March 13, 1962 W. R. RUNYAN 3,025,146

METHOD OF GROWING SHAPED CRYSTALS

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Fig. 1.

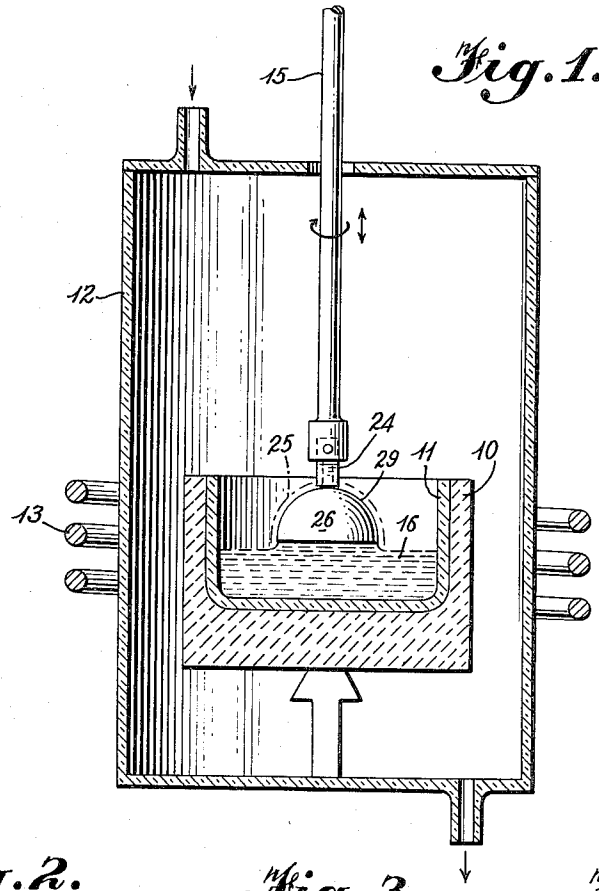

Fig. 2.

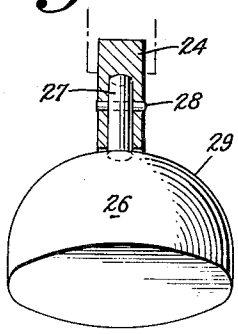

Fig. 3.

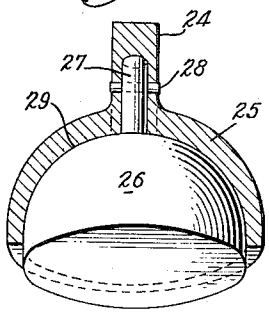

Fig. 4.

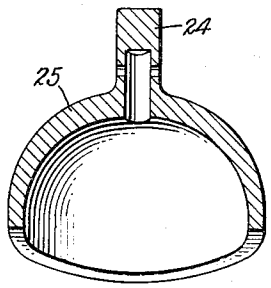

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Fig. 5.

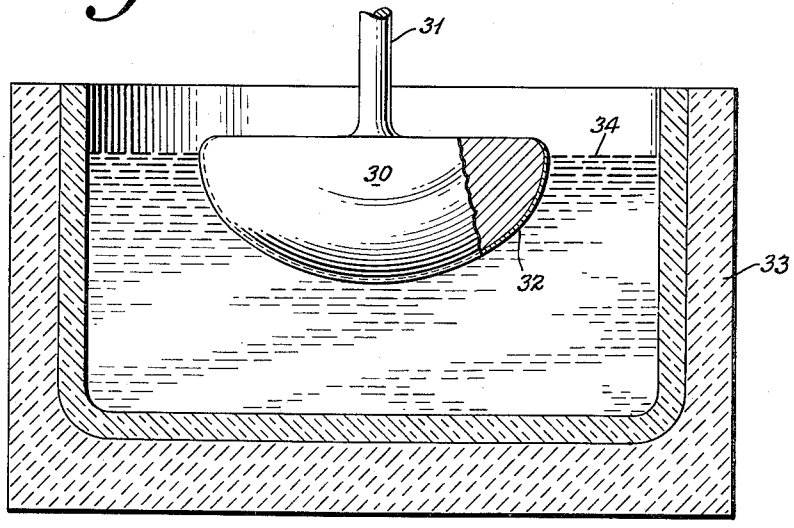
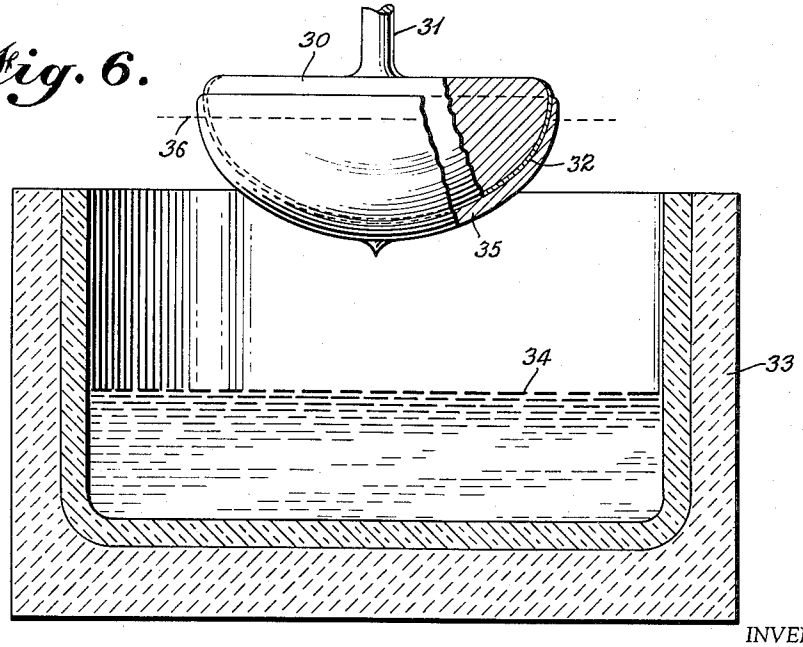


Fig. 6.



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METHOD OF GROWING SHAPED CRYSTALS

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8 Claims. (Cl. 23—301)

This invention relates to a method of producing shaped articles of a semiconductor material, and more particularly to a method of growing shaped germanium or silicon crystals from a melt.

Large crystals of germanium and silicon have been found to be of great practical utility as windows and lenses in infrared systems. The crystals employed are optically finished to various shapes. This is usually accomplished by growing in conventional fashion a large crystal and grinding away unwanted regions to produce the desired shape. In addition to being excessively time consuming, the technique of grinding results in the loss of considerable semiconductor material.

It is therefore an object of the present invention to provide a method of producing shaped crystals of semiconductor material which greatly reduces the amount of grinding required and thus enables substantial saving in time and cost of production, particularly as regards semiconductor material.

The method is particularly adapted to the growing of dome-shaped crystals of germanium and silicon.

These and other objects and the nature and advantages of the instant invention will become readily apparent from the following description taken in conjunction with the accompanying drawings, wherein:

FIG. 1 is a view, partly in section, of one form of apparatus that can be used to carry out the method of the present invention;

FIG. 2 is a view in section showing a plug attached to a seed crystal;

FIG. 3 is a similar view showing the plug about which a shaped crystal has been grown;

FIG. 4 is a similar view showing the shaped crystal formed in accordance with the present invention, after the plug has been removed;

FIG. 5 is a view showing an alternative technique for growing a shaped crystal; and

FIG. 6 is a view similar to FIG. 5 showing the grown crystal.

Referring now to the drawings, a conventional crystal pulling apparatus is shown in FIG. 1 consisting essentially of a graphite crucible **10** having a quartz liner **11** enclosed within a housing **12**. A R.F. coil **13** surrounds the crucible **10** and heats it by induction. Inert gas is passed through the housing as indicated. A pull rod **15** arranged for combined rotary and vertically axial movement extends into housing **12** in conventional fashion.

In accordance with the present invention, shaped crystals of semiconductor material are grown by attaching a plug **26** of suitable shape and design to a crystal seed **24** mounted in the end of rod **15**. A crystal **25** is grown from a melt in the crucible by operation of the apparatus in accordance with standard practice. The crystal grows on the outer surface of the plug **26** to the thickness and shape desired.

The plug **26** is made of any material substantially inert to the semiconductor crystal being grown, or at least the surface of plug **26** is provided with a material substantially inert to the semiconductor crystal being grown. For example, in the case of germanium, the plug **26** can be made of graphite. When growing a crystal of silicon, the plug can be made of silicon coated with an oxide, silicon nitride or boron nitride. The material selected for the plug must be harmonized with the semiconductor material especially from the standpoint of thermal coefficients of expansion and other related properties.

The plug **26** can be attached to the crystal seed **24** by any suitable means. As illustrated in FIG. 2, a hole is drilled in the crystal seed **24** into which a rod-like projection **27** on the plug **26** is inserted. The projection **27** is shown retained therein by means of a pin **28** passing through the crystal seed **24** and the rod-like projection **27**. When it is desired to remove the plug **26**, the pin **28** is removed, thus permitting the plug **26** to be removed. Other means can be utilized, such as by cementing the plug **26** to the crystal seed **24** or by any other suitable means that will permit subsequent removal of the plug **26**.

The plug **26** is provided with an upper surface **29** which corresponds to the desired shape of crystal to be grown. The drawings illustrate a dome-shaped upper surface **29** on the plug **26** that will permit the growth of a dome-shaped crystal **25** as shown in FIG. 4.

An example of the method of growing a crystal in accordance with the teachings of the present invention will now be given. The crucible **10** is first provided with a charge of material **16** of the desired composition. A crystal seed **24** is attached to the bottom end of rod **15** by conventional means. A graphite plug **26** having an upper surface **29** of the desired shape, dome-shaped, for example, is attached to the lower end of the crystal seed **24**. The crystal seed **24**, with the plug **26** attached thereto is carefully lowered into the molten material **16** until the plug **26** is entirely sumberged and the lower end of the crystal seed **24** touches the surface of the molten material **16**. The rod **15** is then rotated and moved vertically upwardly slowly. As the rod **15** is withdrawn, semiconductor material crystallizes onto the crystal seed **24**. The temperature is controlled during crystallization in accordance with standard practice.

After a desired length of crystal has been grown, as for example the length shown in FIG. 3, the crystal-plug assembly is lifted from the melt. The plug **26** is removed after cooling to yield the dome-shaped crystal **25** as shown in FIG. 4.

The shape of the plug may be varied as desired to yield crystals of the desired shape. Besides the configuration shown and previously described, it will be appreciated that it is possible to continue the crystal growing process as discussed above, and grow a crystal of semiconductive material completely surrounding the plug **26**. Under these circumstances, it would be necessary to cut the crystal in half, in order to remove the plug **26**. This would have one advantage, namely, if the plug were formed with a convex lower surface, the lower half of the grown crystal would also have a complementary concave surface.

A further technique for producing shaped crystals is illustrated in FIGS. 5 and 6. This technique for producing crystals of silicon uses a silicon plug, identified by the reference numeral **30**. As shown, the plug **30** is substantially hemispherical and is provided with a small projection **31** substantially centrally of its plane surface. Projection **31** is adapted to be connected with the pull rod of the crystal pulling apparatus in a conventional way. The convex surface of the plug **30** is coated with a layer **32** of material which will inhibit crystal growth on the convex surface when the plug is inserted into a silicon melt. Suitable materials for this purpose are silicon monoxide and silicon nitride. It will be appreciated, however, that any other materials which perform the desired function may be used.

The coated plug is introduced into the silicon melt **34** contained in a quartz crucible **33** of a conventional crystal pulling apparatus such as shown in FIG. 1. The melt

34 is characterized by a temperature gradient such that the portions of the melt around the periphery of the crucible and adjacent the surface are the hottest, whereas, the portion of the melt centrally within the crucible and adjacent its bottom is the coolest. This is due to recognized factors. The silicon material of the melt will commence crystallizing during a normal pulling operation and will stick to the silicon plug especially about the region of the plug adjacent the surface of the melt. A crystal **35** will be grown onto the plug substantially as shown in FIGURE 6, but will be capable of being freed from the silicon plug **30** due to the presence of the layer **32** of material coating the surface of the plug **30**. Removal of the crystal **35** can be accomplished in several ways, for example by making a cut by means of a diamond wheel as indicated in FIG. 6 by dotted lines **36**.

It will be obvious to those schooled in the art that various changes may be made without departing from the spirit of the invention and therefore the invention is not limited to what is described in the specification but only as indicated in the appended claims.

What is claimed is:

1. The method of growing a shaped single crystal of semiconductor material which comprises melting a mass of semiconductor material, introducing into the melted material a single crystal seed portion having a shaped plug portion attached thereto, surface of said plug portion being substantially inert to said semiconductor material, withdrawing the single crystal seed portion and attached plug portion from the melted material to cause the melted material to crystallize onto the single crystal seed portion and around the plug portion to produce a single crystal of said material having a shape complementary to that of said plug portion, and removing said plug portion from said grown single crystal.

2. The method as in claim 1, wherein said single crystal seed portion and melted material are germanium.

3. The method as in claim 2, wherein said plug portion is made of graphite.

4. The method as in claim 1, wherein said single crystal seed portion and melted material are silicon.

5. The method as in claim 4, wherein said plug portion is selected from the class consisting of silicon, silicon nitride and boron nitride.

6. The method as in claim 1, wherein said plug portion has a dome-shaped upper surface.

7. The method as in claim 3, wherein said plug portion has a dome-shaped upper surface.

8. The method as in claim 5, wherein said plug portion has a dome-shaped upper surface.

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