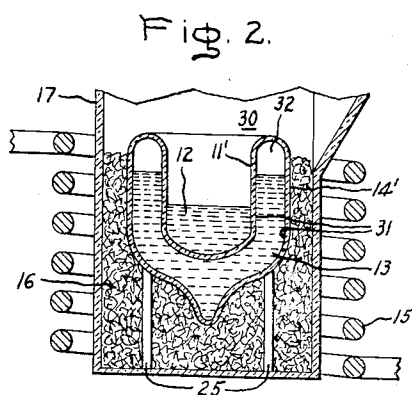
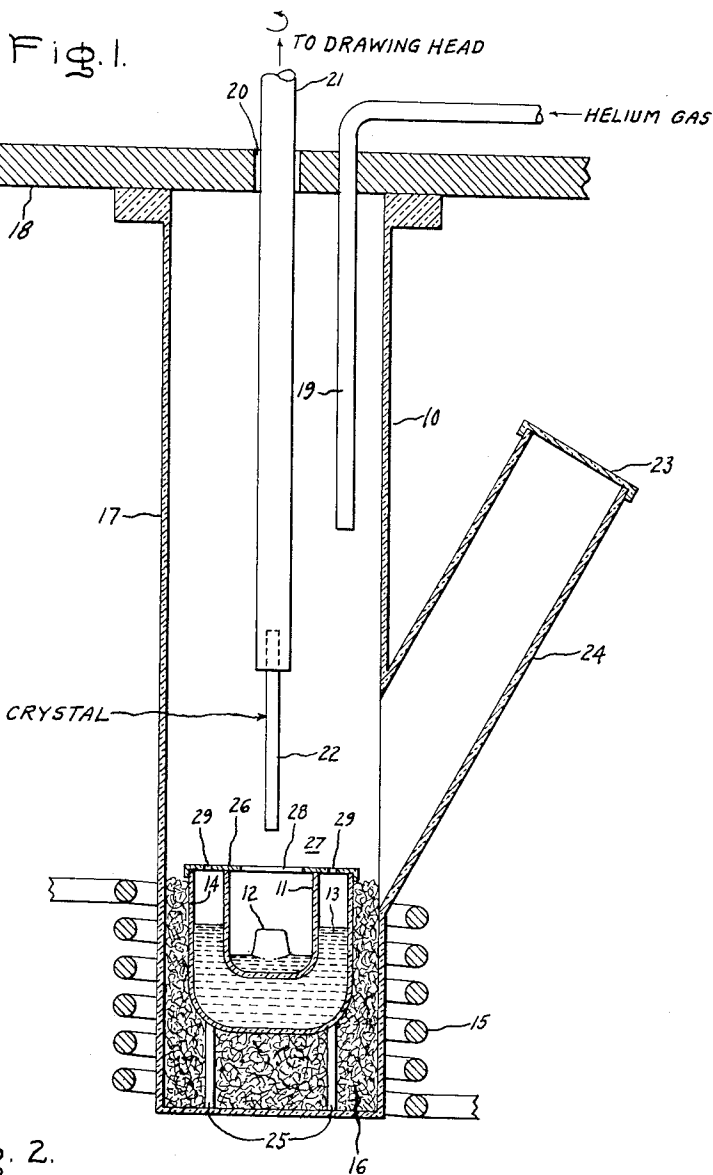


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ELECTRIC HEATING APPARATUS

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ELECTRIC HEATING APPARATUS

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Our invention relates to apparatus for heating and melting materials, and more particularly to high frequency induction apparatus for heating and melting chemically active high melting point materials which may or may not be electrically conductive. Although apparatus in accord with my invention may be used for heating materials over a very wide range of operating temperatures, its chief utility lies in heating materials to operating temperatures lying between 500° C. and 1500° C.

Conventional high frequency induction apparatus has been found in practice to have many shortcomings especially where carefully controlled heating is required, high purity of the charge is to be maintained, or high temperatures are involved. If the charge to be heated is electrically conductive, heating may be accomplished by directly coupling the high frequency field therewith but this often results in localized heating effects and creates a turbulence in a molten charge which may be objectionable for certain purposes such as when a monocrystal is desired to be grown by seed crystal withdrawal from the melt. If the charge to be heated is not electrically conductive or is only semiconductive, the high frequency field is often coupled with and inductively heats an electrically conducting crucible which then transfers its heat to a contained charge. Such apparatus has not found universal application, however, primarily because most electrically conductive crucibles are also chemically active with respect to many materials desired to be heated; especially if they are heated in contact with such materials at fairly high temperatures, for example, above 1000° C. In order to overcome this difficulty, it has become common practice to couple the high frequency field with an electrically conducting cylinder, which surrounds a non-conductive crucible containing the charge to be heated. The electrically conducting cylinder may consist of a metal, such as tungsten, having a melting point considerably above the desired maximum operating temperature and heats the crucible principally by radiation, although some conduction heating is also involved. The heat flow from the external cylinder to the charge within the crucible in such apparatus is rather inefficient at low temperatures and is often non-uniform at high temperatures, with the result that small masses of materials, especially high-melting-point materials, often cannot be successfully melted, and it is often difficult or impossible to grow monocrystals in such apparatus.

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Accordingly, one principal object of the invention is to provide high frequency induction apparatus for heating and melting materials with minimized contamination from the heating and charge-supporting apparatus.

Another object is to provide apparatus for melting, by means of a high frequency electric field, high melting point materials which are non-conductors of electricity or poor conductors of electricity.

An additional object is to provide high frequency induction apparatus which may be employed to heat electrically-conducting charges without direct coupling of the high frequency field with the charge, thereby minimizing the stirring and turbulence of the charge often resulting from the direct induction heating thereof.

A further object of the invention is to provide a high frequency induction heating method and apparatus which functions to produce highly efficient conduction heating of a charge, thus enabling complete melting of even small masses of electrically non-conducting high melting point charges.

A still further object of the invention is to provide high frequency induction apparatus which produces uniform non-turbulent heating of a charge, thus enabling the growth of monocrystals by seed crystal withdrawal from a molten charge within the apparatus.

In general, in accord with the invention, a non-electrically conducting refractory crucible chemically inactive with respect to the charge to be melted is supported in contact with an electrically conducting metal having a melting point substantially lower than that of the destined operating temperature to which the charge is to be raised, and means are provided for coupling a high frequency electric field with the electrically conducting metal. The word "metal" is herein employed to connote both elemental metals as well as their alloys. This electrically conducting low melting point metal preferably completely surrounds the outer surface of the crucible and, especially when melted, makes excellent heat-conductive contact therewith. The high frequency electric field readily heats and melts this electrically conducting metal which, in turn, heats the refractory crucible and its contained charge almost entirely by conduction to temperatures of about 1000° C. and principally by conduction, although with increasing radiant heating effects, to temperatures considerably above 1000° C. Since the liquid metal surrounds the crucible containing the charge, it acts as an

electric shield absorbing the high frequency field and preventing the radiation and coupling of the high frequency field into and with the internal charge. The term "high frequency electric field" is used herein to describe an electric field having an alternating frequency above 100 cycles per second and preferably in the neighborhood of several hundred kilocycles per second.

In a modification, a double-wall non-electrically conducting sealed refractory crucible is employed and the electrically conducting low melting point metal fills the major portion of the internal chamber defined by the walls of the crucible. A self-enclosed crucible unit in accord with the invention is thus produced which may be easily adapted for use in conventional high frequency induction apparatus.

The novel features believed characteristic of the invention are set forth with particularity in the appended claims. The invention itself however together with further objects and advantages thereof can best be understood by reference to the following description taken in connection with the accompanying drawing in which Fig. 1 is a cross-sectional view of high frequency heating apparatus embodying the invention and illustrating its use in the production of a silicon monocrystal, and Fig. 2 is a sectional view of a modified self-contained crucible unit which may conveniently be used in the high frequency induction apparatus of Fig. 1.

Referring to Fig. 1, one form of the invention is shown embodied in apparatus 10 for melting relatively pure silicon. Silicon is a semiconductor having relatively poor electrical conducting properties at room temperature so that it does not readily absorb power from a high frequency electric field while in its solid state. At higher temperatures, however, and especially when in a liquid state, silicon becomes electrically conducting and may be readily heated inductively. In apparatus 10 a crucible unit 27 including a crucible 11 of non-conducting refractory material such as quartz is provided for receiving the silicon charge 12 to be melted. The side and bottom wall of crucible 11 should be quite thin preferably less than 0.1 inches. Charge 12 is shown in Fig. 1 in a partially molten state. Crucible 11 is surrounded by an electrically conducting metal 13 having a melting point considerably lower than the temperature to which the charge 12 is desired to be elevated and, for most applications, preferably not above 500° C. in order that metal 13 may liquefy early in the heating cycle. Metals having a melting point about 300° C. are particularly convenient. In the heating operation illustrated by Fig. 1, the silicon charge 12 is to be melted and metal 13 must have a melting point much lower than 1430° C., the melting point of pure silicon. The temperature to which charge 12 is desired to be elevated is hereinafter referred to as the "operating temperature" of the apparatus. Metal 13 should also have a boiling point above this "operating temperature" for the gas pressure involved, and for most practical applications, should have a boiling point above 1600° C. and preferably above 2000° C. at a pressure of one atmosphere. Metal 13 may conveniently comprise tin, lead, indium, gallium and alloys of these elements preferably including bismuth. Eutectic forming alloys may be particularly suitable because of their low melting point and long "slush" range on solidification. An alloy composition of 75% tin, 25% bismuth by weight, has

been found particularly convenient since it does not crack a quartz crucible 11 even with rapid heating or cooling.

Electrically conducting metal 13 is contained within a second crucible 14 and is supported by crucible 14 in good heat conductive contact with the outer wall of crucible 11. Crucible 11 is maintained partially immersed within metal 13 and is prevented from merely floating on the surface thereof by means of a cover 26 clamped or sealed around the rim of outer crucible 14 and pressed against the top of crucible 11. Cover 26 has a central aperture 28 to permit entrance to the interior of crucible 11 and has a plurality of circumferentially spaced holes 29 to permit gas flow into the upper space between crucibles 11 and 14. Outer crucible 14 may be composed of either electrically conducting or non-conducting refractory material and may conveniently be quartz or graphite. An electrically conducting coil 15 for transferring high-frequency electrical energy surrounds the heating apparatus 10 and generates a high frequency electric field which couples with the electrically conducting low melting point metal 13. Crucible 14 is supported by a quartz stand 25, and a heat and electricity insulating material, such as silica wool 16 is preferably interposed between crucible 14 and the outer casing 17 of apparatus 10. A heat shield spaced from the outer wall of crucible 14 may alternatively be employed in place of the silica wool 16.

In order to melt the silicon charge 12 in a non-oxidizing atmosphere, a cover 18 is sealed in a fluid tight manner around the mouth of outer casing 17, and the resulting enclosed chamber is flushed with a chemically inactive gas such as helium by means of a fluid inlet conduit 19 and a fluid outlet 20 formed in cover 18. A drawing rod 21 is inserted through the center of outlet 20 and carries a seed crystal 22 which may be dipped into charge 12 when molten and then slowly elevated while rotating in order to grow a silicon monocrystal. A window 23 may conveniently be provided in the top of an upwardly extending side arm 24 of casing 17 in order to permit inspection of the charge during the melting operation.

The crucible unit 27 of Fig. 1 comprising crucible 11, crucible 14, metal 13, and cover 26 is preferably assembled separately and then placed on stand 25 within apparatus 10. In making crucible unit 27, metal 13 is merely melted within crucible 14 by any conventional method, crucible 11 is then forcibly immersed to the desired depth within liquid metal 13, and cover 26 is fused to the upper rim of crucible 14 and preferably is also fused to the upper rim of crucible 11. The crucible unit 27 is then allowed to cool, and metal 13 solidifies generally in the same position it assumed while in the liquid state.

In the operation of high frequency induction apparatus 10, the silicon charge 12 is placed within crucible 11, cover 18 is sealed to casing 17 and the interior of the heating chamber thus formed is flushed with helium gas at a pressure sufficient to exclude the outer atmosphere. A high-frequency, electrical current is then supplied to coil 15, and a high-frequency electrical field is produced within apparatus 10. This high-frequency electric field couples with the electrically conducting metal 13 and quickly heats this metal above its melting point. As metal 13 becomes molten, it flows in intimate heat-con-

ducting contact with the side and bottom walls of crucible 11 and conductively heats charge 12. As the temperature of the molten metal 13 is raised by increasing the high frequency electric field, the temperature of the silicon charge 12 rises accordingly with fairly quick response as a result of the improved heat conduction derived from the intimate liquid metal contact with the charge-containing crucible 11. As the temperature of metal 13 rises above 1000° C. it begins to heat charge 12 by radiation as well as conduction but there is still excellent uniformity of heating since the conduction heating remains the primary influence at least until temperatures above 1500° C., especially if crucible 11 is thin-walled as described. At about 1430° C., the pure silicon charge 12 begins to melt, and in its molten state is capable of becoming directly heated inductively from a high frequency electric field. If the molten silicon charge 12 is below the level of liquid metal 13 in the space between the side walls of crucibles 11 and 14, however, no appreciable high-frequency induction heating of molten charge 12 occurs since liquid metal 13 acts as an excellent shield almost completely absorbing the energy of the high frequency field. It will be appreciated that the rate of heating as well as the heat capacity of the apparatus can be easily regulated by the amount of electrically conducting metal employed and the level of the electrically conducting metal along the side walls of crucible 11. In general, the greater the amount of electrically conducting metal 13, the greater the heat capacity of the apparatus; and the higher the level of the electrically conducting metal against the side wall of crucible 11, the more uniform is the heating of the charge contained within the crucible. It will also be appreciated that the tendency of electrically conducting metal 13 surrounding crucible 11 to act as a shield for the high-frequency electrical energy is reduced as the level of electrically conducting metal 13 within the space between crucibles 11 and 14 is lowered.

Referring now to Fig. 2, we have shown a single self-enclosed crucible unit 30 in substitution for the double-crucible unit 27 of Fig. 1. Crucible unit 30 is somewhat more durable than crucible unit 27 and can be fabricated with greater ease and accuracy. Crucible unit 30 has an inner cup-shaped wall 11' and an outer cup-shaped wall 14' joined together at the top and defining an internal cup-shaped chamber 31. Walls 11' and 14' are also preferably fairly thin, less than 0.1 inch, and have constant spacing between their coextending portions, which spacing may conveniently be between $\frac{1}{8}$ and $\frac{1}{4}$ inch, depending upon the size of the crucible unit desired. Chamber 31 is partially filled with electrically conducting metal 13, and the remainder portion of the chamber is either evacuated or filled with a gas at a pressure corresponding at operating temperature to the pressure of the gas to be used in the high-frequency heating apparatus. Chamber 31 is only partially filled with metal 13 in order to provide for the thermal expansion of metal 13 as it is heated. If the remainder portion 32 of chamber 31 is evacuated, the induction apparatus 10 should be flushed with a gas which at high temperatures can permeate through the walls of crucible 30. Otherwise the unequal pressures developed on either side of walls 11' and 14' of crucible 30 may cause the crucible to collapse at high temperatures. If quartz is used for crucible 30, helium gas is

preferably used to flush apparatus 10 since quartz is permeable to helium gas at high temperatures. If a gas is employed to flush apparatus 10 which does not permeate through the material of crucible 30 at high temperatures, the remainder portion 32 of chamber 31 is preferably filled with a similar gas under the same pressure at operating temperature as that to be used in flushing the apparatus 10 thereby to equalize the pressures on both sides of walls 11' and 14' of crucible unit 30.

Crucible unit 30 may easily be made from a Dewar flask of the desired cup-shaped configuration and having a fluid conduit tube opening into the base of the flask. The flask is turned upside down, evacuated, and liquid metal 13 is dripped into the flask until only a small unfilled portion remains. The inlet tube is then sealed off and the sealed flask returned to its upright position while metal 13 is still molten. Metal 13 then seeks its own level in the resulting crucible unit 30 and solidifies when cooled.

In a typical operation of high-frequency induction apparatus 10 using a crucible unit 30 having an overall diameter of $1\frac{1}{4}$ inches with a 2.5 cc. working capacity, about a 5 gram charge of pure silicon was melted in less than 1 hour with a maximum of about two kilowatts of power dissipation through 6 turns of $2\frac{1}{2}$ inches diameter coil 15 at a frequency of 500 kilocycles. The metal 13 employed was an alloy of 75% tin, 25% bismuth by weight and the apparatus was flushed with helium gas at atmospheric pressure. In carrying out the melting operation, the power was initially adjusted such that the metal 13 melted in about 5 minutes; the power was increased at a rate to elevate the temperature of liquid metal 13 to about 1200° C. in about 20 minutes; and then the power was slowly further increased until the silicon charge began to melt and become completely molten within about $\frac{1}{2}$ hour. Much greater amounts, for example 100 grams, of pure silicon have likewise been successfully melted in our high-frequency apparatus using larger crucible units 30 and a maximum power dissipation of about 4 kilowatts at 500 kilocycles.

It will be appreciated that although we have described above particular embodiments of our invention, many modifications can be made and we intend by the appended claims to cover all such modifications as fall within the true spirit and scope of the invention.

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

1. Electric heating apparatus for heating a charge to an elevated operating temperature comprising an electrically non-conducting refractory crucible for receiving the charge, an electrically conducting metal having a melting point lower than said operating temperature, means for supporting said metal while in a liquid state in contact with a surface of said crucible opposite to the surface containing the charge, and means for establishing a high frequency electric field through said metal.

2. The electric heating apparatus of claim 1 wherein the crucible comprises quartz and has a wall thickness less than 0.1 inch.

3. Electric heating apparatus for heating a charge to an operating temperature above several hundred degrees centigrade comprising an electrically non-conducting refractory crucible for receiving the charge, an electrically conducting metal having a melting point substantially

lower than said operating temperature, means for supporting said crucible partially immersed within said metal while said metal is in a liquid state, and an electric coil for establishing a high frequency electric field through said metal.

4. The electric heating apparatus of claim 3 wherein said crucible comprises quartz and has a substantially uniform wall thickness less than 0.1 inches.

5. The electric heating apparatus of claim 3 wherein said electrically conducting metal has a melting point below 500° C.

6. The electric heating apparatus of claim 3 wherein said electrically conducting metal comprises an alloy having a melting point below 500° C.

7. The electric heating apparatus of claim 3 wherein said electrically conducting metal has a melting point below 500° C. and a boiling point above 1600° C. at atmospheric pressure.

8. In high frequency induction heating apparatus for heating a charge to an operating temperature above several hundred degrees centigrade, a crucible unit comprising a first crucible, an electrically conducting metal located within said first crucible and having a melting point substantially lower than said operating temperature, a second crucible composed of electrically non-conducting refractory material and having outer dimensions smaller than the inner dimensions of said first crucible, and means secured to said first crucible for maintaining said second crucible partially immersed within said metal when said metal is in a liquid state.

9. For use in high frequency induction apparatus for heating a charge to an operating temperature above several hundred degrees centigrade, a crucible unit comprising an electrically non-conducting thin-walled refractory crucible for receiving the charge, an electrically conducting metal having a melting point lower than said operating temperature and a boiling point above said operating temperature, and fluid supporting means surrounding said crucible and supporting said metal in contact with the outer surface thereof.

10. For use in high frequency induction heating apparatus, a crucible unit having a cup-shaped quartz inner wall less than 0.1 inches thick and an outer wall surrounding said inner wall in spaced relation therewith, an electrically conducting metal having a melting point less than 500° C. and a boiling point above 1600° C. at atmospheric pressure located in the space between said inner and outer walls, and means for fixedly supporting said inner and outer walls in said spaced relation when said metal is in a liquid state.

11. The crucible unit of claim 10 wherein the electrically conducting metal comprises tin.

12. The crucible unit of claim 10 wherein the electrically conducting metal consists of an alloy of tin and bismuth with tin as the major constituent.

13. For use in high frequency induction apparatus for heating a charge to an operating temperature above several hundred degrees centigrade, a crucible unit having inner and outer cup-shaped, electrically non-conducting, thin, refractory walls defining an internal cup-shaped chamber therebetween, and an electrically conducting metal having a melting point below the operating temperature located within and partially filling said chamber.

14. The crucible unit of claim 13 wherein the inner and outer walls are composed of quartz less than 0.1 inch thick, and the metal has a melting point below 500° C. and a boiling point above 1600° C. at atmospheric pressure.

15. The crucible unit of claim 13 wherein the inner and outer walls are composed of quartz and the electrically conducting metal comprises tin.

16. The crucible unit of claim 13 wherein the cup-shaped chamber defined by said inner and outer walls is enclosed by said walls, and said chamber is filled, in addition to said electrically conducting metal, with a gas under pressure.

17. The crucible unit of claim 13 wherein the cup-shaped chamber defined by said inner and outer walls is enclosed by said walls, and the portion of said chamber that is not filled with said metal is evacuated.

18. The method of heating a charge within a quartz crucible to an operating temperature above several hundred degrees centigrade, which method comprises supporting a metal having a melting point substantially lower than said operating temperature in contact with the outer surface of said crucible, and coupling a high frequency electric field with said metal to heat and liquefy said metal whereby said liquid metal transfers heat to a charge within said crucible primarily by conduction.

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