, repeatedly passing said molten zone along said initially polycrystalline rod in one and the same direction, the passes starting at a point to include the location of fusion of the seed crystal to the polycrystalline rod, the passes converting the polycrystalline rod to a monocrystalline rod, and thereafter passing said molten zone in said direction but starting at another location removed, at least a distance equal to the diameter of the rod, from said location of fusion of the seed crystal but within the originally polycrystalline rod but is now monocrystalline, severing the so produced monocrystalline body at a location therein that is outside of the seed crystal and is within the originally polycrystalline silicon rod, but which is now monocrystalline silicon, and is removed from said location of fusion of the seed crystal at least a distance substantially equal to the diameter of the rod being processed, removing the severed monocrystalline rod portion so produced, fusing a second polycrystalline rod to the other portion, at the location of severing, and repeating the passes defined above.

7. The method defined in claim 6, the semiconductor being germanium.

8. A method for producing a monocrystalline hyperpure semiconductor rod for electric purposes from a polycrystalline rod by crucible-free floating zone melting with the aid of a monocrystalline seed crystal fused to the rod by multiple zone passes in a common direction, comprising supporting the rod vertically, repeatedly passing the molten zone lengthwise through the semiconductor rod commencing each time at the fusion bond of the seed crystal, the method including the step of commencing at least one of the zone passes following the fusion of the seed to the rod at a location away from the fusion point into the seed crystal, the commencement of the last zone passage being placed away from the fusion point into the rod being processed at a distance at least equal to the rod diameter, severing the so produced monocrystalline body at a location therein that is outside of the seed crystal and is within the originally polycrystalline silicon rod which is now monocrystalline silicon, and is removed from said location of fusion of the seed crystal as least a distance

substantially equal to the diameter of the rod being processed, removing the severed monocrystalline rod portion so produced, fusing a second polycrystalline silicon rod to the other portion, at the location of severing, and repeating the passes defined above.

9. The method defined in claim 8, the seed crystal and the rod being silicon.

10. A method for producing a monocrystalline hyperpure semiconductor rod for electronic purposes from a polycrystalline rod by crucible-free floating zone melting with the aid of a seed crystal fused to the rod, comprising supporting the polycrystalline rod vertically; repeatedly passing the molten zone lengthwise through the semiconductor rod commencing each time at a point to include the location of the fusion junction of the seed crystal to the rod, then commencing a zone melting pass at a location in the said rod, namely outside of the seed crystal, and away from the fusion point of the seed crystal, the said location of the rod having lesser crystal lattice dislocations than said fusion junction, severing the so produced monocrystalline rod at a location that is outside of the seed crystal and is within the originally polycrystalline rod which is now monocrystalline, removing the severed monocrystalline rod portion so produced, fusing a second polycrystalline rod to the other portion, at the location of severing, and repeating the passes defined above.

11. A crucible-free, floating zone method for producing a monocrystalline hyperpure semiconductor rod for electronic purposes from a polycrystalline material comprising supporting a rod of the polycrystalline semiconductor material with its axis extending vertically, heat fusing a seed crystal to an end of said rod, diminishing the crystal dislocations in the fusion zone, resulting from the fusion attachment of the seed to the rod, by commencing a molten zone passage at a location displaced away from the fusion bond into the seed crystal a distance of at least two centimeters, said zone passage being then passed through the fusion bond into the rod, thereafter carrying out several molten zone passages commencing from the fusion bond and passing into said rod to convert the rod into monocrystalline material, and thereafter making another zone passage commencing at a location in said rod displaced away from the fusion zone a distance at least equal to the diameter of the rod, where the crystal dislocations are less than at the fusion bond.

12. A crucible-free, floating zone method for producing a monocrystalline hyperpure semiconductor rod for electronic purposes from a polycrystalline material, comprising supporting a rod of polycrystalline semiconductor material with its axis extending vertically, fusing a seed crystal to an end of said rod, crystal dislocations being caused in the fusion bond by said fusion, thereafter carrying out several molten zone passages commencing from the fusion bond and passing into said rod to convert the rod into monocrystalline material, and thereafter making another zone passage commencing at a location in said rod displaced away from the fusion zone a distance at least equal to the diameter of the rod, where the crystal dislocations are less than at the fusion bond.

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