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United States Patent

Keller

[15] 3,688,006

[45] Aug. 29, 1972

[54] **DEVICE FOR CRUCIBLE-FREE, FLOATING-ZONEMELTING A CRYSTALLINE MEMBER**

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[22] Filed: **March 27, 1970**

[21] Appl. No.: **23,357**

[30] **Foreign Application Priority Data**

March 29, 1969 Germany..........P 19 16 318.9

[52] **U.S. Cl.**..................**13/1,** 13/DIG. 1, 174/152 R, 219/10.79

[51] **Int. Cl.**...........................**F27d 11/06,** H05b 5/08

[58] **Field of Search**..........13/DIG. 1, 1, 20; 174/151, 174/152 R; 219/10.79

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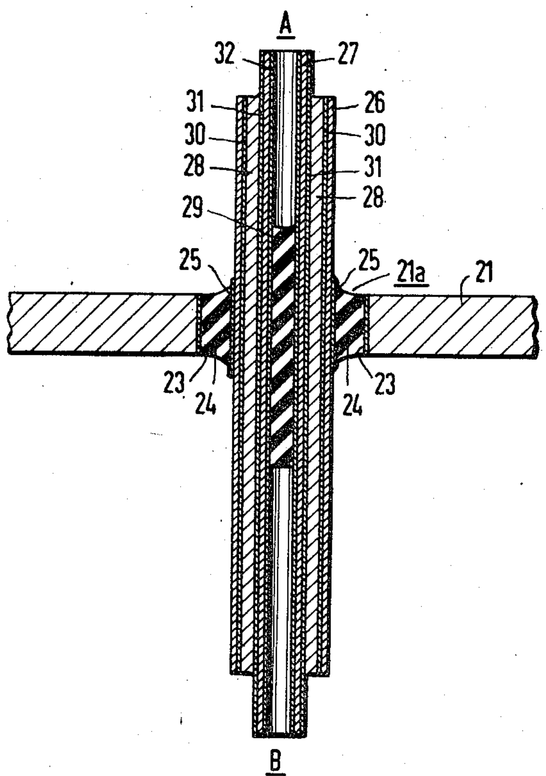
[57] **ABSTRACT**

Device for zone melting a crystalline member includes a vessel, an electrically energizable heating device mounted in the vessel for heating a melting zone formed in a crystalline member, and an electric current supply for the heating device extending into the vessel through an opening formed in a wall of the vessel, the supply comprising at least two conductors insulated from one another by an intermediate layer of insulating material having a Shore hardness in the range of 10 to 100 and an extensibility of at least 20 percent, a gas-tight adhesive connection being provided between the intermediate layer of insulating material and the surface of the conductors.

3 Claims, 3 Drawing Figures



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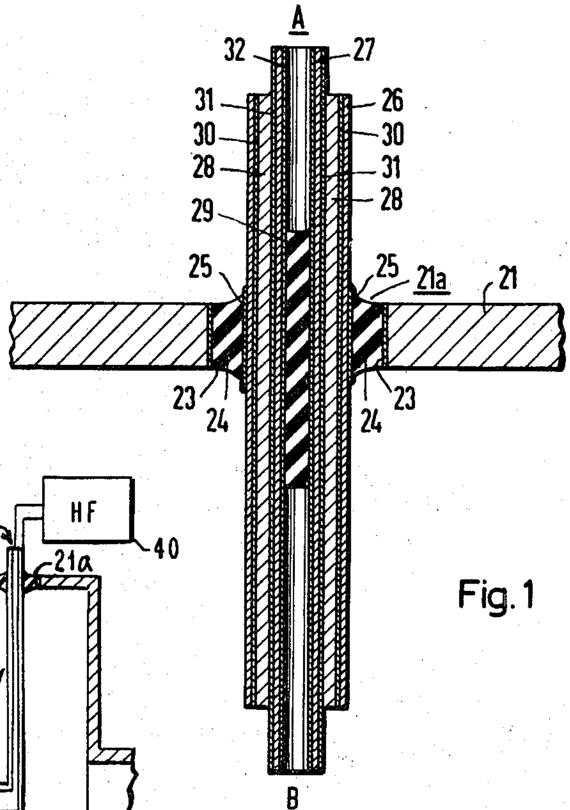

Fig. 1

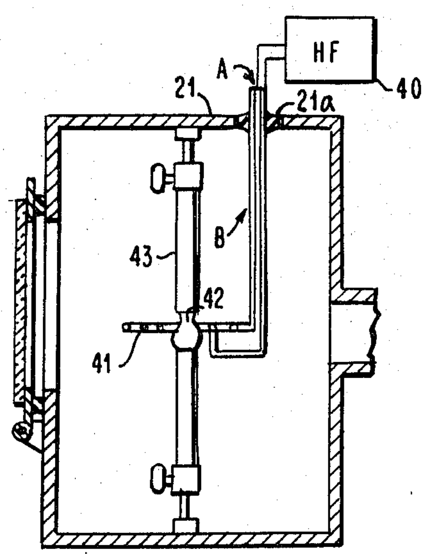

Fig. 3

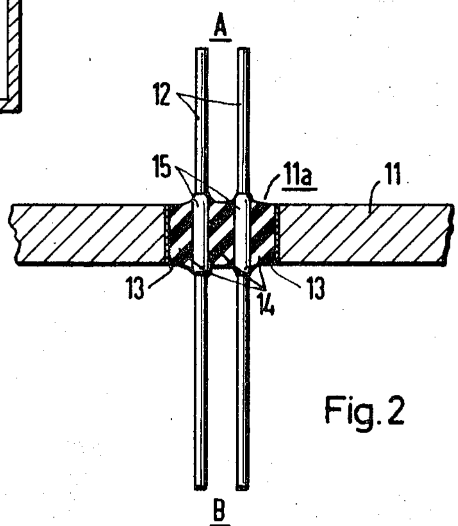

Fig. 2

DEVICE FOR CRUCIBLE-FREE, FLOATING-ZONEMELTING A CRYSTALLINE MEMBER

My invention relates to device for zone melting a crystalline member.

A device foe zone melting, especially for cruciblefree, floating zone melting of a crystalline member, generally includes a vessel in which holder for the crystalline member and a heating device for heating a molten zone in the crystalline member are disposed. The vessel is either evacuated or is filled with protective gas such as noble gases or a mixture of hydrogen and nitrogen, for example. The heating device is energized with electric current and consequently is formed of a current supply made up of at least two conductors and which passes through an opening in the vessel wall from the outside. The current supply can be, for example, a coaxial conductor assembly wherein the intermediate space between both tubular conductors is filled with an insulating material. This intermediate layer of insulating material such as band-forming polytetrafluorethylene cast in paraffin or the material known under the trade name Araldit has been found to be virtually gas-tight when employed in a zone melting process of semiconductor rods which are not too thick.

Especially when zone melting, for example, relatively thick semiconductor rods for which a relatively high power is required and wherein therefore a relatively high power loss occurs in the current supply, it was observed that undesired oxide layers always formed on the semiconductor rods being treated in spite of careful sealing of the melting chamber. The invention of the instant application is based upon the realization that the cause of this are fine leaks which form between the intermediate layer of insulating material and the surface of the conductors of the current supply during the zone melting operation because the insulating material tends to tear at least partly due to thermal expansion and contraction of the conductors. Due to these leaks, the outer atmosphere can penetrate into the melting chamber and produce the oxide coatings.

It is accordingly an object of the invention to provide device foe crucible-free, floating zone melting a crystalline member which avoids the foregoing disadvantage of the heretofore known devices of this general type.

More specifically it is an object of the invention to provide such device which avoids the formation of leaks and consequently prevents penetration of the surrounding atmosphere into the melting chamber and the production of oxide layers.

With the foregoing and other objects in view I provide, in accordance with my invention, a device for zone melting a crystalline member comprising a vessel, an electrically energizable heating device mounted in the vessel for heating a melting zone formed in a crystalline member, and an electric current supply for the heating device extending into the vessel through an opening formed in a wall of the vessel, the supply comprising at least two conductors insulated from one another by an intermediate layer of insulating material having a Shore hardness in the range of 10 to 100 and an extensibility of at least 20 percent, a gas-tight adhesive connection being provided between the intermediate layer of insulating material and the surface of the conductors.

If the current supply is nondisplaceably disposed in the opening extending through the vessel wall, in accordance with another feature of the invention, I fill that opening with insulating material which has a Shore hardness within the limits of 10–100 and extensibility of at least 20 percent, and a gas-tight adhesive connection is provided between the insulating material, on the one hand, and the vessel wall as well as the surface of at least one of the conductors, on the other hand.

Other features which are considered as characteristic for the invention are set forth in the appended claims.

Although the invention is illustrated and described herein as embodied in device for crucible-free, floating zone melting a crystalline member, it is nevertheless not intended to be limited to the details shown, since various modifications and structural changes may be made therein without departing from the spirit of the invention and within the scope and range of equivalents of the claims.

The construction and method of operation of the invention, however, together with additional objects and advantages thereof will be best understood from the following description of specific embodiments when read in connection with the accompanying drawing, in which:

FIG. 1 is a sectional view of part of a wall of a vessel for zone melting a semiconductor rod through an opening of which there extends a current supply formed of two conductor tubes disposed coaxially to one another;

FIG. 2 is a view similar to that of FIG. 1 wherein instead of the coaxial conductor tubes of FIG. 1, a current supply formed of two wire-shaped current conductors extends through an opening in the vessel wall; and

FIG. 3 is a diagrammatic view of a complete zone melting apparatus according to the invention.

Referring now to the drawing and first particularly to FIG. 1 thereof, there is shown a fragmentary section of a wall 21 of a vessel forming part of a device for zone melting a crystalline member. An opening or passage 21*a* is provided in the wall portion 21. Within the passage 21*a* there is disposed a current supply formed of two conductor tubes 26 and 27 of copper which are located coaxial to one another. At the upper end A of the conductors 26 and 27 there is connected a current source 40 (FIG. 3), for example, a high frequency generator, while the lower end B which is within the vessel is connected to a heating device 41 for heating a molten zone 42 formed in a crystalline rod 43 which is end-supported in the vessel. As shown, the heating device 41 is in the form of an induction heating coil.

Both the coaxial conductor tubes 26 and 27 are of different diameters and are insulated from one another by a layer 28 of cold-vulcanized silicon rubber which is disposed intermediate The conductor tubes 26 and 27. A good adhesive connection between the insulating intermediate layer 28 and the conductor tubes 26 and 27 is ensured by means of silicon resin coating 30 applied to the inner surface of the outer conductor tube 26 and a silicon resin coating 31 applied to the outer surface of the inner conductor tube 27.

The inner conductor tube 27 can also have a silicon resin coating 32 applied to the inner surface thereof and can be gas-tightly closed by means of a stopper 29 formed of cold-vulcanized silicon rubber.

Cold-vulcanized silicon rubbers which are suitable for the aforementioned purposes are, for example those of the West German firm Wacker having the trade names "Sil-Gel" and "Vergussmasse K." The silicon resin coatings on the surfaces of the conductor tubes **26** and **27** can be formed of the silicon resin produced by the same firm Wacker under the trade name "G 718."

Cold-vulcanized silicon rubber is of Shore hardness in the range of 10–100 and an extensibility of 20 percent. A cold-vulcanized silicon rubber having a Shore hardness of 50 and an extensibility of about 100 percent is preferred. Cold-vulcanized silicon rubber can therefore readily expand at temperature variations without being torn away from the silicon resin coatings **30**, **31**, and **32** and thereby from the surfaces of the conductor tubes **26** and **27**. Shore hardness is a measure of hardness which is determined by a ball-dropping test (recoil or rebound measuring); note Römpp "Chemie Lexikon," Vol. 6, Frankische Verlagshandlung, Stuttgart, page 5,874.

A further advantage of an intermediate layer of insulating material consisting of cold-vulcanized silicon rubber is that the dielectric loss factor thereof is smaller than 10^{-2} and the dielectric strength thereof lies between 30 and 40 kV/mm so that this intermediate layer is also well suited for a current supply through which a high frequency induction heating coil is provided with electric energy.

The opening **21***a* is also filled with cold-vulcanized silicon rubber **24**. The surfaces of the vessel wall **21** and of the outer conductor tube **26** are provided within the opening **21***a* with silicon resin coating **23** and **25** to which the silicon rubber **24** firmly adheres so that the opening **21***a* is gas-tightly closed.

The current supply formed of the coaxial tubes **26** and **27** can also be displaceable in axial direction in the passage or opening **21***a* formed in the vessel wall **21**. In such a case, instead of the silicon resin coatings **23** and **25** as well as the silicon rubber filling **24**, and oil seal can be located, for example, within the opening **21***a* to effect sealing thereof. Also in such a case it is advantageous moreover for the coaxially disposed conductor tubes **26** and **27**, exactly as shown in FIG. **1**, to be insulated from one another by an intermediate layer of cold-vulcanized silicon rubber and for the inner tube **27** to be gas-tightly closed with stopper **28** of cold-vulcanized silicon rubber.

Advantageously, my invention is also utilizable for a passage through the wall of a vessel employed in a zone melting operation wherein a current supply formed of two wire-shaped conductors is disposed. In FIG. **2**, such an opening or passage **11***a* in a section **11** of the wall of a vessel forming part of a device for zone melting of a crystalline member is illustrated. The current supply is formed of conductors **12** consisting of two copper wires. At the upper end A of the copper wires **12**, a nonillustrated current source such as a high frequency generator is connected, while the lower end B thereof is connected with a nonillustrated heating device for heating the molten zone formed in a crystalline member that is mounted in the melting zone vessel. Within the opening or passage **11***a*, the vessel wall **11** and the surface of the copper wires **12** are provided with coatings **13** and **15** formed of silicon resin. Moreover, the opening or passage **11***a* is filled with cold-vulcanized rubber **14**. A gas-tight and durable adhesive connection exists between the silicon rubber **14** and the silicon resin coatings **13** and **15**.

The mounting of current supplies according to FIGS. **1** and **2** into the openings or passages formed in the vessel wall is especially simple when employing cold-vulcanized silicon rubber. This is because, after the application of silicon resin coatings on the conductors of the current supply and on the vessel wall surface within the opening or openings as well as within the inner spaces of the conductor tubes **26** and **27** in FIG. **1**, molding can be readily effected with the initially liquid silicon rubber to which a hardener such as, for example, a hardener T of the aforementioned firm Wacker has been added.

I claim:

1. Device for zone melting a crystalline member comprising a vessel, an electrically energizable heating device mounted in said vessel for heating a melting zone formed in a crystalline member and an electric current supply for said heating device extending into said vessel through an opening formed in a wall of said vessel, said supply comprising at least two conductors insulated from one another by an intermediate layer of insulating material having a Shore hardness in the range of 10 to 100 and an extensibility of at least 20 percent, a gas-tight adhesive connection being provided between said intermediate layer of insulating material and the surface of the conductors, the opening formed in said vessel wall being filled with insulating material also having a Shore hardness in the range of 10 to 100 and an extensibility of at least 20 percent, and a gas-tight adhesive connection being provided between the insulating material, on the one hand, and said vessel wall as well as the surface of at least one of said conductors, on the other hand.

2. Device according to claim **1**, wherein said insulating material is cold-vulcanized silicon rubber, and the surface of said one conductor has a layer of silicon resin at the location thereof at which it is adhesively connected to the silicon rubber insulating material, said silicon rubber insulating material adhering to said layer of silicon resin.

3. Device according to claim **2**, wherein said silicon rubber has a Shore-hardness of substantially 50 and an extensibility of substantially 100 percent.

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