May 23, 1967 R. J. BINDER 3,321,299

APPARATUS AND PROCESS FOR PREPARING SEMICONDUCTOR RODS

Filed Oct. 13, 1964 2 Sheets-Sheet 1

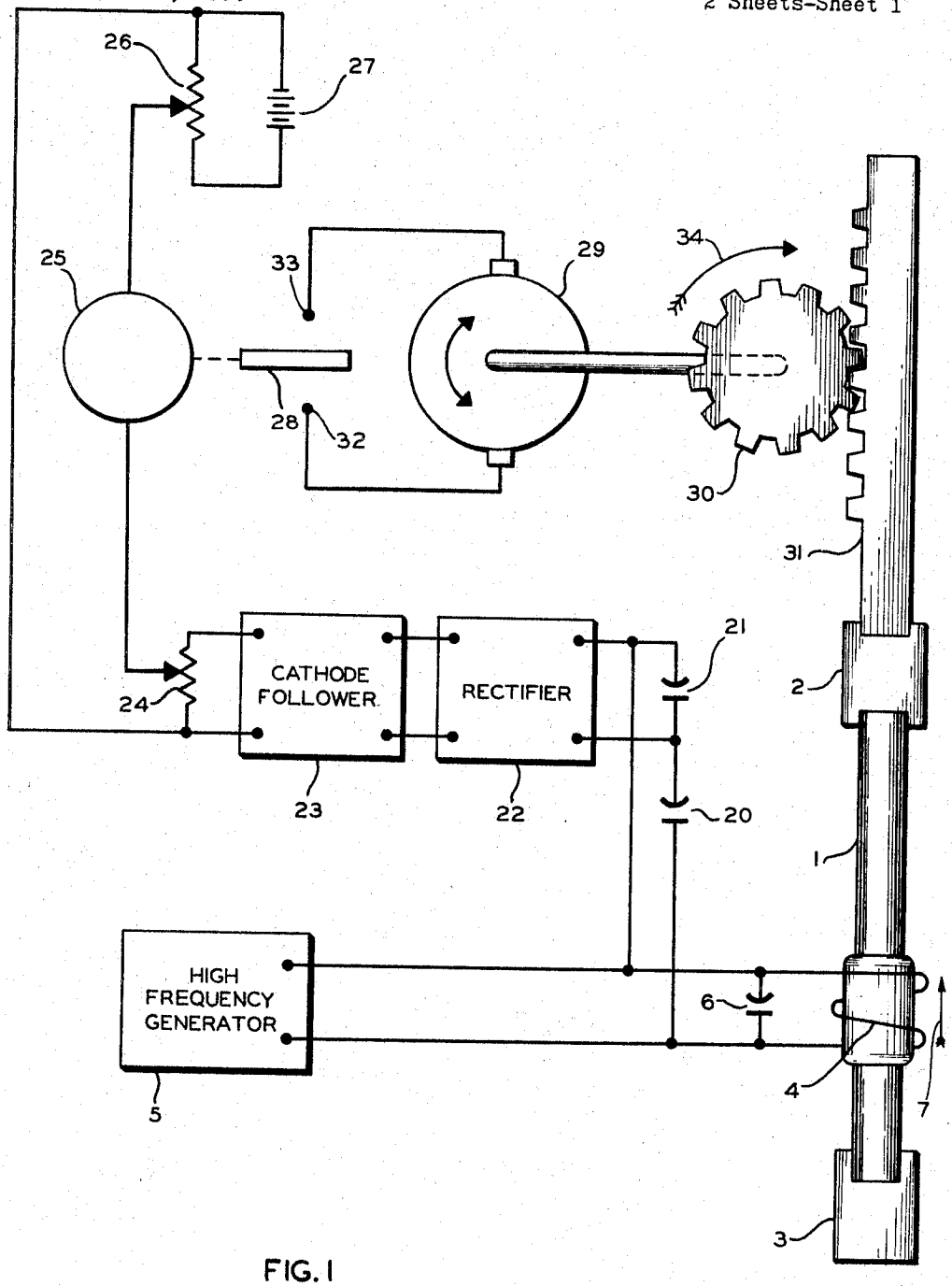


FIG. 1

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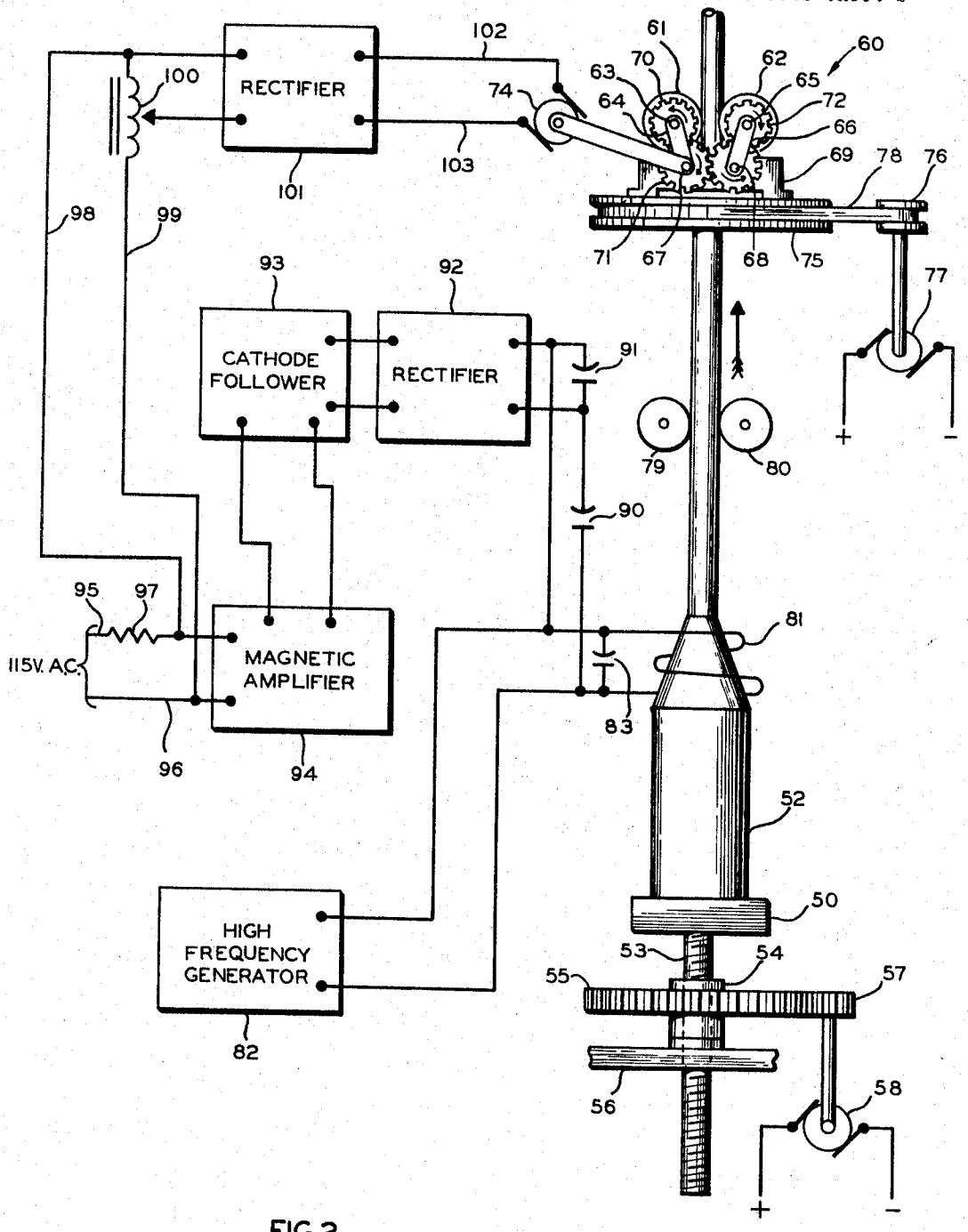


FIG. 2

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3,321,299

APPARATUS AND PROCESS FOR PREPARING SEMICONDUCTOR RODS

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This invention relates to the preparation of semiconductor rods and is particularly concerned with an apparatus and process for automatic thickness regulation incident to zone melting a vertically positioned rod of semiconductor material.

Semiconductor materials are zone melted for a number of purposes. For example, zone melting operations are conducted to effect purification of semiconductor materials, to effect uniform distribution of carrier impurities, in semiconductor materials, and to effect the transformation of polycrystalline semiconductor materials into a rod of semiconductor material formed from a single crystal. In other instances the zone melting operation is conducted primarily to effect a change in the diameter of a rod of semiconductor material or to remove irregularities in the diameter of a rod of material. Instances where it is desirable to effect a change in the diameter of a rod of semiconductor material include the production of slim rods for use in a decomposer and, in many instances, the production of seeds for use in the manufacture of single crystal material.

In all operations of the above type it is normally desirable that the rod of semiconductor material resulting from the zone melting operation be predominantly of a uniform diameter. For example, in the manufacture of mono-crystalline semiconductor rods it is of prime importance that the semiconductor rod produced be of controlled uniform diameter. By accurately controlling the diameter such advantages accrue to the product as exhibiting a more uniform radial resistivity gradient and improved yields due to a more uniform diameter as well as the ability to make larger diameter rods with improved yields.

Various automatic methods for controlling the diameter of the semiconductor rod being formed during the zone melting process are known and such methods rely on monitoring the diameter by optical means, temperature-sensitive means, electrical means and various combinations of these means. For example, in the automatic control by electrical monitoring means the D.C. anode current driving the high frequency source connected to the inductive heating element (work coil), which changes with a changing thickness of the rod, is monitored and used to control through manipulative arrangements the diameter of the semiconductor rod. Such control means has many limitations among which are its sensitivity and responsiveness to changing conditions.

As will be more fully discussed hereinafter, this invention is directed to an improved electrical control means which is highly versatile in that it can be used with many and various types of mechanical arrangements for controlling the diameter of semiconductor rods and which minimizes or obviates the limitations of the foregoing methods.

According to the present invention, there is provided a process and apparatus for automatic thickness regulation of a semiconductor rod as it is being formed from a molten zone of semiconductor material utilizing the voltage of the high frequency work coil used to produce the molten zone by inductive heating, which voltage changes with a changing thickness of the semiconductor rod, for controlling means to effect a change in the diameter of the interface of resolidification which restores the

voltage to a predetermined voltage when said voltage changes therefrom, all of which will be more fully discussed hereinafter. As is known, the interface of resolidification, that is, the boundary between the molten zone and the solid semiconductor rod of desired thickness formed therefrom, when varying in diameter results in a variation of the load impedance. This load variation has now been found to effect a proportional variation in the work coil voltage such that a definite voltage value will correspond to a definite diameter of the semiconductor rod.

The various objects and features of the invention will be brought out in the description and in order to facilitate the description and understanding of the invention, reference is made to the appended drawings in which:

FIGURE 1 shows the principles underlying the operation of one embodiment of the present invention; and

FIGURE 2 shows the principles underlying the operation of another embodiment of the present invention.

Referring now to FIGURE 1 which illustrates schematically one embodiment of the present invention, a vertically positioned semiconductor rod **1**, such as a polycrystalline silicon or germanium rod, is end supported at **2** and **3** as shown. A high frequency work coil **4** is mounted concentrically about a portion of the rod and when energized establishes a molten zone within the portion of the rod. The foregoing arrangement may be surrounded by a tube (not shown), such as a quartz tube, through which a protective atmosphere, such as argon or hydrogen, is passed or the tube may be provided with a vacuum. The work coil is arranged to move axially with respect to the rod at a given speed while being energized by a high frequency generator **5**. Within the circuit of the work coil and the high frequency generator a resonating capacitor **6** is provided in parallel to permit a large circulating current in the resonant circuit of which the work coil is a part, while the high frequency source has to supply only a relatively small current. The frequency of the generator **5** is preferably so adjusted that it is almost, but not quite, identical with the natural frequency of the resonant circuit so that the generator operates on a flank of the resonance peak in the current-versus-frequency characteristic of the resonant circuit. In operation, the work coil when energized moves axially of the rod in the direction indicated by the arrow **7** and establishes a moving molten zone within the rod which by controlling the conditions thereof permits a semiconductor rod of desired diameter to be produced. In cases such as the production of a purified monocrystalline semiconductor rod, the operation can be performed in a reiterative manner in order to obtain a desired degree of purity.

The means for controlling the diameter of the finished semiconductor rod is provided by an electrical circuit, electrically connected in parallel with the high frequency power circuit, which monitors the voltage of the power circuit and, when the voltage of such circuit starts to change from a predetermined value, the change is immediately detected by the monitor circuit which activates and controls a mechanical arrangement for compressing or stretching the rod in order to effect a change in the diameter of the interface of resolidification which restores the voltage to the predetermined value thereby producing a semiconductor rod of a desired uniform diameter.

The monitor circuit is provided with measuring capacitors, **20** and **21**, which prevent any appreciable current from being drawn from the high frequency power circuit, a rectifier **22** which rectifies the alternating current voltage to a pulsed direct current voltage and which may be provided with a smoothing capacitor, if desired, and connected

in series therewith, a cathode follower **23** which permits the pulsed direct current voltage from the circuit to be used and/or measured without drawing any appreciable current from the circuit. A resistor **24** is provided across the pulsed direct current voltage and connected in series therewith a polarized relay **25** and an input resistor **26** provided with a set point input. Connected in parallel with the input resistor **26** is a battery **27**. The polarized relay **25**, through the contact spring **28**, positions, by way of a motor **29** and a suitable gear (not shown), a toothed wheel **30** which drives a rack arrangement **31** attached to one end of the semiconductor rod and which can effect, if desired, either a compressing or stretching force on the rod.

In the operation of the electrical monitor means, the input resistor **24** is adjusted by the set point input so that there is no voltage drop across the polarized relay **25** when the work coil is operating at its predetermined voltage value corresponding to the desired thickness of the rod and the contact spring **28** of the polarized relay **25** is positioned in a neutral position as shown. If the diameter of the interface of resolidification changes thereby changing the thickness of the rod during the operation, the voltage supplied by the high frequency power source to the work coil will also change due to a change in the electromagnetic coupling between the semiconductor rod and the work coil, and this change in voltage will immediately be detected by the capacitively coupled monitor circuit which will register a change in voltage across the polarized relay **25** without materially influencing the voltage of the work coil, thereby establishing a current which will cause the relay **25** to energize and position the contact spring **28** to establish contact with either point **32** or **33** depending on the voltage change taking place. When closure of the circuit is established by contact through the point to the motor **29**, the motor will operate the toothed gear **30** which effects movement of the rack **31** such that a compressing or stretching force is maintained on the rod until the semiconductor rod is again at the desired uniform thickness and the voltage in the work coil circuit is again at its predetermined value. For example, if the diameter of the semiconductor rod starts to become smaller during the operation, the voltage of the work coil will start to increase and this increase in voltage will be detected by the monitor circuit which will energize the relay **25** and position the contact spring **28** in such a manner that it closes, for example, contact **33** to cause the operation of the motor **29**, so as to rotate the toothed wheel **30** in the direction of the arrow **34**, resulting in the compression of the semiconductor rod until the work coil voltage is again at its predetermined voltage value, causing the relay via the monitor circuit to restore the contact spring to the neutral position. In this manner, the thickness of the semiconductor rod is controlled by the highly accurate and rapidly reacting voltage change in the monitor circuit which occurs when the diameter of the interface of resolidification changes thereby permitting a semiconductor rod to be produced with a desired uniform diameter.

With particular reference to FIGURE 2 of the drawings there is illustrated schematically another embodiment of the present invention. In this figure there is shown a lower rod holding means **50** designed to secure the lower end of a rod of semiconductor material **52** in a vertical position. The rod holder **50** is supported upon a threaded vertically disposed shaft **53** which engages an internally threaded collar **54** carrying a horizontally disposed gear **55**, the collar **54** being rotatably supported upon a support frame member **56** bored to receive the vertical shaft **53**. Horizontally disposed gear **55** is in operative engagement with a second gear **57** which is driven through a conventional reduction gear train, not illustrated, by a suitable electric motor **58**. Shaft **53** is free to move vertically but is keyed against rotation so that by rotation of gear **55**, the shaft **53** and rod holder **50** can be moved vertically as desired.

The reference numeral **60** generally indicates a second rod holding means which in this instance is shown to comprise a pair of grooved rollers **61** and **62**. The roller **61** is mounted upon a shaft **63** which is journaled through a pair of support arms, one of which is shown at **64**, on either side of the roller **61**. Roller **62** is similarly mounted upon a shaft **65** which is journaled at either end through a pair of support arms one of which is shown at **66**. Support arms **64** and **66** are respectively carried by a pair of shafts **67** and **68** which are journaled through a support member **69**. The support arms **64** and **66** are rotatable about shafts **67** and **68**, respectively, but relative movement between the arms and shafts **67** and **68** is preferably not friction free for reasons which will subsequently be made apparent.

Secured to one end of roller **61** is a gear **70** which meshes with a gear **71** secured to shaft **67**. Similarly, secured to one end of roller **62** is a gear **72** which meshes with a gear **73** secured to shaft **68** and which in turn meshes with gear **71** so that gear **71** drives both rollers **61** and **62** but in opposite directions. A suitable electric motor schematically illustrated at **74** is provided for driving shaft **67**, to which gear **71** is secured, through a suitable conventional gear reduction train, not illustrated.

The reference numeral **75** designates a rotary support platform upon which the rod holding means **60** is positioned. While it is not necessary that platform **75** rotate it has been found and is well known in the art that rotation of one or both rod holding means securing the ends of a rod of semiconductor material being zone refined is normally desirable and gives improved results. A small pulley **76** driven by an electric motor schematically illustrated at **77** is provided for rotating platform **75** by means of a belt **78** running in a suitable groove in the periphery of the platform. To reduce vibration and assist in maintaining the upper solid portion of rod **52** in proper alignment, a pair of freely rotatable idler rollers **79** and **80** are disposed below platform **75** and are yieldably biased toward each other to form a nip through which rod **52** passes.

The reference numeral **81** designates a conventional high frequency work coil (heater coil) operatively disposed around the rod of semiconductor material **52**. The work coil **81** is electrically connected to a suitable source of high frequency current such as a conventional high frequency generator schematically illustrated at **82** so that by passing a high frequency current through the coil **81** a molten zone can be created in the upstanding rod of semiconductor material **52**. Within the circuit of the work coil and the high frequency generator a resonating capacitor **83** is provided in parallel to permit a large circulating current in the resonant circuit of which the work coil is a part, while the high frequency source has to supply only a relatively small current. The frequency of the generator **82** is preferably so adjusted that it is almost, but not quite, identical with the natural frequency of the resonant circuit so that the generator operates on a flank of the resonance peak in the current-versus-frequency characteristic of the resonant circuit.

With the upper extremity of the rod held by rollers **61** and **62** in contact with lower extremity of the rod held by holder **50** through the molten zone, the motors **58**, **77** and **74** are placed in operation and the rate of operation of motors **58** and **74** are correlated so that the upward movement of the lower solid extremity of rod **52** relative to the rate of upward movement of the upper solid extremity of rod **52** is such that a small diameter rod of the desired mean diameter is withdrawn from the molten zone. As the lower extremity of rod **52** is pushed upwardly into the molten zone by means of motor **58**, the upper solid extremity of rod **52** is withdrawn from the molten zone by motor **74** at a rate such that the two solid portions of the rod are continuously receding from each other, and the large diameter feed stock is transformed into a small diameter product by means of the zone refining operation.

It was previously mentioned that movement of arms **64** and **66** relative to shafts **67** and **68** is preferably not friction free. It will now be seen that the reason for this is that, in normal operation, friction between shaft **67** and arm **64** and between shaft **68** and arm **66** results in rollers **61** and **62** being urged toward each other so that the upper solid portion of rod **52** is gripped more forcefully. This reduces the possibility of slippage between rod **52** and rollers **61** and **62** and reduces the extent to which rollers **61** and **62** need be spring biased toward each other.

The means for controlling the diameter of the finished semiconductor rod is provided by an electrical circuit, in parallel with the high frequency power circuit, which monitors the voltage of the power circuit and, when the voltage of such circuit starts to change from a predetermined value, the change is immediately detected by the monitor circuit which activates and controls means for restoring the voltage to the predetermined value thereby maintaining the interface of resolidification with a uniform desired diameter and thus producing a semiconductor rod of a desired uniform diameter.

The monitor circuit is provided with measuring capacitors, **90** and **91**, which prevent any appreciable current from being drawn from the high frequency power circuit, a rectifier **92** which rectifies the alternating current voltage to a pulsed direct current voltage and which may be provided with a smoothing capacitor, if desired, and connected in series therewith a cathode follower **93** which draws no appreciable current from the work coil circuit and which provides a biasing means for controlling a magnetic amplifier **94**. The amplifier, connected in series with the cathode follower, amplifies the pulsed direct current voltage input. The amplifier, in turn, is connected through suitable leads **95** and **96** to a source of alternating current electricity. A resistor, indicated by the reference numeral **97**, is disposed in lead **95** between the source of electric power and the magnetic amplifier, and a lead **98** is connected to lead **95** intermediate resistor **97** and magnetic amplifier **94**. It will be seen that with this arrangement the voltage across leads **98** and **99** varies with changes in the voltage across the work coil circuit without materially influencing the work coil voltage. A lead **99**, connected to lead **96**, together with lead **98** supplies power to an adjustable auto-transformer **100** which in turn is connected to a full wave rectifier indicated by the reference numeral **101**. Rectifier **101** supplies a controlled rectified voltage to motor **74** through commutator rings, not illustrated, and leads **102** and **103**. The voltage supplied to motor **74** with this arrangement can be manually controlled by means of variable transformer **100** and is automatically varied in response to variations in the voltage of the work coil circuit.

In the operation of the electrical monitor means, the motor **74** is adjusted to a proper pull rate, i.e., the rate at which the upper solid extremity of rod is withdrawn from the molten zone, when the work coil is operating at its predetermined voltage value corresponding to the desired thickness of the rod. During the pulling operation, if the diameter of the interface of resolidification changes thereby changing the desired thickness of the rod, the voltage supplied by the high frequency power source to the work coil will also change due to a change in the electromagnetic coupling between the semiconductor rod and the work coil, and this change in voltage will immediately be detected by the capacitively coupled monitor circuit which will register a change in voltage across the magnetic amplifier **94**, i.e. leads **95** and **96**, which in turn increases or decreases the speed of motor as the case may be. The motor speed, therefore, will adjust the pull rate such that the diameter of the interface of resolidification returns to its desired dimension, thereby, restoring the voltage in the work coil circuit to its predetermined value, thus maintaining the semiconductor rod at the desired uniform thickness.

It will be understood that rod diameter control apparatus in accordance with this invention can be utilized with any zone refining apparatus, whether of the gas or vacuum type. It will likewise be understood that various modifications may be made in the electrical or mechanical sytems of the embodiments illustrated without departing from the spirit of the invention.

Having thus described my invention and several specific embodiments thereof, what I desire to claim and secure by Letters Patent is:

1. In a method of zone melting a rod of semiconductor material in which the rod is vertically supported at both ends and a molten zone, produced by inductive heating through the medium of a high frequency work coil surrounding said rod, is formed in said rod intermediate the two ends thereof, said molten zone being caused to move relative to said rod along the longitudinal axis thereof to establish an interface of resolidification between said molten zone and the rod formed therefrom, the improvement which comprises utilizing the voltage of said work coil, which voltage changes with a change in the diameter of the interface of resolidification, for controlling means to effect a change in the diameter of the interface of resolidification which restores said voltage to a predetermined voltage when said voltage changes therefrom thereby maintaining said interface of resolidification with a substantially uniform diameter and thus forming said semiconductor rod with a substantially uniform diameter.

2. In a method of zone melting a rod of semiconductor material in which the rod is vertically supported at both ends and a molten zone, produced by inductive heating through the medium of a high frequency work coil surrounding said rod, is formed in said rod intermediate the two ends thereof, said molten zone being caused to move relative to said rod along the longitudinal axis thereof to establish an interface of resolidification between said molten zone and the rod formed therefrom, the improvement which comprises monitoring, by means of a control circuit electrically connected in parallel to said work coil, the voltage of said work coil, which voltage changes with a change in the diameter of the interface of resolidification, and activating means to effect a change in the diameter of the interface of resolidification which restores said voltage to a predetermined voltage when said voltage changes therefrom thereby maintaining said interface of resolidification with a substantially uniform diameter and thus forming said semiconductor rod with a substantially uniform diameter.

3. A method according to claim **2**, wherein said control circuit is capacitively coupled to said work coil.

4. A method according to claim **3**, wherein said means effects a change in the diameter of the interface of resolidification by an applicable stretching and compressing force applied to said semiconductor rod, said applicable force being in the direction to restore the diameter of the interface of resolidification to a substantially uniform diameter.

5. A method according to claim **4**, wherein said force is applied to the end of said semiconductor rod toward which the molten zone is caused to move.

6. A method according to claim **5**, wherein said control circuit activates said means to reduce the diameter of the interface of resolidification when the voltage of said work coil increases above the predetermined voltage.

7. A method according to claim **5**, wherein said control circuit activates said means to increase the diameter of the interface of resolidification when the voltage of said work coil decreases below the predetermined voltage.

8. In an apparatus for zone melting a rod of semiconductor material comprising upper and lower holding means for supporting the ends of a rod of semiconductor material such that the longitudinal axis of said rod extends vertically, a high frequency work coil surrounding

said rod for providing a molten zone by inductive heating in said vertically extending rod of semiconductor material, traverse means for effecting relative movement of said work coil longitudinally of said vertically extending rod of semiconductor material to establish an interface of resolidification between said molten zone and the rod formed therefrom, the improvement which comprises circuit means for monitoring the voltage of said work coil, which voltage changes with a change in the diameter of the interface of resolidification, and control means responsive to said circuit means to effect a change in the diameter of the interface of resolidification which restores said voltage to a predetermined voltage when said voltage changes therefrom to thereby maintain said interface of resolidification with a substantially uniform diameter thus forming said semiconductor rod with a substantially uniform diameter.

9. Apparatus according to claim **8**, wherein said circuit means is capacitively coupled to said work coil.

10. Apparatus according to claim **9**, wherein said control means effects a change in the diameter of the interface of resolidification by an applicable stretching and compressing force applied to said semiconductor rod, said applicable force being in the direction to restore the diameter of the interface of resolidification to a substantially uniform diameter.

11. Apparatus according to claim **10**, wherein said force is applied to the end of said semiconductor rod toward which the molten zone is caused to move.

12. Apparatus according to claim **11**, wherein said circuit means activates said control means to reduce the diameter of the interface of resolidification when the voltage of said work coil increases above the predetermined voltage.

13. Apparatus according to claim **11**, wherein said circuit means activates said control means to increase the diameter of the interface of resolidification when the voltage of said work coil decreases below the predetermined voltage.

14. In an apparatus for zone melting a rod of semiconductor material comprising upper and lower holding means for supporting the ends of a rod of semiconductor material such that the longitudinal axis of said rod extends vertically, a high frequency work coil surrounding said rod for providing a molten zone by inductive heating in said vertically extending rod of semiconductor material, traverse means for effecting movement of said work coil longitudinally of said vertically extending rod of semiconductor material to establish an interface of resolidification between said molten zone and the rod formed therefrom, the improvement which comprises a monitoring circuit comprised of, in combination, measuring capacitors for preventing any appreciable current from being drawn from said work coil, a rectifier for rectifying the alternating current voltage to a pulsed direct current voltage and a cathode follower for premitting the pulsed direct current voltage to be used without materially influencing said work coil voltage, and control means responsive to said circuit means comprised of, in combination, a polarized relay disposed between said cathode follower and a direct current voltage source, a motor actuated by said polarized relay when a voltage drop is impressed across said relay, a toothed wheel driven by said motor when actuated and a rack arrangement attached to the end of said rod for effecting a compressing and stretching force on said rod when driven by said wheel.

15. In an apparatus for zone melting a rod of semiconductor material comprising upper and lower holding means for supporting the ends of a rod of semiconductor material such that the longitudinal axis of said rod extends vertically, a high frequency work coil surrounding said rod for providing a molten zone by inductive heating in said vertically extending rod of semiconductor material, traverse means for effecting movement of said vertically extending rod through said work coil to establish an interface of resolidification between said molten zone and the rod formed therefrom, said traverse means adapted to withdraw said rod from sail molten zone at a rate in excess of that at which said rod is passed to said molten zone, the improvement which comprises a monitoring circuit comprised of, in combination, measuring capacitors for preventing any appreciable current from being drawn from said work coil, a rectifier for rectifying the alternating current voltage to a pulsed direct current voltage, a cathode follower for permitting the pulsed direct current voltage to be used without materially influencing said work coil voltage and an amplifier for amplifying the direct current voltage, and control means responsive to said circuit means comprised of, in combination, an alternating current source, an adjustable auto-transformer and a rectifier for supplying a controlled rectified voltage to said traverse means.

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