July 24, 1962 W. KELLER ET AL 3,046,379

METHOD AND APPARATUS FOR ZONE MELTING OF SEMICONDUCTOR MATERIAL

Filed Sept. 9, 1960 2 Sheets-Sheet 1

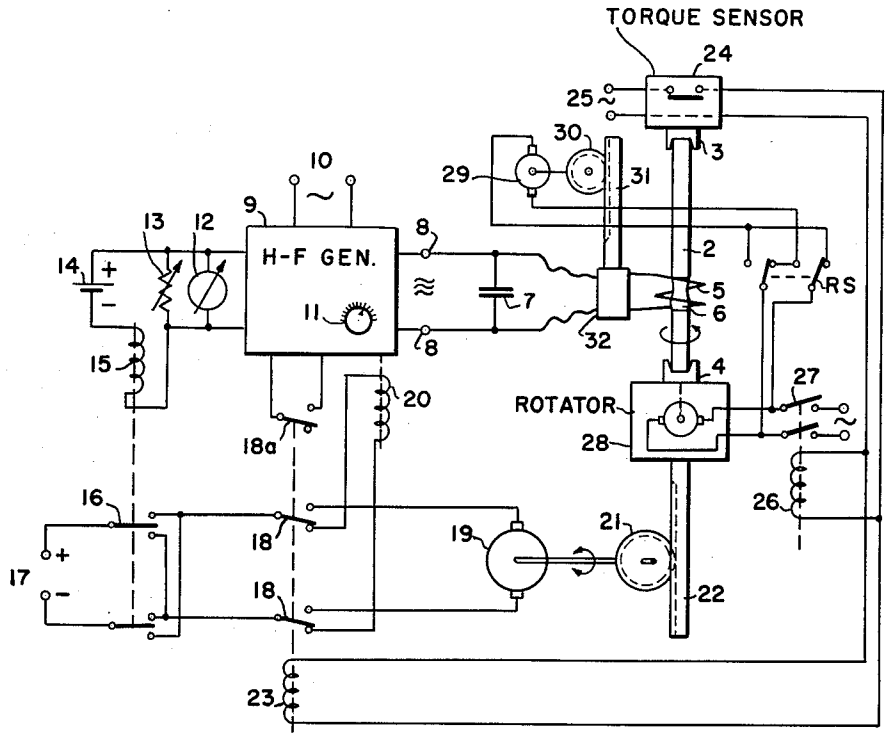


FIG. 1

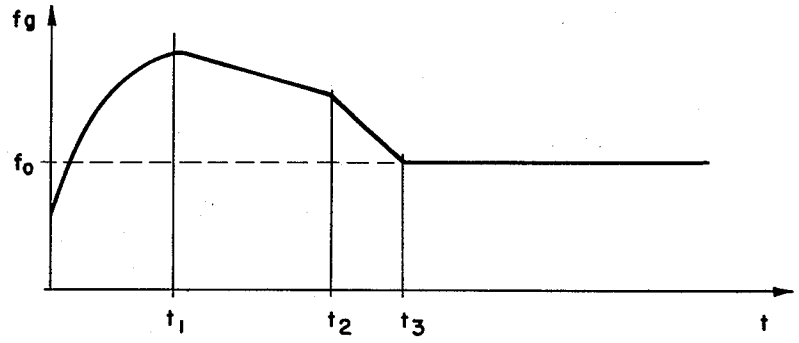


FIG. 2

July 24, 1962 W. KELLER ET AL 3,046,379
METHOD AND APPARATUS FOR ZONE MELTING
OF SEMICONDUCTOR MATERIAL
Filed Sept. 9, 1960 2 Sheets-Sheet 2

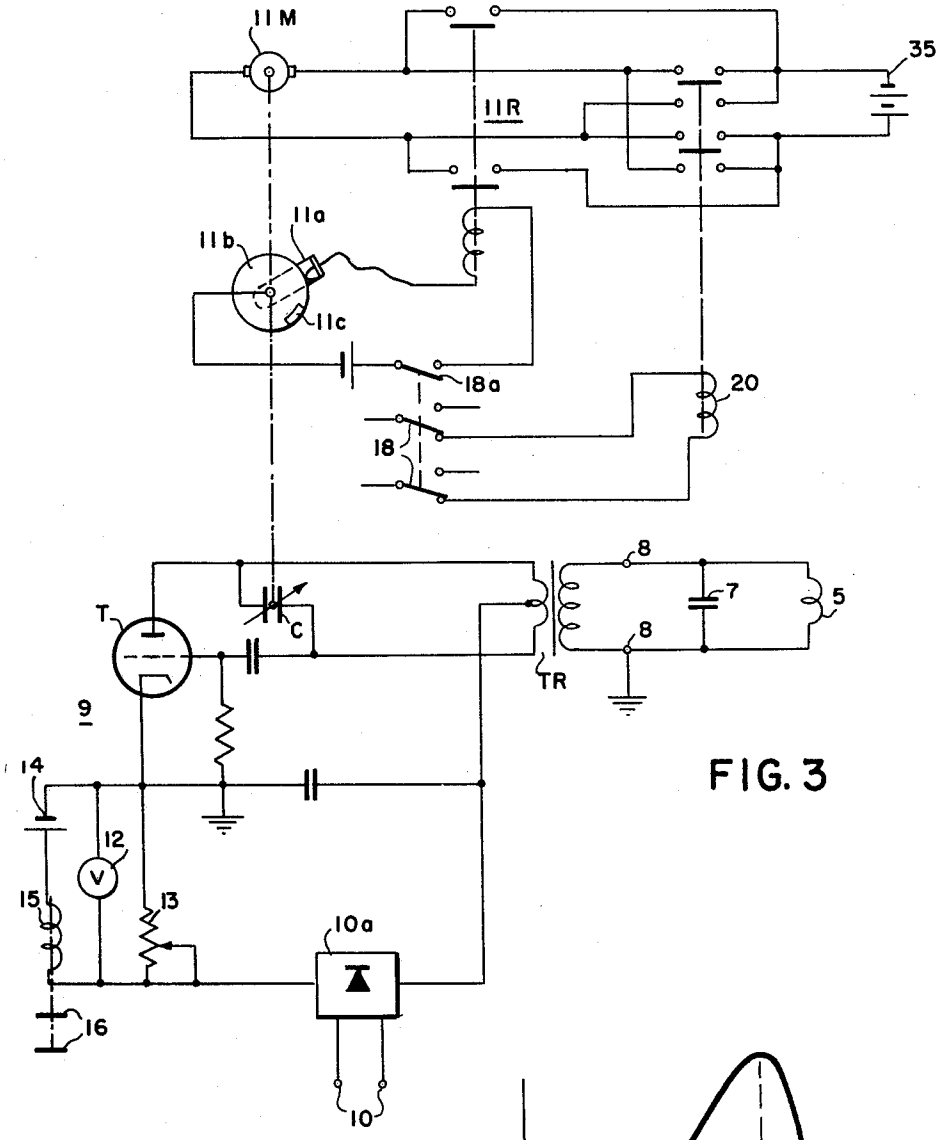


FIG. 3

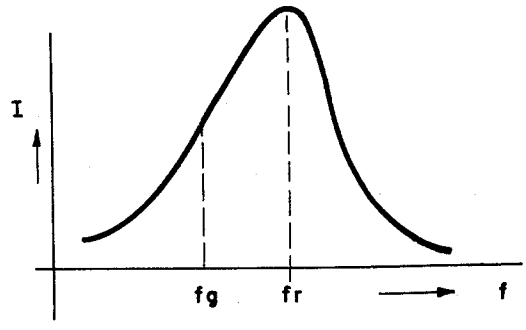


FIG. 4

United States Patent Office 3,046,379 Patented July 24, 1962

3,046,379

METHOD AND APPARATUS FOR ZONE MELTING OF SEMICONDUCTOR MATERIAL

Wolfgang Keller and Arnulf Hoffmann, Pretzfeld, Germany, assignors to Siemens-Schuckertwerke Aktiengesellschaft, Erlangen, Germany, a corporation of Germany

Filed Sept. 9, 1960, Ser. No. 55,051
Claims priority, application Germany Sept. 11, 1959
9 Claims. (Cl. 219—10.77)

Our invention relates to a method and apparatus for the crucible-free zone melting of semiconductor material according to which a melting zone between the two ends of a semiconductor rod is passed longitudinally along the rod while being heated by an inductance coil which surrounds the rod and is energized from a high-frequency generator. In such a method the magnitude of the high-frequency heating current passing from the generator through the coil is dependent upon the rod diameter. It has been proposed to sense this current magnitude as a controlling criterion for varying the spacing between the two holders in which the respective rod ends are held, thus causing relative motion between the two holders toward or away from each other until the current flowing through the heating coil, upon departure from a datum value, again assumes that value.

As will be more fully explained below, difficulties and irregularities are encountered during the starting phase of such a zone melting operation; and it is an object of our invention to obviate such shortcomings.

To this end, and in accordance with our invention, we initiate the first melting of the axially narrow zone in the semiconductor rod by varying the frequency of the generator current passing through the inductance heater coil, and as the initial liquefaction of the zone progresses, we continue this frequency variation so as to maintain the current magnitude substantially constant at the datum value until the zone is entirely melted through. We then change the frequency of the generator current by shifting it to a fixed value that corresponds to the rod diameter and the datum value of current magnitude, and vary the axial spacing between the rod ends so as to maintain the current at said datum magnitude while the melting zone is being passed along the rod.

Further objects and features of the method as well as of apparatus according to the invention will be apparent from, and will be mentioned in, the following with reference to the embodiment of equipment illustrated by way of example on the accompanying drawings, in which:

FIG. 1 is a schematic diagram of the equipment.

FIG. 2 is an explanatory graph of a frequency control program as carried out by the apparatus.

FIG. 3 is an electric circuit diagram of details; and

FIG. 4 is an explanatory graph of a typical current-resonance curve as occurring in such apparatus.

It will be understood that the zone melting is preferably carried out within a bell or other recipient under high vacuum and that accordingly the semiconductor rod with its holders, as well as the inductance heater coil and, if desired also with the devices for moving the holders or coil, are mounted within the recipient, whereas the other components of the apparatus together with the recipient may be mounted on a common support in order to constitute a single and preferably transportable unit. Such design features, not essential to the invention proper and known as such for apparatus of this general type, are not illustrated on the drawing.

As apparent from FIG. 1, the semiconductor rod **2**, for example of silicon, germanium, indium arsenide, indium antimonide, gallium arsenide or other semiconducting substance, has its two ends clamped in respective holders **3** and **4** so as to extend vertically. The rod **2** is surrounded by an axially flat inductance coil **5** by means of which a melting zone **6** is to be produced in the rod. A capacitor **7** connected parallel to the heater coil **5** compensates the reactive current. The coil **5** and the capacitor **7** form an oscillatory circuit (tank circuit), hereinafter called "heating circuit," which is energized at terminals **8** from a high-frequency generator **9**.

The generator **9** preferably operates on a flank of the resonance curve of the heating circuit. Such a resonance curve is typified by the one shown in FIG. 4 showing frequency values f of the abscissa and current values I on the ordinate. The resonance frequency of the heating circuit is indicated at f_r. The normal operating frequency of the generator is preferably chosen as exemplified by the frequency value f_g which is located on a slanting flank portion of the resonance curve so that a change in operating frequency from the value f_g in one or the other direction will result in increasing or decreasing the current intensity I as required for the method more fully described hereinafter.

The generator **9** is energized from a current supply line at terminals **10**. If this supply line furnishes alternating current, a rectifier as shown at **10***a* in FIG. 3 is inserted. According to FIG. 1 the generator **9** is provided with a rotary knob **11** by means of which the frequency of the generator current can be adjusted at will from the outside. Such change in frequency may be effected by varying the inductance or capacitance of the oscillator which forms part of the high-frequency generator. Thus according to FIG. 3, the generator may comprise an electronic tube T and an oscillator circuit comprising a capacitor C and an inductance coil which forms the primary winding of a transformer TR coupling the generator with the heating circuit.

A resistor **13** is serially connected in the plate circuit so as to develop a voltage drop proportional to the magnitude of the generator current drawn by the heating circuit. A voltmeter **12** is shown connected across resistor **13** for indicating the magnitude of this current. The IR-drop voltage of resistor **13** acts upon the winding of a relay **15** in differential relation to adjustable datum voltage from a source **14**. The relay **15** is polarized. Its contacts **16** are in an inactive position of rest when the voltage drop of resistor **13** is substantially equal to the datum voltage from source **14**, while the contacts **16** close either one or the other of two stationary contacts (FIG. 1) when the voltage drop and hence the generator current magnitude depart in one or the other direction from the datum value.

The plate current of the generator tube T depends upon the power consumed in the heating circuit and thus is dependent upon the diameter of the semiconductor rod **2** because a change in diameter results in a corresponding change in the degree of coupling between coil **5** and zone **6**. Such a current change is indicated by instrument **12** and causes a corresponding change in voltage drop of resistor **13**. This resistor is to be so adjusted that a plate current as occurring with the diameter of the particular semiconductor rod being processed causes the voltage drop at resistor **13** to be equal to the datum value determined by the voltage from source **14**. Under such operating conditions, the polarized relay **15** is inactive.

When the rod diameter varies, the current of the generator changes accordingly and thus also the voltage drop (pilot value) of resistor **13**. This causes the relay **15** to actuate its contacts **18** by placing them in one or the other closed position depending upon the direction in which the pilot value departs from the datum value. In either case, the contacts **16** connect a direct-voltage source **17** with the contacts **18** of another relay **23**. The relay **23** then operates to connect the direct voltage from source

17 at contacts 18 either to a reversible control motor 19 or to a relay 20. In the first case, the motor 19 turns in one or the other direction depending upon whether the contacts 16 of relay 15 have shifted to the upper or lower active position. The motor 19 then drives a pinion 21 meshing with a rack 22 which displaces the holder 4 toward or away from the holder 3. By suitably poling the relay 15 and the motor 19, the motor 19 will always run in the proper direction, namely so that when the rod diameter is too large, the holder 4 is pulled down, whereas when the rod diameter is too small the holder 4 is moved up, thus either effecting an axial expansion and thinning or an axial compression and thickening of the melting zone. As a result, the rod diameter is always maintained substantially at the proper value.

In the other case of operation, namely when the contacts 18 of relay 23 are in the illustrated positions, the voltage from source 17 is connected to the relay 20 which controls the generator 9 so as to adjust its frequency in the desired sense. In this manner the frequency is increased when the plate current is too small, thus increasing the current, and the frequency is decreased when the plate current is too large, thus effecting a current regulation.

The relay 23 is controlled by a sensing device 24 which is firmly connected with the upper rod holder 3 and which responds to melting-through of the zone. The sensing device 24, shown in FIG. 1 simply as a normally open switch, is preferably given a design as illustrated and described in U.S. Patent No. 3,009,973 (copending application Serial No. 831,289). Accordingly, the device 24 is essentially a torque-producing device which acts upon the upper rod holder 3 but cannot cause rotation of that holder as long as the entire rod is solid. However, as soon as the zone 6, at the beginning of the zone- melting operation, is melted through, the torque is effective to turn the upper rod holder 3 sufficiently for closing the electric contact which is available for actuating a signalling or controlling device. The sensing device 24 is energized from a current supply line at terminals 25 and, when closing its contact, energizes the relay 26 whose contacts 27 then connect the current supply line to a rotating device 28 with whose aid the lower rod holder 4 is placed in rotation about the rod axis for the purpose of securing an axially symmetrical growth of the semiconductor rod during the zone melting performance. Simultaneously, the contacts 27 when closing, energize a motor 29 which, by means of a pinion 30 and a rack 31, displaces the support 32 of the heater coil 5 along the rod axis, thus shifting the molten zone longitudinally of the rod. A switch RF is shown for reversing the motor 29 to perform the return stroke of coil motion.

It has been found that for a given heating circuit, a constant coupling between heating circuit and high frequency generator and a given travelling speed of the melting zone, there is a definite relation between generator frequency, generator current and rod diameter. Consequently, by adjusting given values of frequency and generator output current, a predetermined, reproducible rod diameter can be constrainedly obtained. This relation, however, applies only to stable-state conditions at which the melting zone travels at constant speed along the rod, whereas the starting-up conditions involve irregularities and difficulties. At the commencement of the zone melting performance it is necessary to first melt the zone entirely through the cross section of the rod. For this purpose the generator current is regulated to a constant magnitude by correspondingly controlling the frequency. This can be done, for example, by varying the capacitance of the oscillatory circuit in the generator by means of a control motor. This motor is shown at 11M in FIG. 3. The control of motor 11M is effected by the above-mentioned relay 20 which is also the polarized type so that its contacts are normally inactive. When the contacts close they connect the motor 11M to the direct-current source 35 with the polarity required to make the motor run in the proper direction depending upon whether the contacts 18 of relay 23 are in one or the other of their active positions. Thus the actuation and poling of motor 11M is dependent upon the operation of relay 15 and thus upon the departure of the generator load current from the datum value. When the zone 6 in rod 2, at the beginning of the zone-melting performance, is melted through, the sensing device 24 causes the relay 23 to switch from relay 20 to motor 19, thus discontinuing the above-mentioned current regulation by frequency variation. In lieu thereof, the generator current is now regulated with the aid of motor 19 by moving the holder 4 toward or away from the holder 3.

During the transition from constant current regulation by change in frequency to current regulation by varying the spacing between the rod holders, the generator frequency must be gradually adjusted to the value that corresponds to the diameter of the rod and to the datum magnitude of generator current. The need for such gradual adjustment will appear from the following.

When the rod is first melted through, the initial melting zone is initially relatively short in the axial direction. Accordingly, the frequency of the heating circuit and thus also the generator frequency is initially higher than during the subsequent travel of a stable melting zone. Therefore, if the generator frequency were to remain on the value which it happened to assume at the moment when the rod melts through, the subsequent current regulation by varying the spacing between the rod holders would cause thickening of the melting zone and hence also of the semiconductor rod when finished. In practice, with all types of crucible-free zone melting (purifying by zone melting, zone levelling for uniformly distributing impurity substance, growing a mono-crystal with the aid of a seed crystal fused to a polycrystalline rod), a repeated passage of the melting zone through the length of the rod is necessary. Hence, the just-mentioned thickening of the zone may result in considerably increasing the rod diameter. However, if, at the melting-through moment, the generator frequency is shifted to the value that corresponds to the given rod diameter and the datum magnitude of generator current for a travelling stable melting zone, then the generator load current is first reduced because the melting zone is still axially shorter than in its subsequent, stable condition. The consequence is an increase in spacing between the rod holders and thus an axial lengthening of the melting zone whereby the generator current is reduced to its datum value. Thus this has the effect that the thickened bulge at the lower end of the melting zone is eliminated. Due to the rotation of the lower rod holder about the rod axis and the relative motion between heating coil and rod, the melting zone is then enlarged to the stable value. The rod diameter remains constant even with a relatively great number of zone passes.

It is preferable to prevent an abrupt change from the frequency value reached by the generator at the melting-through moment to the frequency value that corresponds to the travelling stable melting zone, but to effect the transistion in accordance with a given time-delay program. Otherwise, the melting zone may again freeze because the heating power for the enlarged melting zone, due to reduced frequency, cannot be supplied instantaneously. As described above, the frequency of the generator 9 can be set to a given value by means of the knob 11, this value corresponding to the diameter of the rod to be processed and also to the desired generator load current. The indicating scale for knob 11 is preferably calibrated in terms of diameter. Now, in order to achieve the time delayed transition from one to the other frequency, the relay 23 (FIG. 1) is preferably provided with an auxiliary contact 18*a*. During the initial stage of operation, i.e. when the motor 19 is at rest and the relay 20 controls the motor 11M to vary the frequency of the

generator toward maintaining the heating current at the datum value, the contact 18*a* is open. At the moment when the sensing device 24 causes the relay 23 to de-energize relay 20 and to energize the motor 19, the auxiliary contact 18*a* closes. This causes the motor 11M to return the frequency of the generator back to the datum value pre-set by means of knob 11.

Such delayed return of the frequency to the datum value at the beginning of the normal zone melting performance under control by the motor 19 may be effected, for example, by the following components. According to FIG. 3 the motor 11M is connected with a contact disc 11*b* which is shown to have an insulating portion 11*c*. The contact disc cooperates with a switch contact on a rotatably adjustable arm 11*a*. When the knob 11 (FIG. 1) is set to the desired frequency, the arm 11*a* (FIG. 3) firmly connected or geared with the knob 11, is set to a corresponding angular position. When, during the operation of the equipment described above, the switch contacts 18 disconnect the relay 20, the contact 18*a* energizes the relay 11R which controls the motor 11M to run in the frequency-lowering direction. As soon as the portion 11*c* of disc 11*b* reaches the switch contact on the stationary arm 11*a*, the relay 11R drops out and the capacitor C is now set in accordance with the proper operating frequency. This transitional operation corresponds to the portion of the frequency curve between the moments t_2 and t_3 in FIG. 2, more fully explained below.

It will be understood that the device 11*a*, 11*b*, 11*c* is essentially an adjustable limit switch and may be substituted by a cam-controlled switch or other conventional limit devices available for such purposes.

It is preferable to use only the upward travel of the melting zone for the zone melting proper, because the zone has greater stability during upward travel. For that reason, the heater coil 5, during downward travel, is preferably supplied with reduced heating power so that the zone is no longer liquid but is kept in incandescent condition. When the heater coil 5 arrives at the lower end of the rod 2, the melting zone is again produced simply by increasing the heating power without requiring such auxiliary heating means as needed for initially melting a cool semiconductor rod. The reversal of the coil travelling direction by means of switch RS is preferably coupled with the simultaneous switching of the generator power output from a higher to a lower current value, and this switching can be effected automatically by conventional limit contacts which are mounted in the vicinity of the rod holders 2, 3 and are acted upon by the travelling coil support 32. The reduction in heating current may be effected by inserting a resistor between generator and coil or by greatly reducing the generator frequency.

FIG. 2 shows a curve of the generator frequency f_g versus time t, commencing at the moment at which the glowing zone has arrived at the lower end of the rod. Slight frequency fluctuations due to the above-described regulating operations are neglected. The heater coil may be operated at a frequency of about 1,000 to about 10,000 kilocycles per second. For maintaining the generator current at the datum value, the frequency must at first increase rather steadily. At the moment t_1 the rod is melted through and the frequency commences to decline. At the moment t_2, and hence with some delay, the sensing device 24 controls the relay 23 and thus discontinues the current regulation by frequency variation, and the frequency is now shifted to the datum value f_0, for example 4,000 kc. p.s., which is reached at the moment t_3. The progressive and time-delayed decline in frequency between moments t_2 and t_3 is controlled by the auxiliary motor 11M in the manner described. The frequency value f_0 is maintained during the entire subsequent processing period in which the melting zone passes longitudinally through the rod. The period of time delay between t_1 and t_2 may amount to about 3 minutes, whereas the interval from t_2 to t_3 is much shorter, for example about 15 seconds when operating at a frequency of about 4,000 kc. p.s.

In summary, the zone-melting method according to the invention performable by the illustrated apparatus is as follows. The initial melting of a narrow zone in the semi-conductor rod is effected by energizing the heating circuit 5, 7 with current of constant magnitude. The regulation for constancy of current is effected by correspondingly varying the frequency of the generator 9. The melting-through of the semiconductor rod is responded to by the sensing device 24, which, by delayed action, causes the relay 23 to switch off the frequency control and to initiate the regulation of current by varying the spacing between the rod holders. At the same time, the relative motion between heater coil and semiconductor rod, as well as the rotational motion of the lower rod holder, are switched on. The frequency now progressively passes to a different value which corresponds to the adjusted datum value of generator current for the given rod diameter, and the frequency value then remains constant (f_0 in FIG. 2). After the melting zone has passed through the entire semiconductor rod and has reached the uppermost limit position or limit switch, all regulating operations as well as the rotation of the semiconductor rod are switched off, and the travelling motion of the heating coil is reversed. Now the coil is energized by reduced heating power, just sufficient to maintain a glowing zone in the rod, and the heating coil then travels toward the lower limit position, whereafter the cycle of operation is continued the desired number of times.

It will be obvious to those skilled in the art, upon a study of this disclosure, that our invention is amenable to a variety of modifications and may be given specific embodiments other than particularly illustrated and described herein, and that the components and circuitry of the apparatus according to the invention used for performing the method may likewise be modified in various ways, without departing from the essential features of our invention and within the scope of the claims annexed hereto.

We claim:

1. In a method of zone melting a semiconductor rod, in which the rod is vertically supported at both ends and a molten zone is formed in the rod by a surrounding inductive heater coil energized by current from a high-frequency generator and the zone is caused to move lengthwise of the rod, the improvement comprising the steps of initiating the melting of the zone near the lower end of the rod by varying the frequency of the generator current as the initial liquefaction of the zone progresses so as to maintain said current at a desired constant magnitude until the zone is melted through, thereafter shifting the frequency of the generator current to a value corresponding to the rod diameter and the desired current magnitude, and simultaneously varying the axial spacing between the rod ends to maintain the current at said desired magnitude while moving the molten zone upwardly along the rod to the vicinity of the upper end, then reducing the current magnitude so as to maintain an incandescent solid zone in the rod while returning the zone back to the lower rod end, and repeating the above-defined steps.

2. Apparatus for zone melting a semiconductor rod, comprising two coaxially spaced rod-end holders defining a rod axis, an axially narrow inductance heater coil surrounding said axis for melting a zone of the rod, a mechanism for producing relative axial motion between said coil and said holders, an alternating-current generator of adjustable frequency electrically connected with said coil for passing heating current therethrough, one of said holders being axially displaceable relative to the other, current sensing means connected with said generator and responsive to the magnitude of said heating current, a first control device connected with said generator for varying said frequency to control said current in dependence

upon the current magnitude sensed by said sensing means, a second control device connected with said displaceable holder for varying its spacing from the other holder to control said current in dependence upon said sensed current magnitude, said two control devices being actuable alternatively, whereby the apparatus, during initial melting of the zone, permits maintaining said current magnitude substantially constant by frequency variation under control by said first control device and, after melting-through of the zone, maintaining said constant current magnitude by varying said spacing under control by said second device.

3. Apparatus for zone melting a semiconductor rod, comprising two coaxially spaced rod-end holders defining a rod axis, an axially narrow inductance heater coil surrounding said axis for melting a zone of the rod, mechanism for producing relative axial motion between said coil and said holders, an alternating-current generator of adjustable frequency electrically connected with said coil for passing heating current therethrough, one of said holders being axially displaceable relative to the other for varying the diameter of the melting zone, a first control device connected with said generator for varying said frequency to control said current in dependence upon the magnitude sensed by said sensing means, a second control device connected with said displaceable holder for varying its spacing from the other holder to control said current in dependence upon said sensed current magnitude, current sensing means connected with said generator and responsive to departure of said current magnitude from a datum value, said current sensing means being connected to said first and second control devices for controlling them to regulate said current magnitude to remain substantially constant at said datum value, and selector means connected with said two control devices for placing either one of them in active condition, whereby the apparatus, during initial melting of the zone, permits activating said first control device for current regulation by frequency variation and, after melting-through of the zone, activating said second control device for current regulation by varying the spacing between said holders.

4. Zone-melting apparatus according to claim 3, comprising capacitance means connected with said coil to form a resonant circuit together therewith, said generator having a range of frequency variation coincident with a flank portion of the current resonance characteristic of said resonant circuit.

5. Zone-melting apparatus according to claim 3, comprising a sensing device responsive to melting-through of the zone and controllingly connected with said selector means for switching it from said first control device to said second control device when the zone is melted through.

6. In apparatus according to claim 5, said sensing device comprising torque means mechanically connected with the other holder imparting rotary motion thereto when the zone is melted through, electric contact means actuable by said rotary motion, and circuit means connecting said contact means with said selector means for controlling the latter by actuation of said contact means.

7. In apparatus according to claim 5, said sensing device being also connected with said mechanism for controlling it to start relative motion of said coil in response to melting-through of the zone.

8. Zone-melting apparatus according to claim 5, comprising a rotating device connected with said one holder for rotating it about said axis during zone melting, said sensing device being also connected with said rotating device for initiating its operation when the zone is melted through.

9. In a method of zone melting a semiconductor rod, in which the rod is vertically supported at both ends and a molten zone is formed in the rod by a surrounding inductive heater coil energized by current from a high-frequency generator and the zone is caused to move lengthwise of the rod, the improvement comprising the steps of initiating the melting of the zone near the one end of the rod by varying the frequency of the generator current as the initial liquefaction of the zone progresses so as to maintain said current at a desired constant magnitude until the zone is melted through, thereafter shifting the frequency of the generator current to a value corresponding to the rod diameter and the desired current magnitude, and simultaneously varying the axial spacing between the rod ends to maintain the current at said desired magnitude while moving the molten zone along the rod to the vicinity of the other end, then reducing the current magnitude so as to maintain an incandescent solid zone in the rod while returning the zone back to the said one rod end, and repeating the above-defined steps.

References Cited in the file of this patent

UNITED STATES PATENTS

2,743,199	Hull	Apr. 24, 1956
2,868,902	Mika et al.	Jan. 13, 1959
2,913,561	Rummel et al.	Nov. 17, 1959