

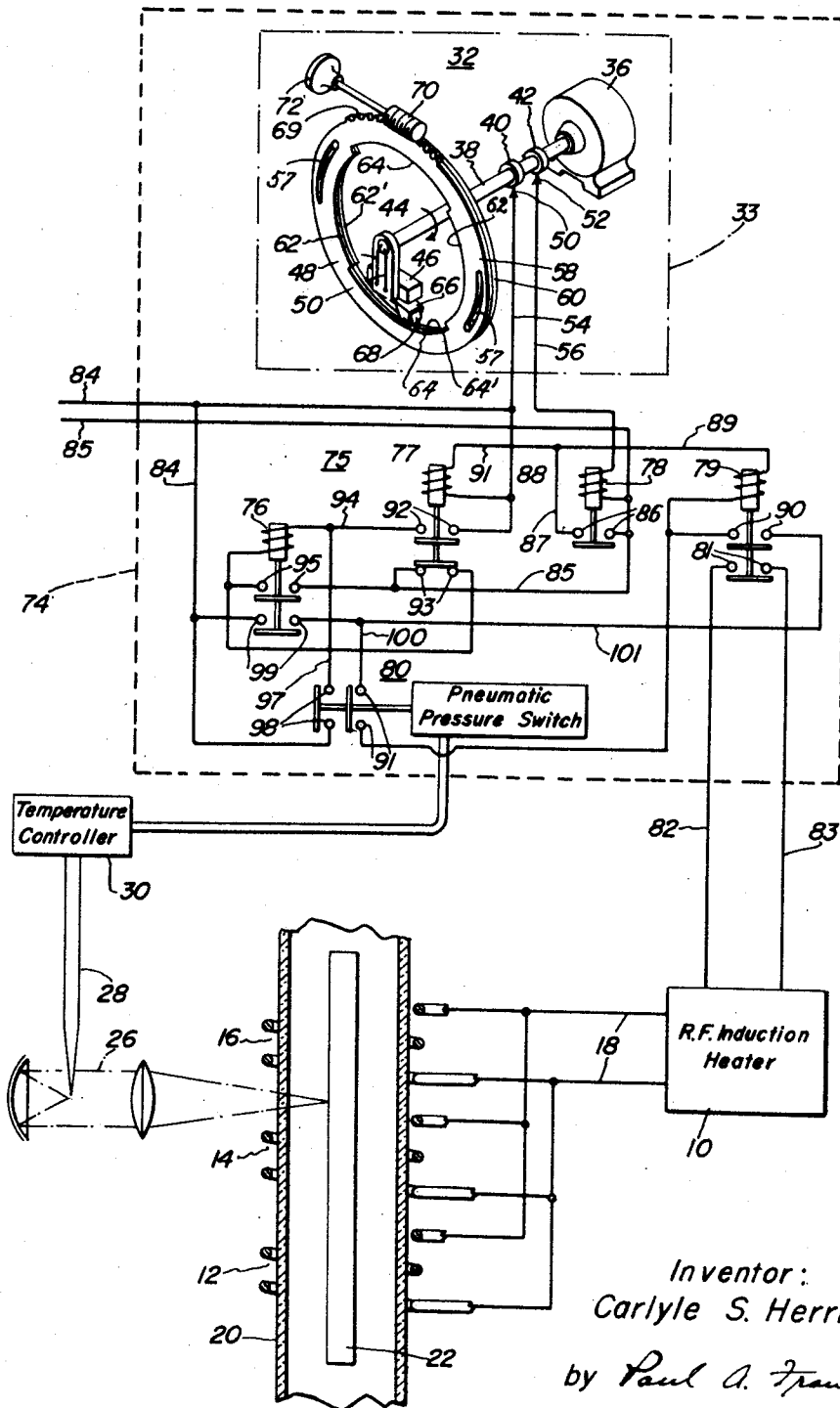
Dec. 8, 1959

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2,916,593

INDUCTION HEATING APPARATUS AND ITS USE IN SILICON PRODUCTION

Filed July 25, 1958



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2,916,593

INDUCTION HEATING APPARATUS AND ITS USE IN SILICON PRODUCTION

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Application July 25, 1958, Serial No. 751,091

10 Claims. (Cl. 219—10.61)

This invention relates to the temperature control of a silicon production method, and more particularly, to a device and related circuitry for the maintenance of the temperature of a silicon decomposition work piece within desired limits.

While several methods of producing silicon are available, this invention is directed to that method whereby silicon is converted to silicon tetraiodide, distilled, and the vapor thermally decomposed to a purer form of silicon. The silicon tetraiodide employed in the present invention may be prepared from silicon and iodine. Silicon powder, in particle form having a purity of from about 95% to a silicon having an impurity of only a few parts per million, is placed in a furnace and heated to a temperature of about 600° to 800° C. Iodine vapor, which is generated by heating iodine at atmospheric pressure to a temperature of about 180° C., the boiling point of iodine, is then passed over the silicon in a nitrogen atmosphere. The resulting silicon tetraiodide and nitrogen are carried through a distillation column and condenser to a collection flask. The purified silicon tetraiodide from the distillation column is then decomposed into silicon by heating a vessel containing the tetraiodide to cause vaporization of the material, and then by passing these vapors in contact with a heated surface composed of a material which will not contaminate the silicon and which is maintained at a temperature high enough to cause thermal decomposition of the tetraiodide. The heated surface or the work piece employed for decomposing this silicon tetraiodide is generally formed of tantalum, or quartz, or, in this invention, of substantially pure silicon heated to a temperature in the range of 900°–1000° C.

One method of heating the silicon work piece is resistance heating, but this method has the very pronounced disadvantage of requiring contaminating leads or contacts to be affixed or otherwise joined to the work piece. This disadvantage may be overcome by employing induction heating of the work piece. Where, however, the initial work piece is of substantially pure silicon, "du Pont Hyperpure Silicon" zone refined to 100 to 200 ohm-centimeter resistivity, it presents to the induction heater an electrical load which is initially of quite high impedance, and which then decreases rapidly as the temperature increases. It would therefore seem desirable in this set of circumstances to employ a series tank coil work coil or constant current arrangement on the induction heater because it matches the high impedance of the silicon, which characterizes the load at lower temperatures, and thus provides a self-balancing feature.

The operation of this system is generally stable because an increase in temperature of the work piece decreases impedance and reduces the impedance match between the load and the heater. The efficiency of the power transfer is reduced and tends to limit further temperature increase. A temperature decrease of the work piece increases the impedance, improves the impedance match, and increases the power transfer efficiency, which in turn tends to limit further temperature decrease. These principles are realized with commercially available induction heaters in the temperature range of 700° C. to 1420° C., a range which includes the range which is important

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to the decomposition reaction of this invention. Among the faults with the system just described is that when silicon tetraiodide is decomposed in a vessel that is long compared with the mean free path, a tapered deposit is obtained at a constant temperature. Therefore, if a proper temperature is maintained, a uniformly thick deposit may be had, but adjusting the temperature gradient by varying the turn-to-turn spacing in a series coil is not only awkward but hazardous. A further fault of the series system is that the "du Pont silicon" after being pre-melted and zone-refined still contains carbon, hydrogen and oxygen to the approximate extent of 1 part in 10,000. Accordingly, when the silicon is heated in a vacuum, the carbon and hydrogen gradually volatilize over a period of several days. The electric field strength of the series system is high enough to cause these volatile gases to ionize and the ionized gas acts as an electromagnetic shield around the work piece to affect the impedance match between the work piece and the heater in a manner which effectively prevents good transfer of power to the silicon work piece.

Accordingly, it is an object of this invention to provide improved induction heating for the work piece in the process of thermal decomposition of silicon.

It is a further object of this invention to overcome the problems of evolved gases when using an induction heater for the work piece in the process of thermal decomposition of silicon.

It is another object of this invention to provide a parallel work coil induction heater for the work piece in the process of thermal decomposition of silicon.

It is yet another object of this invention to provide novel means for maintaining a given temperature gradient along the work piece in the thermal decomposition of silicon.

It is still another object of this invention to provide a cycle timer for use with an induction heater in the thermal decomposition of silicon.

This invention will be better understood when taken in connection with the following description and the drawings, in which:

The single figure is a schematic representation of the apparatus utilized in this invention as applied to the thermal decomposition of silicon.

Briefly described, this invention in one form includes the heating of a silicon rod or cylinder by a parallel coil induction heater in such a manner that, at the desired temperature level of the work piece, every part of the work piece is rising in temperature, and also, that the work piece is allowed to partially cool within the desired temperature range in an equalized manner. The induction heater of this invention is employed at full or partial power in an on-off manner with a particular time cycle and cycling device chosen such that the temperature is matched to the work piece.

In Fig. 1, induction heater 10 includes a plurality of coils 12, 14 and 16 connected in parallel relationship from a common connector 18. While only 3 coils are shown, the particular number may be varied to match the desired length of work piece and in one form of this invention, 7 such coils were employed. Coils 12, 14 and 16 are illustrated as being wound about or encircling a cylindrical vessel 20 in which there is suitably positioned the length of silicon rod or work piece 22. Vessel 20 may be of various materials but, for the purposes of this invention, quartz is used because it is inert chemically toward tetraiodide and iodine, and because it is stable at the high operating temperatures. The induction heater 10 is connected to a suitable source of power (not shown) to derive proper current and voltage to heat the silicon rod to a temperature of approximately 800°–1000° C.

In order to measure the temperature rise for further temperature control, a temperature device, shown broadly as 24, is employed. Temperature device 24 includes a temperature sensing element 26, of the well known thermocouple type 28, and a temperature controller 30. Each of the components 24, 26, and 28, 30 are well known in the art and commercially available. More specifically, component 26 is referred to as a radiation pyrometer and essentially comprises a lens and mirror arrangement to focus the radiation from the heated silicon rod 22 upon the thermocouple 28 junction. The temperature controller 30 as employed in this invention is a commercially available "Foxboro EMF Dynalog Recorder-Pneumatic Controller." Briefly described, controller 30 amplifies the small current signal from the thermocouple 28 to provide a stronger signal to actuate various devices such as mechanical or electrical relays or in this invention a pneumatic signal. Controller 30 also contains suitable adjusting and comparing arrangements by which the controller may maintain a given temperature by actuating the heater 10. It is to be understood, however, that the temperature device 24 may be one of many such devices applicable to provide a desired signal output from a given input. When the temperature of the silicon rod is required to be raised or lowered, the temperature controller provides a signal to operate the induction heater 10.

The use of a parallel or constant voltage tank coil work coil arrangement provides a lower electric field strength in comparison with the series constant current tank coil work coil. The ionizing potential of the gases is greater than the field strength so that the use of a parallel induction heater is possible. This arrangement also permits a convenient control of the temperature gradient but does not possess the self-stabilizing features described for the series tank coil work coil constant current arrangement, but generally the reverse. For example, with the parallel work coil heating a semiconductor, an increase in work temperature lowers the impedance, which is a better match for the induction heater, and increases the efficiency of power transfer. Consequently, the temperature will tend to continue to increase and a runaway condition is indicated. In practice, with the silicon workpiece heated by a parallel work coil, the heat loss relation is sufficient to keep the temperature from rising out of control. In the other direction, a decrease in work temperature causes an increase in impedance of the load and a poor match with the heater, which reduces the efficiency of power transfer. Consequently, once the temperature starts to decrease, it will tend to continue to do so. With the silicon workpiece heated by a parallel work coil, this effect becomes important below 1100° C. For example, if the silicon rod is heated to 1400° C. initially, the temperature may be reduced by reducing the power input to the heater and maintained at any desired temperature below about 1400° C. until the temperature of the rod reduces to approximately 1100° C. Below this temperature, the impedance mismatch and loss of power transfer efficiency are great enough so that the heater does not maintain the temperature and the workpiece cools to room temperature.

These disadvantages are overcome by the arrangement of Fig. 1 as illustrated and described, since the temperature controller 30 operates the induction heater 10 in an on-off manner at full power. An exemplary application of Fig. 1 utilizes a silicon rod 22, $\frac{5}{8}$ inch in diameter, 14 inches in length, and 200 ohm-centimeter resistivity, while quartz tube 20 has an internal diameter of 2 inches with the parallel induction coils on the outside thereof. Induction heater 10 is a commercially available 25 kilowatt R.F. model having an output k.v.a., sufficient for this size tube and rod. The heating process may be facilitated in starting by having a thin ring of molybdenum around the bottom of the rod 22 as a starter. The

temperature sensor is a focussed thermocouple type of temperature sensing element actuated by a "Foxboro E.M.F. Dynalog Recorder-Pneumatic Controller." The temperature signal from the temperature controller 30 passes through a suitable cycle timer 32 before actuating the induction heater.

Cycle timer 32 of this invention needs be necessarily more than a mere off-on type of switch, since several features are controlled thereby. First, the cycle switch must be adjustable over a predetermined range in order to provide various arrangements of cycles, frequencies, or length of time duration of the individual cycle. Second, the cycle switch must incorporate a protective circuit to avoid cumulative effects of electrical transients which may be caused by rapid variations in temperature recordings from the temperature unit and the relay circuit operating the induction heater. Thus, the timer and the incorporated circuit prevent a change in condition in that the induction heater is prevented from being energized in an on-off-on condition in rapid order.

In Fig. 1 there is shown in perspective, circumscribed by the dash-dot line 33, the cycle timer 32 as employed in this invention. Timer 32 comprises a motor or other turning device 36 which rotates a projecting shaft 38. Projecting shaft 38 has suitably mounted thereon a pair of current conducting slip rings 40 and 42, for purposes to be hereafter described, and an arm member 44. Arm 44 has positioned on the free end thereof a suitable electrical switching device 46 to establish or interrupt an electrical circuit. In the preferred form of this invention, the switching device 46 is a well known form of microswitch which includes a pair of electrical leads 48 and 50 connected to the slip rings 40 and 42. This circuit is continued by a pair of brush type current collectors 50 and 52 and leads 54 and 56 whose connections will be described hereafter.

Rotation of shaft 38 rotates arm 44 and switch 46, clockwise in viewing Fig. 1, to describe a circle whose periphery is defined by a pair of relatively flat annular cam members 58 and 60. Cam members 58 and 60 have inner camming surfaces 62 and 64 and 62' and 64' which present a series of circumferential alternate plateaus and depressions such as 62 and 64, respectively. In order to actuate the microswitch 46 by means of the cam surfaces 62, 62', 64 and 64', the microswitch 46 includes a switch arm 66 on the end of which is a cylindrical roller 68. Roller 68 engages the cam surfaces of each cam member 58 and 60 and it can thus be seen that as the arm 44 rotates, the roller 68 rolls over the cam surfaces 62, 62', 64, 64' to actuate switch 46 through suitable mechanism therein.

In the preferred form of this invention, the shaft 38 is rotated by means of a 6 r.p.m. electric motor 36 and each cam member 58 and 60 includes, alternately, 2 plateaus and 2 depressions. The arrangement provides a sequential series of 1 revolution of the motor in 10 seconds with the switch 46 being closed and opened alternately for $2\frac{1}{2}$ -second intervals.

The $2\frac{1}{2}$ -second interval may be varied to match different processes by an adjustability feature which permits adjustment without interruption of the process. This adjustment is provided by mounting cam members 58 and 60 such that one, for example 58, is rotatable with respect to the other by suitable means such as the pin slot connection 57. Briefly described, cam member 58 includes a threaded portion 59 engaged by a worm 70. Rotation of knob 72 rotates worm 70 to, in turn, rotate cam member 58. Rotation of cam member 58 moves its cam surfaces 62 and 64 out of coincidence with respect to cam surfaces 62' and 64' on cam member 60, or in other words, a plateau of cam member 58 progressively overlies a depression in cam member 60. As mentioned before, the switch roller 68 engages the cam surfaces of both cam members so that the

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adjustment essentially decreases the circumferential length of a depression, maintaining the switch in a given position for a longer or shorter cycle, as the case may be. The adjustable feature of the cycle timer 32 is quite applicable to various means of adjustment such as manual, electrical, mechanical, or combinations thereof, or may be of the automatic variety to be actuated by a suitable signal from the temperature controller 30.

The cycle timer 32 in this invention is included within dash line 74 which outlines an anti-coincident circuit 75. The cycle timer 32 provides the proper electrical cycles to anti-coincidence circuit 75, which in turn acts as a gate through which the on-off signal from the cycle timer 32 must pass on the way to the induction heater 10. The gate action is controlled by the start-stop signals from the temperature controller 30. The anti-coincidence feature prevents the gate from changing its condition, except during an "off" period of the cycle timer 32. Thus, the open gate cannot be closed while an "on" period is passing through, and a closed gate cannot be opened while an "on" period is offered for passage. The purpose of circuit 75 is to keep the induction heater 10 from being turned on-off-on in rapid order which may happen when cycle timer signals are gaited to the heater 10 by a simple pneumatic relay connected to the temperature controller output. The cumulative effects of electrical transients from such rapid operation are sufficient to cause component failure in the heater.

In Fig. 1, anti-coincidence circuit 75 includes the cycle timer 32, a plurality of electrical relays 76, 77, 78, and 79 and a pressure switch 80.

Switch 80, as employed in one form of this invention, is a pneumatic or pressure operated switch to energize the circuit for heater 10 operation from a suitable source, not shown. A supply of air under pressure controlled at temperature controller 30 is admitted to a simple diaphragm assembly or other well known actuating devices for switch 80 operation. It is understood that such a switch 80 may be one of many well known types of switches either electrically, mechanically, or hydraulically operated by a suitable signal from controller 30.

Relay 79 is a dual circuit relay employed to control a circuit within the anti-coincidence circuit 75 and also to establish or interrupt the circuit for the induction heater 10 for on-off operation. As a part of relay 79, the contacts 81 together with leads 82 and 83 comprise the induction heater 10 energizing circuit.

The particular electrical connections of the components of circuit 75 proceed with the following description, assuming that the pressure switch 80 is open, no heat is required, and micro-switch 46 is closed. A pair of leads 84 and 85 are connected to a suitable source of power, not shown, and in the preferred form of this invention, to a 110-115 volt system. Connected across the line, the line being leads 84 and 85, is the cycle timer 32 in series with the coil of relay 78. The connection is made by leads 54 and 56 of timer 32 as illustrated in Fig. 1. The motor 36 of timer 32 is continually operating so that a circuit is established and interrupted through relay 78 every 2½ seconds. Relay 78 includes a pair of normally open contacts 86 which are therefore also engaged every 2½ seconds. Since no "on" or "off" condition of the induction heater is called for, the remainder of circuit remains dormant. This is easily understood by following through the circuit established by engagement of contacts 86. Contacts 86 are connected on one side directly to line lead 85. From the other side, a lead 87 proceeds to a branch connection 88. From branch connection 88, a lead 89 connects to the coil of relay 79, one side of normally open contacts 90 and to one side of a set of open contacts 91 in the pressure switch 80.

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From the other side of branch connection 88, a lead 91 connects to the coil of relay 77, to line lead 84, thus completing a circuit through the coil of relay 77. When relay 77 is energized, the normally open contacts 92 are closed and the normally closed contacts 93 are opened. From closed contacts 92 a lead 94 connects the coil of relay 76 to one side of a set of normally open contacts 95 in relay 76 and to one side of contacts 93 which are now open. A further lead 97 connects one side of open contacts 98 in pressure switch 80 to lead 94. It can thus be seen that, when no signal for heat is forthcoming and pressure switch 80 is not energized, the relay 79 is not energized and contacts 81 in the induction heater 10 circuit remain open.

In the second case it is assumed that heat is required and the signal from the controller 30 actuates pressure switch 80 at a time when the micro-switch 46 is open.

When pressure switch 80 closes, relay 76 is energized by means of the closing of contacts 98 in pressure switch 80 and establishing a circuit as follows. From line lead 84 to one side of contacts 98 to lead 97 on the other side of contacts 98 and from lead 97 to lead 94 which is connected to the coil of relay 76 through normally closed contacts 93 to the other line lead 85. When relay 76 is energized, normally open contacts 95 and 99 are closed and contacts 95 act as holding contacts for relay 76 since a circuit is completed from the coil across the contacts 95 to line lead 85. The other components of the circuit remain dormant until the micro-switch closes.

It is recalled from the previous description of the electrical circuit connections, that the microswitch 46 and the coil of relay 78 are in series and connected across the line so that closing of the microswitch 46 energizes relay 78 to close contacts 86. The contacts 86 are also connected in series and across the line by means of leads 87, 91, through the coil of relay 77 and connecting to line lead 84 on one side, and then on the other side, directly to line lead 85. Therefore, relay 77 is energized to close contacts 92 and open contacts 93. The opening of contacts 93 does not de-energize relay 76 because of the previously described holding contacts 95 and the circuit therewith. The relay 79 is energized upon closing of pressure switch 80 to actuate the contacts 81 in the induction heater 10 circuit, the circuit being as follows. From line lead 84 across contacts 99 in the relay 76 to lead 100, across contacts 91 in pressure switch 80, through the coil of relay 79 to lead 89, to branch connection 88, and from branch connection 88 to lead 87, and across contacts 86 in relay 78 to line lead 85. The contacts 90 in relay 79 act as holding contacts for relay 79, a circuit being completed from line lead 84 across contacts 99 in relay 76 to lead 101, across contacts 90 through coil 79 to lead 89, to branch connection 88 and from thence to line lead 85 as before mentioned.

The anti-coincidence circuit 75 includes the important features of preventing a change in conditions such as rapid on-off condition of heater 10 or an immediate off condition while the cycle timer is in the "on" cycle.

The above two particular circuit conditions have been set forth for purposes of describing the circuit and its component operations, and there is included in the following table a more complete illustration of circuit and component operations for all relevant conditions.

CASE #1

Pressure switch 80 open

NO LOAD CONDITION

Time	Action
0.....	Microswitch opens—No action.
2.5 sec.....	Microswitch closes—Relay 78 closes.
5 sec.....	Microswitch opens—Relay 78 opens.
7.5 sec.....	Microswitch closes—Relay 78 closes.

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CASE #2

Pressure switch 80 closes while microswitch 46 is open

Time	Action
0-----	Microswitch opens—Pressure switch already open.
Ca. 1 sec-----	Pressure switch closes—Relay 76 closes.
2.5 sec-----	Microswitch closes—Relay 78 closes; relay 77 closes; relay 79 closes heating.
5 sec-----	Microswitch opens—Relay 78 opens; relay 79 opens; relay 77 opened.
7.5 sec-----	Microswitch closes—Relay 78 closes; relay 79 closes 77.
8 sec-----	Suppose pressure switch opens—Heating continues; relay 79 remains on.
10 sec-----	Microswitch opens—Relay 78 opens; relay 77 opens; relay 76 opens; relay 79 opens; heat is off.

CASE #3

Pressure switch 80 closes while microswitch 46 is closed

Time	Action
0-----	Microswitch open—no heating—pressure switch open.
2.5 sec-----	Microswitch closes—relay 78 closes; relay 77 closes; no heating.
3 sec-----	Assume pressure switch 80 closes; relay 76 remains open.
5 sec-----	Microswitch open—relay 78 opens; relay 77 opens.
7.5 sec-----	Microswitch closes—relay 78 closes; relay 77 closes; relay 76 closes; relay 79 closes—heating begins.
10 sec-----	Reverse of previous step.
12.5 sec-----	Microswitch closes—relay 78 closes; relay 77 closes; relay 76 closes; relay 79 closes.
15 sec-----	Microswitch opens—relay 78 opens; relay 77 opens; relay 76 opens; relay 79 opens.
16 sec-----	Assume pressure switch 80 opens. Reverts to beginning of any cycle.

With the apparatus of the present invention, the silicon rod temperature may be easily controlled to maintain temperature values in the range of 850° C. to 1100° C. with good reliability, and the use of parallel multiple coil induction heater provides good adjustability of work piece temperature gradient by varying the spacing of the heater coils.

It will be apparent to those skilled in the art that variations in the apparatus exist which have not been described, and the invention is intended to include all such modifications and variations as are embraced in the following claims.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. An induction heating apparatus to heat and maintain a prescribed temperature gradient along an article comprising in combination, means to raise the temperature of the article in accordance with an even temperature gradient, means to sense the temperature of the article, and an anti-coincidence circuit connected to the temperature sensing means and said heating means to receive on-off signals from the temperature sensing means for the heating means operation, and a predetermined time interval cycle timer in said circuit and operatively connected to said testing means to provide a control for said circuit and proper timing cycles of the heating means.

2. An induction heating apparatus to heat and maintain a prescribed temperature gradient along an article comprising in combination, an induction heater having a plurality of parallel connected coils spaced along the article, a temperature sensing device to sense the temperature of the article, an anti-coincidence circuit connected to the temperature sensing device and the induction heater to receive on-off signals from the temperature sensing device for induction heating operation, and a cycle timer in said circuit to provide a control for said circuit and proper timing cycles of induction heater operation.

3. In the processes for the thermal decomposition of silicon compounds to form silicon upon a silicon surface, an induction heating apparatus to heat and maintain a prescribed temperature gradient along the silicon surface which comprises in combination, an induction heater having a plurality of parallel connected coils spaced along

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the silicon surface, a temperature sensing device to sense the temperature of the article, and means to provide an on-off cycle operation of the induction heater, said means including a cycle timer to provide time cycles of on-off operation of the induction heater, and an anti-coincidence circuit connected to the temperature sensing device and interconnected with said induction heater and said cycle timer to receive on-off signals from the temperature sensor to provide the proper on-off signal at the proper cycle timer operation.

4. The invention as claimed in claim 3 wherein said cycle timer comprises a rotating assembly, means for establishing and interrupting an electrical circuit in said rotating assembly, a pair of coincident cam members coaxially positioned about said rotating assembly, said cam members having inner-camming surfaces, means on said rotating assembly engaging said cam surfaces for on-off circuit operation, and means to adjustably position one of said cam members with respect to the other to change the timing of said on-off circuit operation.

5. The invention as claimed in claim 3 wherein said cycle timer includes a pair of adjacent coincident annular cam members, said cam members having a series of circumferentially elongated raised and depressed inner surfaces, a rotating apparatus coaxially positioned within said cam members, an electrical switching device connected to said rotating apparatus, said switching device engaging each cam surface of each cam member for on-off operation, and means to adjustably rotate one of said cam members with respect to the other to change the timing of said on-off operation.

6. An anti-coincidence circuit to receive on-off signals from one sensing means for transmittal to an operating device which comprises in combination, an electrical switching means connected to said sensing means for circuit operation, a cycle timing means in said circuit for establishing and interrupting a circuit to said operating device at predetermined intervals of on-off operation, electrical relay means in said circuit to prevent operation of said operating device when the sensing means actuates the circuit for operation of the operating device when the cycle timer is in off operation, and electrical relay means to prevent operation of said operating device upon an "off" signal from said sensing device when said cycle timer is in "on" operation.

7. The invention as claimed in claim 6 wherein said cycle timing means comprises a rotating assembly, means for establishing and interrupting an electrical circuit in said rotating assembly, a pair of coincident cam members coaxially positioned about said rotating assembly, said cam members having inner-camming surfaces, means on said rotating assembly engaging said cam surfaces for on-off circuit operation, and means to adjustably position one of said cam members with respect to the other to change the timing of said on-off circuit operation.

8. The invention as claimed in claim 6 wherein said cycle timing means includes a pair of adjacent coincident annular cam members, said cam members having a series of circumferentially elongated raised and depressed inner surfaces, a rotating apparatus coaxially positioned within said cam members, an electrical switching device connected to said rotating apparatus, said switching device engaging each cam surface of each cam member for on-off operation, and means to adjustably rotate one of said cam members with respect to the other to change the timing of said on-off operation.

9. An on-off electrical cycle timer comprising in combination a rotating assembly, means establishing and interrupting an electrical circuit on said rotating assembly, a pair of coincident cam members positioned adjacent said rotating assembly, said cam members having camming surfaces engaging said means on said rotating assembly each for on-off circuit operation, and means to adjustably position one of said cam members with

respect to the other to change the timing of said on-off operation.

10. An electrical on-off cycle timer comprising in combination, a pair of adjacent coincident annular cam members, said cam members having a series of circumferentially elongated raised and depressed inner surfaces, a rotating apparatus coaxially positioned within said cam members, an electrical switching device connected to said rotating apparatus, said switching device engaging each cam surface of each cam member for on-off operation, 10

and means to adjustably rotate one of said cam members with respect to the other to change the timing of said on-off cycle.

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