

June 26, 1962

J. E. HICKS ETAL

3,041,133

METHOD AND APPARATUS FOR PREVENTING BREAKAGE OF LINERS

Filed Sept. 27, 1960

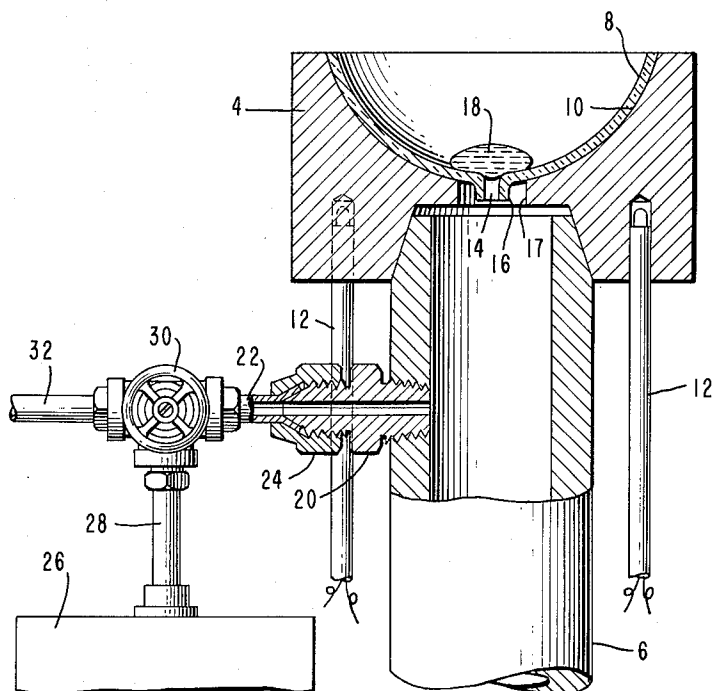


FIG. 1.

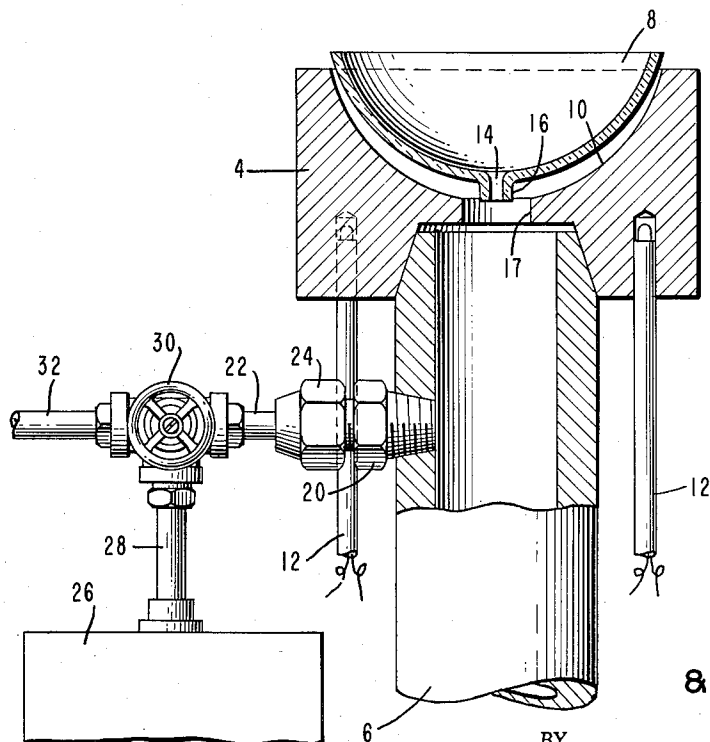


FIG. 2.

INVENTORS

John E. Sigler
& John E. Hicks

BY *Stevens, Davis, Miller & Mosher*
ATTORNEYS

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**METHOD AND APPARATUS FOR PREVENTING
BREAKAGE OF LINERS**

John Edwin Hicks, Richardson, and John Elmer Sigler,
Dallas, Tex., assignors to Texas Instruments Incorporated,
Dallas, Tex., a corporation of Delaware
Filed Sept. 27, 1960, Ser. No. 58,736
3 Claims. (Cl. 23—1)

This invention relates to the production and purification of silicon crystals and more particularly to a method and apparatus for producing silicon crystals in a graphite crucible having a quartz liner in a manner to prevent the breakage of the quartz liner.

Crystals of various chemical elements and compounds such as germanium, silicon, and the like are used in semiconductor devices which are assuming a role of increasing importance in the electronic industry. Various methods are in use today for producing and refining semiconductive materials having the essential properties of high purity and homogeneity. However, in the refining of such materials, difficulties may be present when working with one particular metal or material which may not occur when working with another. For instance, when working with germanium, which has a relatively low melting temperature of approximately 980° C., the material can be handled easily in graphite containers or crucibles, and does not appear to attack the graphite container appreciably during the refining processes. Also, germanium can be melted and solidified in a graphite crucible without destroying the crucible upon subsequent cooling. In addition, graphite is a very common material and is easily machined. On the other hand, silicon has a much higher melting point, about 1420 to 1440° C., and graphite is unsuitable as a container to hold the material during the refining processes. Experimentation has shown that although quartz is the most acceptable crucible material at the present time, at these high temperatures an undesired reaction occurs between the quartz and the silicon. If a portion of the molten silicon is allowed to solidify in the quartz crucible, bonding of the silicon to the quartz occurs and the crucible will shatter or crack upon cooling due to the difference in thermal expansion and contraction of the two materials.

During the melting of a silicon mass within a quartz crucible or container, there usually results a small amount of leaching of impurities from the quartz container, such as boron, and it is readily apparent that if the quartz container or crucible could be recovered and reused after the initial operation, any subsequent operation with the same quartz container would occur with a substantial reduction in the amount of leaching of such impurities from the container. It is believed that after a number of repeated reuses of the same quartz crucible, leaching of the impurities, such as boron, from the container by the silicon mass during the refining operation would be reduced.

In a copending application of Walter R. Runyan, Serial No. 758,379, filed September 2, 1958, and assigned to the assignee of the present invention, a method and apparatus for producing and refining silicon materials is fully described and claimed. In accordance with one form of the invention of the copending application, there is provided a graphite crucible or container having a quartz liner in which the silicon is melted prior to the formation of the end product. A small aperture is formed in the bottom of the quartz liner which is small enough to prevent the escape of the molten silicon under normal gravitational force. After the silicon material is completely melted and homogeneous, and the well-known "crystal pulling" process is employed for producing a single

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crystal of high purity silicon, the molten mass remaining in the quartz liner is removed by increasing the pressure above the upper surface of the material so as to force the material through the aperture. The crucible is then free of any residue silicon, and may be reused in subsequent operations. However, when the crucible is permitted to cool, it has been found that the difference in the expansion coefficients of quartz and graphite during cooling cause the valuable quartz liner to become broken.

It is an object of the present invention to prevent the breakage of such a crucible liner by providing a method and apparatus for lifting the liner out of contact with the crucible during cooling.

It is another object of the present invention to provide a method and apparatus utilizing a cushion of air for lifting a quartz liner out of the supporting well of a graphite crucible to prevent breakage of the quartz liner during cooling.

It is a further object of the invention to provide a method and apparatus for preventing the breakage of quartz liners during cooling which is simple and effective in operation, economical, and does not require mechanical engagement or contact with the liner.

Further objects and advantages of the present invention will become readily apparent as the following detailed description of the invention unfolds and when taken in conjunction with the drawings wherein:

FIGURE 1 is a fragmentary sectional view of a crucible embodying features of the present invention; and

FIGURE 2 is a fragmentary sectional view similar to that of FIGURE 1 illustrating the quartz liner in its raised position out of engagement with the supporting wall of the crucible.

Referring to FIGURE 1, one embodiment of the present invention comprises a crucible 4 made of a suitable material, such as graphite, which is supported on the upper end of the tubular support 6 preferably made of the same material as the crucible 4. A quartz liner 8 is supported within a cavity 10 in the upper surface of the crucible 4. Suitable thermocouple tubes 12 may also be provided for sensing the temperature of the crucible.

An aperture 14 with a downwardly flared annular lip 16 is provided in the bottom of the quartz liner 8, and a larger opening 17 is formed in the bottom of the crucible 4 to enable the aperture 14 to communicate directly with the central opening in the tubular support 6. As described in the aforementioned copending application, the silicon material is melted in the crucible. The diameter of the aperture 14 is small enough to hold the molten silicon in the liner by surface tension, thereby preventing leakage. A diameter in the order of $\frac{1}{16}$ to $\frac{1}{2}$ of an inch has been found to produce satisfactory results with the conventional "crystal pulling" apparatus.

After an ingot of silicon is melted in the liner 8 and a single crystal of the material is formed by the conventional pulling operation, as described in the copending application, a small residue 18 of silicon remains. To remove the residue from the liner, the pressure of an inert gas above the residue 18 is increased to a value sufficient to overcome the surface tension of the residue with respect to the aperture 14. A gas pressure of 2" to 3" of mercury (Hg) was found to be sufficient to produce the desired results. Suitable cooling and collecting means (not shown) are provided beneath the tubular support 6 to quickly solidify and collect the silicon after it passes through the aperture 14.

After the residue 18 is completely cleared from the crucible, and the crucible is permitted to cool, the difference in the expansion coefficients of the quartz liner 8 and the graphite crucible 4 can cause the liner to become broken. The present invention prevents the breakage of

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the quartz liner 8 during this cooling period by lifting the liner upwardly to the position illustrated in FIGURE 2 by increasing the gas pressure beneath the liner 8. Thus, the liner is in effect floated upwardly on a cushion of gas. A pressure of 2 to 5 p.s.i. is usually sufficient to float the liner 8. When introduced below the liner, some of the gas will escape upwardly through the aperture 14, but enough of the gas will be directed around the annular lip 16 and between the liner 8 and the wall of the cavity 10 to float the liner upwardly to the position illustrated in FIGURE 2.

The gas pressure may be increased beneath the liner 8 in any suitable manner but, in accordance with one embodiment of the invention, gas is introduced into the tubular support 6 through a threaded plug 20 threaded into the wall of the tubular support. A suitable conduit 22 is detachably connected to the threaded plug 20 by a nut 24 on the end of the conduit which threadably engages the left end of the threaded plug. A suitable source 26 of pressurized gas may then be connected to the conduit 22 by a conduit 28 with a valve 30 disposed therebetween to regulate the flow and pressure of the gas. With this construction, gas can be introduced into the central opening of the tubular support beneath the liner 8 to float the liner upwardly as previously described. When the crucible and liner have cooled sufficiently, the gas pressure is reduced to allow the liner to rest in the crucible 4.

Thus, the present invention provides a method and apparatus for effectively and dependably preventing breakage of the quartz liner. The liner is very gently floated upwardly and lowered on a cushion of gas which eliminates damage to the liner that might occur if it were physically lifted by some type of mechanical gripping means. Further, by introducing the gas from beneath the liner, the need for apparatus which would require valuable head room above the cavity to perform the lifting operation is eliminated. This avoids interference with "crystal pulling" apparatus which may be positioned above the cavity 10.

If desired, the apparatus of the present invention can also be utilized to decrease the gas pressure beneath the liner 8 by connecting a conduit 32 to a suitable evacuating device, for example. In this manner, the pressure differential across the aperture 14 required to force the residue 18 out of the liner can be created, and this will eliminate the need for providing the means necessary to increase the gas pressure above the cavity 10 to remove the residue 18, as previously described.

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Although the present invention is especially useful in the production of silicon, it would find utility in any application in which it is desirable to physically separate the crucible and crucible liner. The above-described method and apparatus are specific embodiments of the invention, and many modifications may be made thereto without departing from the spirit and scope of the invention, which is limited only as defined in the appended claims.

What is claimed is:

1. The method of preventing the breakage of a quartz liner of a crucible during cooling, which includes the step of introducing pressurized gas between the liner and the crucible to float the liner in spaced relation from the crucible during the period that the liner and crucible are cooling.

2. The method of preventing the breakage of the quartz liner of a crucible during cooling, said crucible having an aperture at the bottom thereof, said liner having an annular lip at the bottom thereof enclosing an aperture extending into the aperture of said crucible, said lip being positioned in spaced relation with the wall of said crucible, which includes the step of introducing pressurized gas between said crucible and said liner through the space between said crucible wall and said annular lip, whereby the gas, working its way between the liner and the crucible, gently lifts the liner upwardly out of engagement with the crucible.

3. The method of preventing the breakage of a quartz liner during cooling, said crucible having an aperture at the bottom thereof, said liner having an annular lip at the bottom thereof enclosing an aperture extending into the aperture of said crucible, said lip being positioned in spaced relation with the wall of said crucible, which includes the steps of introducing pressurized gas between said crucible and said liner through the space between said crucible wall and said annular lip, whereby the gas, working its way between the liner and the crucible, gently lifts the liner upwardly out of engagement with the crucible, and removing said pressurized gas after the liner and crucible have cooled sufficiently, thereby to gently lower the liner back into engagement with the crucible.

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