

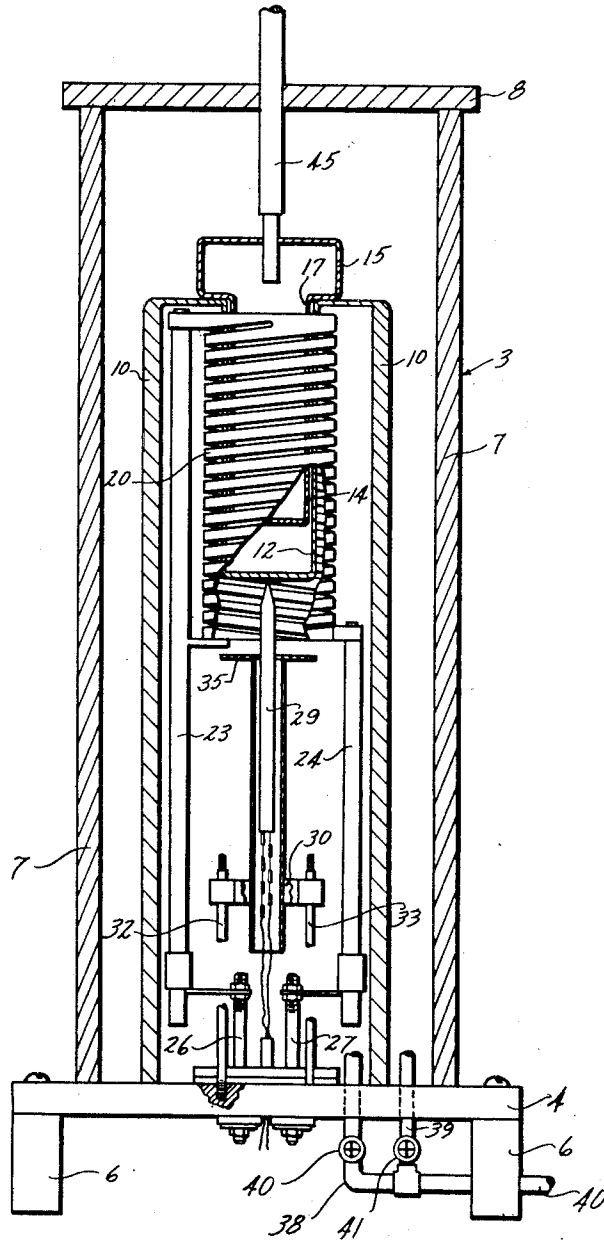
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FURNACE FOR PRODUCING SINGLE CRYSTALS FOR TRANSISTORS

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FURNACE FOR PRODUCING SINGLE CRYSTALS FOR TRANSISTORS

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3 Claims. (Cl. 13—31)

This invention relates to electric furnaces.

The furnace of this invention is the only one, so far as known, which does not add impurities to a melt.

The hereinafter disclosed furnace is especially used to melt materials which should not be in the least contaminated, such materials for example being silicon and germanium, used to produce transistors, it being known that an ultra high degree of purity should be maintained in the materials used for such purpose. In fact, it is most desirable that there be no contaminants which are detectable spectroscopically, and the present furnace meets this requirement, and consistently so. Consistency or uniformity of the product is highly important, and is not obtained by other furnaces.

The furnace is also adapted to be used with pulling mechanism, in the formation of a single crystal of silicon or germanium, for example, single crystals being essential, as known, for the making of transistors, and success in obtaining a single crystal being largely dependent on maintaining high purity of the molten material in a furnace. Moreover, the higher the purity, the better the lifetime characteristics of transistors.

Another advantage of the furnace, hereinafter described and claimed, is that it avoids the present radio frequency induction heating system, now much used, which is undesirable not only because it produces some contamination, but also because it requires very elaborate, and therefore very expensive, control equipment, costing about \$25,000 for use with a very small furnace. The R. F. system of heating was resorted to in an attempt to avoid contaminating the melt, but has been unsuccessful because the structure of such furnaces has not been correct, notwithstanding the great amount of effort, time and money, expended on them in view of the importance of transistors and the desired extremely high purity.

The drawings illustrate the invention, as required, and in these:

The single figure is a longitudinal section of a furnace, partly broken away.

Referring to the drawing for a more detailed description thereof, the numeral 3 indicates the furnace in general. The bottom 4 of the furnace is elevated on legs 6 and is secured thereto, and may be of metal or other suitable material. An outer cylindrical tube 7 rests on the bottom 4 and is hermetically sealed thereto, and a top or cover 8 for the tube is also so sealed. Said tube, with its cover, is preferably a quartz, SiO_2 , tube, but is not necessarily of that material. An inner tube 10 of quartz, SiO_2 , also rests on the mentioned base 4 and is hermetically sealed thereto, and is spaced from the outer tube 7. It is here to be remarked that the term quartz, as used in this specification, includes any form of SiO_2 which forms an impervious tube or enclosure.

Said inner tube 10 is formed with a cylindrical reentrant well 12 in its upper portion, the well also being of quartz, and in this well a cylindrical quartz, SiO_2 , crucible 14 is disposed, being spaced from said well laterally and at its bottom. The crucible has an enlarged upper

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end 15 which rests on top of the inner tube 10, being thereby supported in proper position, and it has a reduced opening 17 at its upper end to receive a charge to be melted, and also to withdraw a crystal from the molten charge.

A heater 20, of helical form, surrounds the well 10 and extends below it, being adjacent to the well but spaced slightly from it. The heater is preferably of graphite, but may be of other high melting point material, as tantalum or molybdenum for example. The heater is supported by quartz-enclosed conductors 23 and 24 which may be of tantalum or other high melting point material, and which are secured respectively to the upper and lower end of the helix, said conductors being connected to, and supported by, bolts 26 and 27 respectively, which pass thru the bottom 4 of the furnace, to be connected to a source of power. The drawing also shows a thermocouple 29, which includes a quartz enclosing-tube and which contacts the bottom of the well 12 so that the temperature may be known and controlled, and is supported by a clamp 30 which is held in position by rods 32 and 33, the lower ends of these rods being embedded in the bottom 4 of the furnace. The thermocouple passes thru a heat-reflecting tantalum shield 35, the latter being horizontally disposed, and spaced a little below the lower end of the heater, being provided to reflect and therefore conserve the heat of the latter.

Means are provided for exhausting air and other gases and vapors from the furnace, and preferably for exhausting independently from the space inside and outside the inner tube 10; such means being shown as comprising pipes 38 and 39, which are connected to a common pipe 40 which leads to a vacuum pump. It will be noted that pipes 38 and 39 are provided respectively with shut off valves 40 and 41 and that pipe 38 leads into the bottom of inner quartz tube 10, and also that pipe 39 leads into the lower end portion of the outer tube 7.

The drawing further shows pulling means extending thru the top 8 of the outer tube 7 and into the quartz crucible 14, this means comprising a vertical rod 45 slidably passing thru the top or cover 8, which may be thru a seal, and is intended to be simultaneously rotated and pulled outwardly, when attached to a "seed" crystal of the same material as that in the crucible. The "seed" crystal is lowered into the molten material in the crucible and the rod 45 is then slowly pulled outwardly, while rotating. Thereby the molten material crystallizes on the seed crystal and thus produces a large crystal.

The crucible wall, and also the wall of the inner tube 10, is about $\frac{1}{16}$ inch thick, and the crucible inner diameter about $1\frac{1}{2}$ inch. The space between the bottom of the crucible and the bottom of the well 12 is about $\frac{3}{4}$ inch, while the lateral space between the crucible and the well is about $\frac{1}{8}$ inch. The given dimensions are suitable for a furnace about 3 times the size shown in the drawing, and the usual charge in the crucible may be from 50 to 60 grams of silicon. For such charge, about 60 amperes of current are sent thru the heater.

It will be seen from the foregoing description that the heater 20 is enclosed by impervious walls, and is isolated from the crucible and its contents, whereby particles and gases from the heater may not contaminate the crucible charge, the heater, running as it does at a high temperature, being the main source of such contamination, and it will be appreciated by those skilled in the art that the construction of the furnace is otherwise such as to eliminate contamination.

Before running the first melt, a new furnace should be thoroly degasified by running the vacuum pumps with the furnace heated. Subsequently, with a charge of silicon in the crucible, the furnace is pumped down to a vacuum of about 10^{-3} mms. After about a half hour at

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this pressure, the temperature is run up to about 1000° C. and held there for about 2 hours, with the vacuum held as stated. The temperature is then increased to about 1420° C., which is about the melting point of silicon, and the vacuum brought to 10⁻⁵ mms. of mercury, the charge being held under these conditions for an hour or more. The process stated serves to purify the silicon by removing gases, and impurities volatile at the stated temperatures, which are removed from the furnace by the vacuum condition, and the process also serves to remove gases and some impurities from components of the furnace itself. A similar process is followed for other metals, as germanium, for example, regard being had for the melting point of the metal.

For more precise heating, the tantalum reflector is replaced with a graphite heater which is made with a spiral base. Provision is then made to position the shorter of the two power electrodes to the center of the graphite base. The molten charge is thereby also heated directly from below as well as from the sides. This reduces the temperature gradients in the molten mass since the charge is heated evenly throughout.

What is claimed is:

1. An electric furnace comprising an upright quartz tube, including in its upper portion a reentrant well, a quartz crucible within said well and spaced above the bottom, and from the side, of the well, a helical graphite heater enclosed within said tube and disposed around and adjacent said well and adapted to be electrically heated conductively, tantalum electrical conductors con-

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nected to the ends of said heater and supporting the latter, quartz shields around said conductors, a tantalum heat reflector below and adjacent to the lower end of said heater, a second tube around and spaced from said quartz tube and crucible, and means for exhausting gases and vapors from the furnace.

2. An electric furnace comprising an upright quartz tube including in its upper portion a reentrant well, a quartz crucible within said well, a heater around and adjacent said well and adapted to be electrically heated, a second tube around said quartz tube and spaced therefrom, and means for independently exhausting gases and vapors from around the well and within the quartz tube, and from within the well and crucible and the space between the quartz and second tubes.

3. In an electric furnace, a quartz tube comprising in its upper portion a reentrant well, a crucible within said well and spaced above the bottom and from the sides of said well, and a heater around and adjacent said well and within said quartz tube and sealed off airtight from said crucible, and a second airtight tube around said quartz tube and said crucible, both of said tubes being exhaustible of air.

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