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Knudsen

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[54] APPARATUS FOR CONTROL OF HEAT RADIATION IN ZONE MELTING

[72] Inventor: **Poul E. Knudsen**, Skodsborg, Denmark

[73] Assignee: **Haldor Frederik Axel Frydenlundsvej**, Vedbaek, Denmark

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[58] **Field of Search** ..... 23/273 SP, 301 SP

[56] **References Cited**

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Primary Examiner—Norman Yudkoff
Assistant Examiner—R. T. Foster
Attorney—Arnold Robinson

[57] **ABSTRACT**

Control of heat radiation from the "critical zone" adjacent the molten material in a floating-zone purification of a semiconductor monocrystal to achieve a low level of dislocations.

1 Claims, 2 Drawing Figures

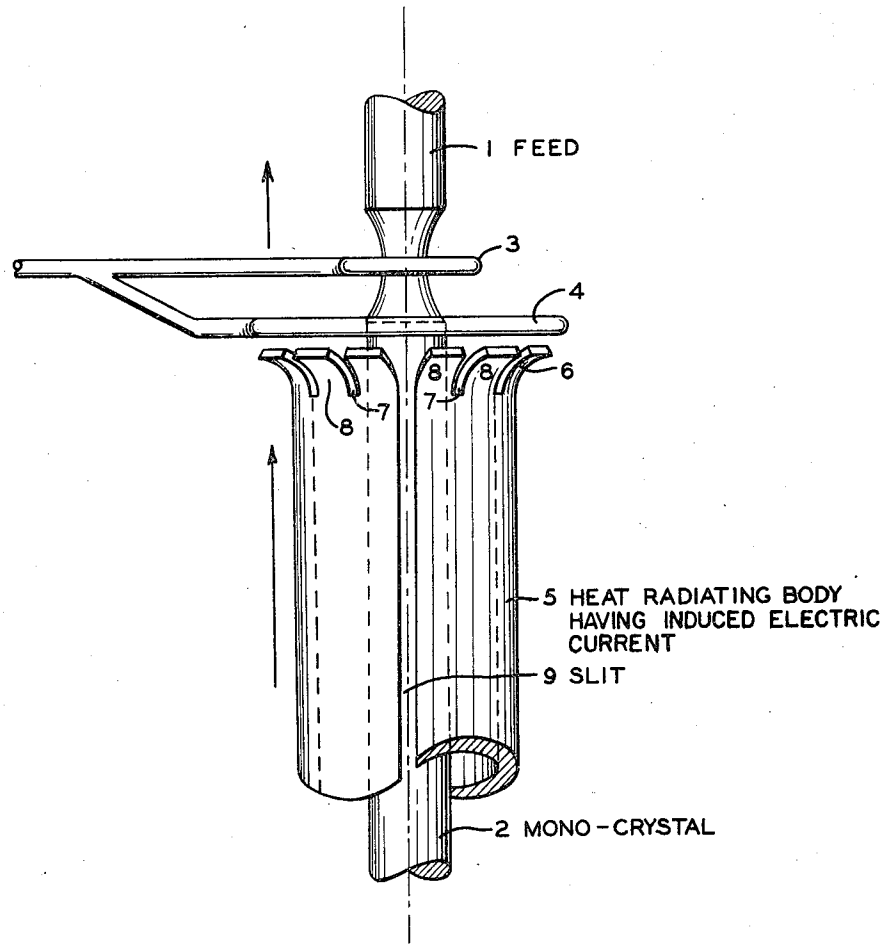


Fig. 1

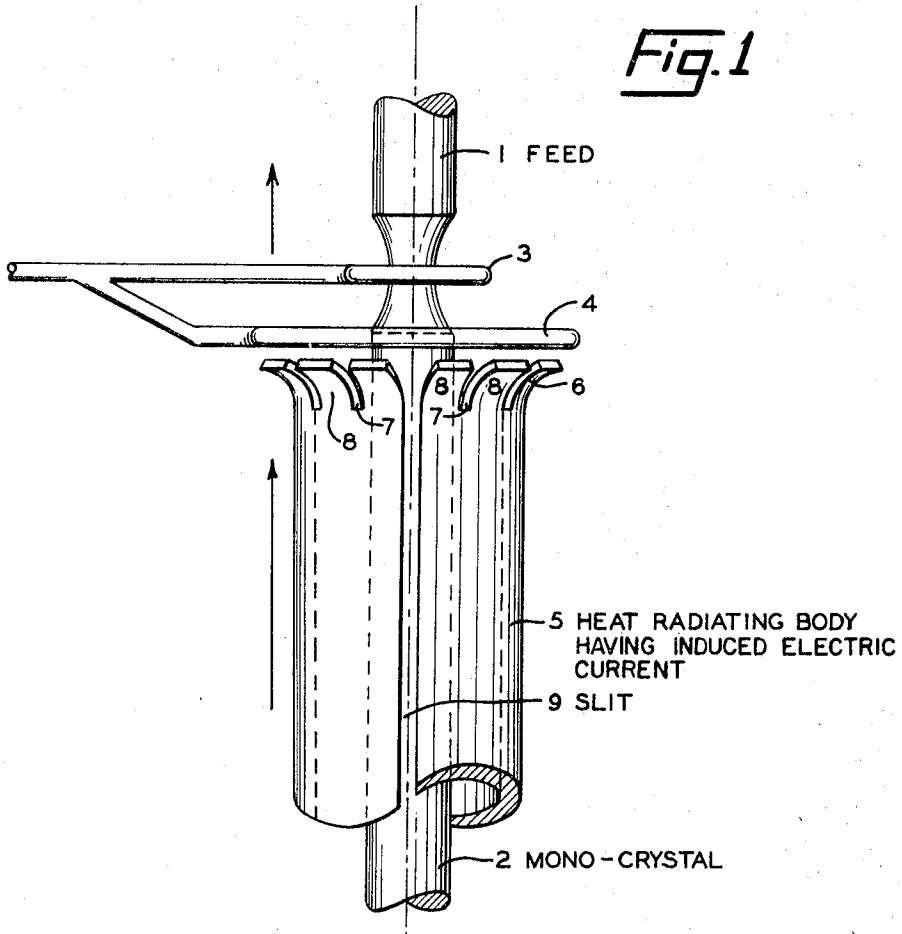
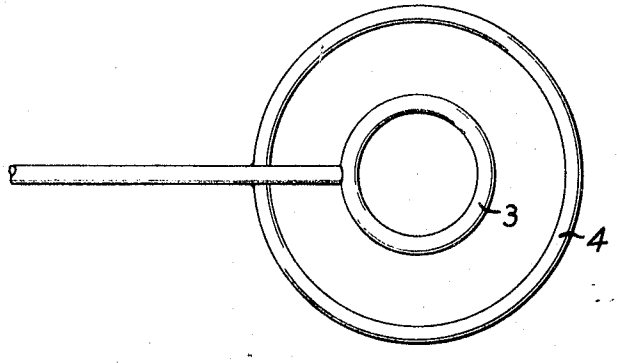

1 FEED
3
4
8
8
6
7
7
8
5 HEAT RADIATING BODY HAVING INDUCED ELECTRIC CURRENT
9 SLIT
2 MONO-CRYSTAL

Fig. 2


3
4

APPARATUS FOR CONTROL OF HEAT RADIATION IN ZONE MELTING

The present invention relates to the production of monocrystalline bodies of semiconductor materials, particularly but not exclusively materials such as silicon in a highly purified state having only a small number, approaching zero, of dislocations (expressed per square unit). I provide both an improved process for the production of such materials and apparatus for carrying out the process.

These materials are often produced by means of the so-called floating-zone process in which a heated zone comprising molten material is caused to move relative to a rod of crystalline semiconductor material, beginning from a seed crystal placed at one end of and united with the rest of the rod, which is called the feed. The seed material must be a single crystal and may have been made by a previous floating-zone process. The feed may consist either of polycrystalline material or of a single crystal in which it is desired to reduce the number of dislocations.

The heated zone may pass in an upward direction, the seed crystal being at the lower end of the rod, or in a downward direction, the seed being at the top. The relative movement between the heated zone and the rod may be effected by moving either or even both.

Hereinafter, when speaking in terms of up and down without further explanation, these terms are to be understood as referring to the arrangement where the seed is at the lower end. Further, for the sake of simplification, the invention will be explained in terms referring to the arrangement where the heated zone is moving and the rod is stationary, though this arrangement may well not be the most convenient and most commonly used.

In the heated zone the material is present partially in the molten and partially in the solid state. At a distance of only a few centimeters from the seed the number of dislocations in the recrystallized rod is independent of the number of dislocations prevailing in the seed, and an equilibrium will establish itself in which the number of dislocations created is equal to the number that disappears from the crystal. The extent to which such new dislocations form depends on the configuration of the interface between the molten and the crystalline surface called the solidification surface, and on the temperature gradients in radial and axial directions respectively, within the "critical zone" defining the temperature interval where dislocations can be formed by plastic deformation. If the radial gradient is zero and the axial gradient is constant in this "critical zone" and if, at the same time, the configuration of the solidification surface is planar, the lowest number of dislocations will result.

It has, however, been found difficult to produce and maintain in this "critical zone," the state in which ideal conditions exist for obtaining a low number of dislocations in the single crystal.

The heating of the material to produce melting within a heated zone passing along the rod may be effected by producing heat in the semiconductor material itself by generating an electric current therein by means of one or more induction coils surrounding the rod and carrying a current of high frequency, i.e., 200 kHz. to 5 MHz. In this way the heat is generated substantially within a shell of the material comprising the outermost few millimeters, whereas the heating of the remaining central parts of the rod depends on conduction of heat therefrom. In various ways, such as by using two induction coils of different diameter, the bigger one being placed a small distance below the smaller one, the field produced will be such that the heat produced per square unit of cross section of the rod decreases downwardly. Then by adjusting the current and the velocity with which the heated zone travels upwardly through the rod it has been found possible to obtain an approximately planar solidification surface. The approximation with which this has been attained is such that the finished monocrystal contains between 10,000 and 50,000 dislocations per square centimeter.

It is the aim of the invention to reduce this frequency of dislocation further, by producing surface temperature conditions in the "critical zone" which are closer to the above-named ideal conditions than has hitherto been possible.

In the "critical zone," cooling of the rod takes place by conduction of heat downwardly through the material of the rod and by radiation from the surface of the rod to the surroundings. This mechanism of cooling is not favorable to the achievement of the temperature gradients aimed at, even if the solidification surface is planar.

According to the present invention, in a process for the purification of a monocrystalline body of semiconductor material a heated zone comprising molten material is passed relatively through said body while controlling the heat radiated from the surface of said body within the hereinbefore defined "critical zone" adjacent said heated zone.

The configuration of the solidification surface behind the molten material of the heated zone is also maintained in a highly planar form.

Preferably, the heat radiated from the surface of said body in the "critical zone" is controlled by secondary heat-radiating means surrounding said body at least in part.

Further to the present invention, apparatus for the purification of a monocrystalline body of semiconductor material comprising zone-heating means adapted for movement relative to said body whereby a heated zone comprising molten material passes through the body, is characterized by secondary heat-radiating means surrounding said body at least in part whereby in operation the heat radiated from the surface of said body within the hereinbefore defined "critical zone" adjacent said heated zone is controlled.

The secondary heat-radiating means, normally a furnace, increases the ambient temperature in the "critical zone" and Thus acts to decrease the amount of heat radiated from the surface of the body within this "critical zone" and compensates to a desired degree for the loss of heat which would otherwise be suffered by radiation.

The furnace may surround the whole of the rod undergoing purification, but preferably surrounds little more than the "critical zone" and is arranged to move in conjunction with the heated zone comprising molten material.

This furnace can be heated in various ways such as creating ohmic heat therein by simply passing an electric current from a suitable source therethrough.

However, it has been found that the heat supplied in this way to the rod should have maximum intensity adjacent to the solidification surface where the temperature of the single crystal is high and tends to produce a high loss of heat to the surroundings.

Preferably, therefore, the heating furnace consists of an annular body of revolution surrounding the body, which is usually in the form of a rod, and heated by currents generated from the same induction coil or coils which are used for supplying the energy by which the travelling heating zone is created. This considerably simplifies the construction of the apparatus and the operation thereof.

If, however, the annular body has a good conductivity the currents induced in the upper part of the body may well be so strong that the electric field from the high frequency coil is unduly deformed, in some cases so much that the field in the "critical zone" just below the solidification surface will be seriously reduced. An appreciable alteration of said field must, however, be avoided because it would also cause an adverse alteration of the configuration of the solidification surface.

For this reason, the annular body is preferably cut through along a generatrix whereby current circulation in a plane parallel to that of the induction coil is prevented. On the other hand this does not prevent the annular body being heated by currents induced by the high-frequency coil since it is still possible to define closed circuits which intersect lines of flux from the field of the coil.

Although the currents thus generated will to some extent still deform the field produced by the induction coil adjacent

the solidification surface, including the "critical zone" just below the said surface, this undesirable effect not so unfavorable as in the case of an uninterrupted annular body. In fact, it is usually quite tolerable since it has proved possible to obtain in this way some decrease in the number of dislocations.

The best effect is, however, obtained by using an annular body that has been cut along a generatrix as mentioned above and in which additional slits have been cut in axial planes, said slits extending from the upper edge down to a certain depth, thereby separating the material of the upper part of the body into a number of flaps. Preferably, the flaps extend outwardly so as to define a conical part at about 45° with the axis of the body. When, during the passage along the rod, the induction means comprising a coil or coils is followed by such a body, the lowermost part of which has a shape of cylinder cut along a generatrix, and the upper part has the shape of flaps diverging from the cylinder as above described, eddy currents will be produced, not only in the cylinder but also in the flaps, without involving any appreciable decrease in the density of the part of the high-frequency field passing through the semiconducting material in the "critical zone" immediately at or below the solidification surface. In fact, the eddy currents within the individual flaps are generated by a part of the electrical field which, because of the diversion of the flaps in an outward direction from the axis, is more remote from the rod. The said eddy currents heat the flaps which in their turn radiate the heat to the surface of the rod. The cylindrical part of the body is heated by heat conducted from the flaps and by a current induced in this part of the material of the body from the induction coil or coils. In each flap a separate circular current of considerable strength is induced, particularly when the tips of the flaps are bent still further outwardly, in which case the tip portion of the flaps may well be approximately perpendicular to the lines of the field. This current produces a high temperature in the flaps, but contrary to what would be the case if the top end of the cylinder was not so incised, the direction of the circular currents in the lower part of the flaps, which is closest to the rod, is the same as in the coil and this does not weaken the field through the rod. On the contrary, owing to the fact that they are produced by means of a part of the field which is otherwise useless, they contribute to maintain or increase the current within the crystal in the "critical zone" just below the floating zone. At the same time, of course, the tips of the flaps must carry currents running in the opposite direction (since the said currents are circular ones) but these parts are more remote from the crystal rod and their weakening influence on the current induced therein is negligible. The axial temperature gradient in the cylindrical part can be regulated by adjusting the thickness of the wall or by an auxiliary induction coil encircling the annular body at a suitable level.

The annular body must be of conductive material having a high melting point. If a metal is used, care must be taken that it does not produce vapors tending to contaminate the semiconductor material. In some cases therefore nonmetallic substances such as pyrolytic graphite or silicon, are to be preferred.

Monocrystalline silicon having a dislocation number which is practically zero has been produced using this method and apparatus. Assuming that the feed is a rod having a diameter of 2.2–2.5 centimeters the smaller coil may have a diameter of 2.5–3.0 centimeters and the larger coil a diameter of 4.0–6.0 centimeters. The upper end of the conical part of the body of revolution may have a greater diameter of such as 4.2–6.5 centimeters and the lower cylindrical part may have an inner diameter of 3.0–5.0 centimeters. The axial progression rate may be 0.5 to 5.0 millimeters/minute but it must be understood that these dimensions all depend on the factors mentioned in the foregoing general description of the invention and in the prior art and that they must be such as to produce an approximately plane solidification surface. However, with dimensions of this order it is preferred that the annular body should have an axial length of 7 centimeters or more and the upper part thereof should operate at a temperature of at least 1,400° C.

A preferred embodiment of the present invention will now be described by way of example with reference to the accompanying drawings which represents a schematic arrangement for refining a silicon rod by zone melting.

FIG. 1 is a side view of the apparatus of this invention in which the elements are shown in schematic arrangement;

FIG. 2 is a plan view of the heating coils 3 and 4 of FIG. 1.

In the drawing a silicon rod 1,2 about 2.5 centimeters in diameter comprises the feed 1 and the refined monocrystal 2 during zone-melting. Heating means comprising coil 3 and coil 4 are arranged and adapted so that they can be moved axially in relation to the silicon rod. The rod is held vertically and the heating means are moved upwards during the refining process. The upper coil 3 has a smaller diameter than coil 4 and both are connected to a source of high-frequency electricity (not shown). Mounted in common with the coils 3,4 is the heat-radiating body comprising a cylindrical portion 5 and a diverging portion 6. A plurality of slits 7 of 1 cm. length are formed at 1 cm. intervals around the periphery of portion 6, thus defining flaps 8 which are bent to diverge by 45° from the axial direction of the body. In operation, eddy currents are produced in the flaps 8 which are thus heated by energy which would otherwise be lost. When refining silicon, the flaps 8 are preferably heated to a temperature of about 1,400° C.

A slit 9 is provided in cylindrical portion 5 of the body to inhibit the formation therein of currents which would adversely affect the operation of the apparatus.

This apparatus can of course be modified in detail without departing from the principles of our invention. For example, the thickness of material forming the cylindrical portion 5 may increase from the bottom thereof to the top, or portion 5 may be surrounded by a further induction coil (not shown).

In operation, the coils 3,4 and the body comprising the portions 5,6 are slowly moved upwards over the silicon rod so that a heated zone as hereinbefore described is slowly moved up through the rod, the conditions giving a solidification surface which is planar to a high degree. Using my process and apparatus I have produced silicon monocrystals having only 2,000 dislocations/square centimeter in the core and skin portions of the crystal and no detectable dislocations at all in the remaining portion of the crystals. This is to be compared with 30,000 dislocations/square centimeter in crystals of silicon refined by conventional floating-zone techniques.

What is claimed is:

1. Apparatus for the purification of a monocrystalline body of semiconductor material comprising zone-heating means adapted for movement relative to said body whereby a heated zone comprising molten material can be passed through the body, said zone-heating means comprising at least one induction coil and being mounted in common with secondary heat-radiating means comprising an annular wall forming a body of revolution arranged with its axis common with that of the body of semiconductor material to be purified, said wall being divided along its length by a slit lying parallel to said axis, and having one end portion divided into small flaps which diverge from the common axis, the arrangement being such that in operation the induction coil heats the heated zone by induction currents and also supplies heat to the secondary heat-radiating means in the same manner, thereby controlling the heat radiated from the surface of said body within the so-called "critical zone" adjacent said heated zone, being the temperature interval where dislocations can be formed by plastic deformation.

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