

March 1, 1960

W. SHOCKLEY

2,927,008

CRYSTAL GROWING APPARATUS

Filed Oct. 29, 1956

4 Sheets-Sheet 1

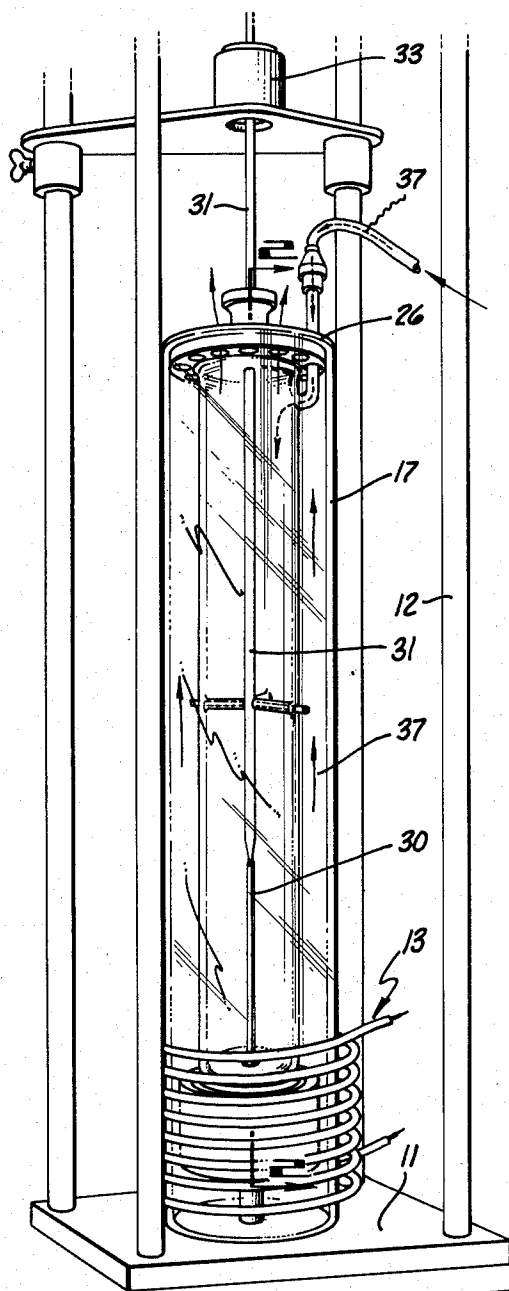


FIG. 1

INVENTOR.
WILLIAM SHOCKLEY
BY
Alfred E. Swain
ATTORNEYS

March 1, 1960

W. SHOCKLEY
CRYSTAL GROWING APPARATUS

2,927,008

Filed Oct. 29, 1956

4 Sheets-Sheet 2

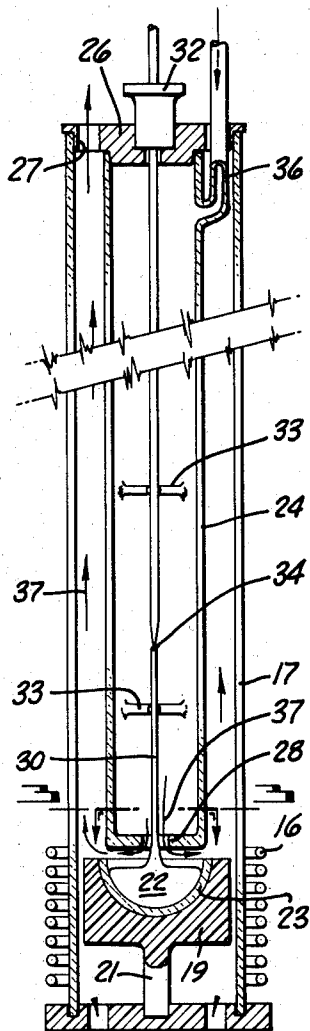


FIG. 2

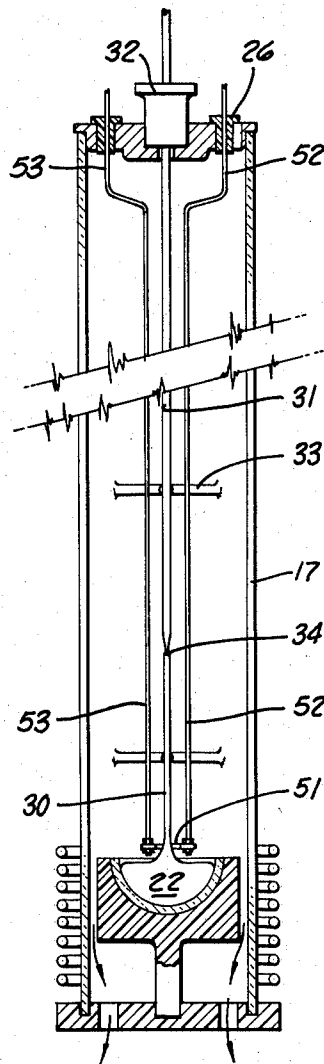


FIG. 4

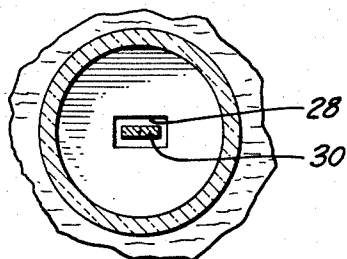


FIG. 3

INVENTOR.
WILLIAM SHOCKLEY
BY *Lehr & Surin*
ATTORNEYS

March 1, 1960

W. SHOCKLEY
CRYSTAL GROWING APPARATUS

2,927,008

Filed Oct. 29, 1956

4 Sheets-Sheet 3

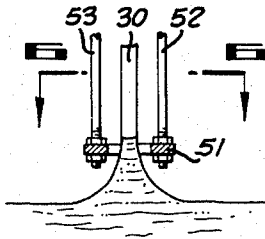


FIG. 5

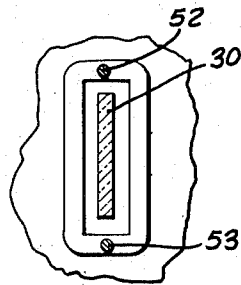


FIG. 6

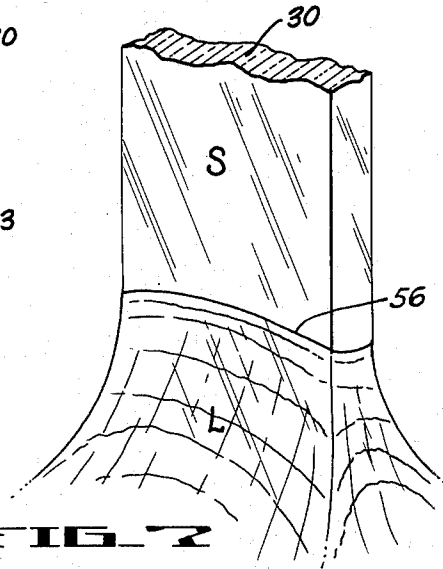


FIG. 7

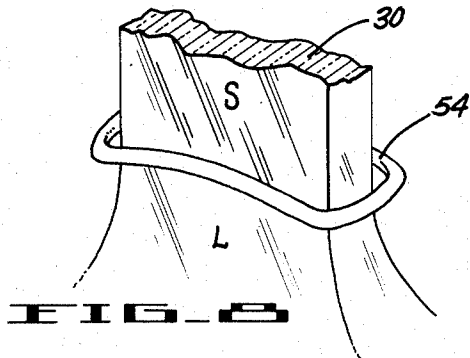


FIG. 8

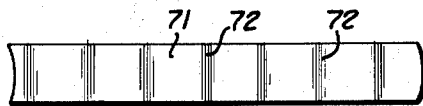


FIG. 10A

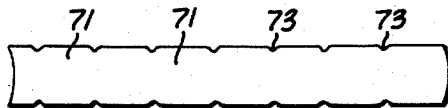


FIG. 10B

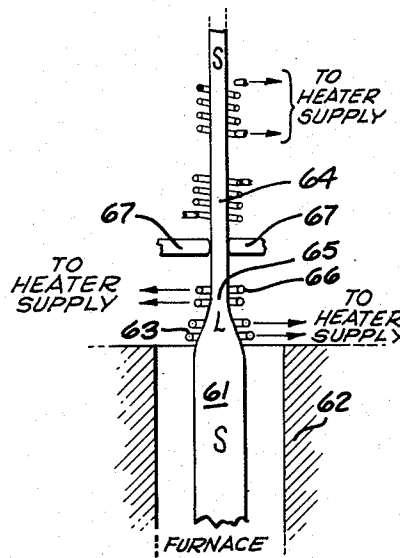


FIG. 9

INVENTOR.
WILLIAM SHOCKLEY
BY *Flaherty & Swain*
ATTORNEYS

March 1, 1960

W. SHOCKLEY
CRYSTAL GROWING APPARATUS

2,927,008

Filed Oct. 29, 1956

4 Sheets-Sheet 4

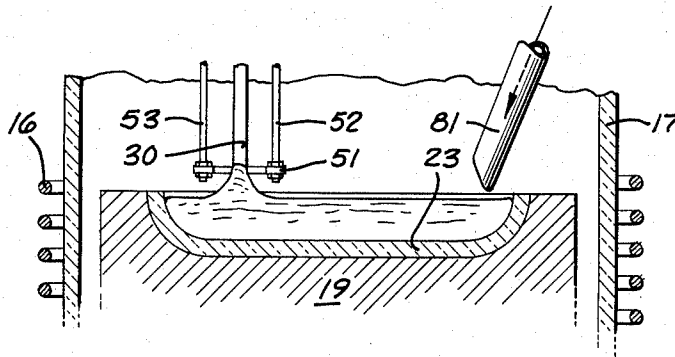


FIG. 11

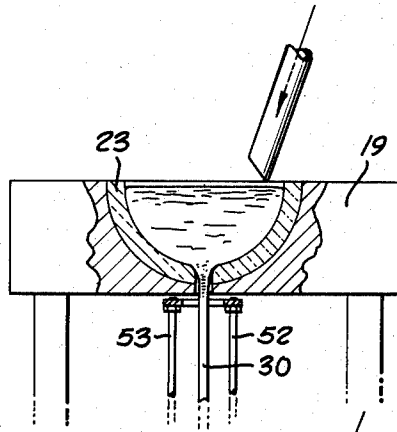


FIG. 12

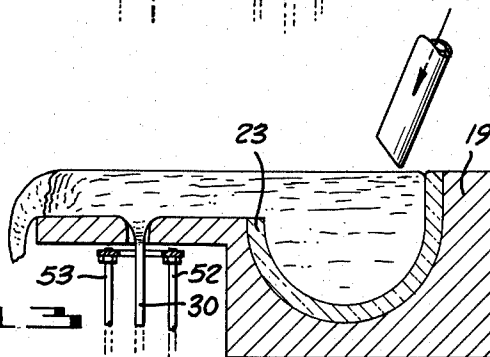


FIG. 13

INVENTOR.
WILLIAM SHOCKLEY
BY
Flehr & Swain
ATTORNEYS

1

2,927,008

CRYSTAL GROWING APPARATUS

William Shockley, Los Altos, Calif., assignor to Shockley Transistor Corporation, Palo Alto, Calif., a corporation of California

Application October 29, 1956, Serial No. 618,899

1 Claim. (Cl. 23—273)

This invention relates generally to a crystal growing method and apparatus, and more particularly to a crystal growing method and apparatus for growing elongated crystals having small cross sectional areas.

In the prior art, single crystals of semiconductive material, for example, germanium or silicon, have been grown by the so-called "crystal pulling" process. In this process, pure semiconductive material is melted in a crucible in a non-reactive atmosphere. A seed, a small piece of a single crystal, is properly oriented and dipped into the melt. The seed is then pulled upward at a suitable rate. In practice the rates vary over a wide range, for example, between 0.1 inch per hour to as high as 4 inches per hour and more. Generally, the crystal is simultaneously rotated at rates up to 150 r.p.m. and higher.

As the seed is withdrawn, the molten semiconductive material solidifies about it and the grown crystal is formed. Generally, the diameter of the grown crystal is much larger than the diameter of the seed. A typical example is a seed with a maximum dimension on a side of approximately 0.25 inch will produce a resulting crystal of dimensions of 1½ inches in diameter and more.

In a grown junction transistor the melt is doped as the crystal is withdrawn to thereby give zones of the proper conductivity types within the crystal. The zones may also be formed by the rate grown process.

The grown crystal is cleaned and etched, and the location of the various boundaries is noted. The crystal is then sliced and diced by means of diamond saws and the like to form wafers which are suitable for use in the end semiconductive devices.

Because of the size of the saws and relative width of the various cuts in slicing and dicing, as much as 30% or more of the grown crystal is wasted. The process is uneconomical. Strains are frequently set up in the grown crystal due to thermal gradients across its large cross section.

It is a general object of the present invention to provide an improved crystal growing method and apparatus.

It is another object of the present invention to provide a crystal growing method and apparatus in which an elongated crystal of small cross section can be grown.

It is a further object of the present invention to provide a crystal growing method and apparatus in which an elongated crystal having a small predetermined cross section may be grown, the crystal being suitable for cutting into short pieces for directly making the desired wafers.

It is a further object of the present invention to provide a crystal growing method and apparatus in which heat exchange means in the crystal growing region control the temperature of the region to thereby control the size and shape of the crystal which is pulled from the melt.

It is a further object of the present invention to provide a crystal growing method and apparatus in which a crystal having a predetermined cross sectional contour

2

and area may be grown by controlling heat transfer in the region of solid liquid interface.

It is a further object of the present invention to provide a crystal growing method and apparatus in which a heat sink of predetermined contour and size is provided adjacent the crystal growing region whereby an elongated crystal having a predetermined cross sectional area and contour is grown.

It is a further object of the present invention to provide a crystal growing method and apparatus in which a heat source is placed in the crystal growing region to supply heat thereto for controlling the size and shape of the grown crystal.

It is a further object of the present invention to provide a crystal growing method and apparatus in which a large crystal grown by prior art techniques can be re-grown to form an elongated crystal having a predetermined cross sectional area and contour.

It is a further object of the present invention to provide a crystal growing method and apparatus in which an elongated crystal of small predetermined cross sectional area and contour may be grown and in which regions are formed in the crystal which are mechanically weaker than the remainder of the crystal whereby by mechanically stressing the crystal it breaks up to form suitable wafers.

The invention possesses other objects and features of advantage, some of which, with the foregoing, will be set forth in the following description of the invention. It is to be understood, of course, that the invention is not to be limited to the particular disclosure as other variant embodiments thereof may be adopted within the spirit and scope of the appended claim.

Referring to the drawing:

Figure 1 is a perspective view of a crystal growing apparatus incorporating my invention;

Figure 2 is a sectional view taken along the line 2—2 of Figure 1;

Figure 3 is a sectional view taken along the line 3—3 of Figure 2;

Figure 4 is a sectional view showing another embodiment of my invention;

Fig. 5 is an enlarged view of the crystal growing region of the embodiment shown in Figure 4;

Figure 6 is a sectional view taken along the line 6—6 of Figure 5;

Figure 7 is a perspective view showing a typical meniscus formed when a crystal is pulled from a melt;

Figure 8 shows a heater having a shape corresponding to the meniscus, encircling a rod;

Figure 9 shows another embodiment of my invention in which elongated crystal of small cross sectional area is grown from a larger crystal;

Figures 10A, 10B show a portion of an elongated crystal grown in accordance with another feature of my invention;

Figure 11 shows another embodiment of the invention in which the crystal is grown by a continuous process;

Figure 12 shows another embodiment of the invention in which the crystal is withdrawn through the crucible; and

Figure 13 shows a modification of the apparatus of Figure 12.

Generally my invention contemplates the use of heat exchange means in the crystal growing region to control the heat transfer in the region. By suitably controlling the heat transfer an elongated crystal of small predetermined cross sectional area may be grown. For example, the heat exchange means may comprise a heat sink or a heat source which serves to remove or supply sufficient heat to maintain a thermal equilibrium in the growing region. The heat sink or source has a prede-

terminated size and shape whereby the crystal will grow having a predetermined small cross sectional area with a desired contour.

The crystal growing apparatus illustrated in Figure 1 comprises a base 11 to which vertical support rods 12 are attached. The rods 12 provide means for mounting guide means and other working parts of the apparatus. The oven which is designated generally by the reference numeral 13 is mounted on the base 11. The oven may comprise, for example, an induction heater 16. The outer chimney 17 which preferably is also made of quartz extends downwardly and rests on the base 11. The coils of the induction heater 16 surround the outer chimney. A suitable graphite boat 19 which may include a pedestal 21 is mounted on the base 11 and is disposed within the bottom portion of the chimney 17. The graphite boat 19 is heated by the induction coil and serves to form a melt 22 of semiconductive material therein. If the semiconductive material is of the type, for example silicon, which reacts with graphite, a suitable liner 23, for example quartz, may be inserted in the graphite boat.

Concentric with the quartz chimney is a cylinder 24. The cylinder 24 may be suitably attached to a cover 26 which then serves to suspend the same from the top edge of the chimney 17 and concentric therewith. A plurality of ports 27 are formed in the cover 26 and provide means for gases to circulate outwardly between the chimney 17 and the cylinder 24 as will be presently described. The bottom of the cylinder 24 is flared inwardly to form an opening 28 of predetermined contour and size. The size and contour of the opening 28 serves to determine the cross sectional area and contour (shape) of the crystal which is pulled from the melt 22, as will be presently described.

The crystal 30 is pulled from the melt by means of the rod 31, which is axially disposed with respect to the chimney 17 and the cylinder 24. The rod extends upwardly through the bushing 32 carried by the cover 26 and is guided by the guide means 33 mounted on the vertical posts 12. Guide means 33 may be provided for guiding the rod or grown crystal within the cylinder 24.

As previously described, a seed is mounted on the lower end 34 of the rod and the seed is dipped into the melt 22. Then the rod is pulled upward at a constant rate whereby the molten semiconductive material solidifies as the rod is withdrawn and forms a crystal.

A suitable lead-in connection is provided for introducing an inert gas into the inside of the cylinder 24. For example, the lead-in may include the nipple 36 which may then be attached to a suitable supply of gas. The gas flows downwardly about the growing crystal in the cylinder 24, outwardly through the opening 28 in the region between the opening and the crystal, over the surface of the melt, and upwardly between the chimney 17 and the cylinder 24 and is exhausted through the openings 27. The direction of flow is schematically illustrated by the arrows 37.

By employing a gas supply of constant pressure, the volume of gas which flows through the opening 28 is then dependent upon the size of the passage between the opening 28 and the outside of the growing crystal, that is, it will be dependent upon the difference in cross sectional area of the hole 28 and the cross sectional area of the grown crystal. As the cross section of the crystal increases, the flow of gas is reduced, and as the cross section decreases, the flow is increased. However, as the opening decreases and the flow of gas decreases, the cooling due to the flow of gas is reduced whereby the temperature in the crystal growing region is raised. The cross sectional area of the crystal is reduced. Conversely, as the crystal size is reduced and the opening increased, more gas flows through, thereby providing a

greater cooling effect. This tends to decrease the temperature of the crystal and as a result, a larger crystal is grown.

Thus it is seen that by properly adjusting the size of the opening 28 and the pressure of the gas, an equilibrium condition will result wherein a crystal is grown having a cross section of constant predetermined cross sectional area and contour. It is apparent that a crystal having a cross section of any predetermined desired contour may be grown by appropriately contouring the opening 28 formed in the flange.

It is, of course, apparent that rather than providing a heat sink to control the growth of the crystal, that is, rather than withdrawing heat from the growing region, it is possible to control the size of the grown crystal by adding heat. Thus a heater having a predetermined shape and capacity may be employed.

Referring particularly to Figures 4-6, a crystal growing apparatus including a heat source is illustrated. Like reference numerals refer to like parts of Figures 1 and 2 and will not be described in detail. A heater 51 encircles the crystal in the growing region. The leads 52 and 53 may extend upwardly and out through the cover 26 and have their lower extremity in electrical contact with the heater. In all other respects, the apparatus is similar to that previously described. Referring particularly to Figures 5 and 6, an enlarged view of the heater is shown. It is apparent that by properly contouring the heater and controlling the electrical power supplied thereto, a crystal of predetermined cross sectional area and configuration may be grown.

Referring to Figure 7, a perspective view showing a crystal 30 with a solid-liquid interface 56 is illustrated. The solid-liquid interface 56 will have a meniscus having a shape which depends upon the rate at which the rod is withdrawn and upon the surface tension of the liquid phase. The shape of the meniscus may be calculated knowing the constants involved. Knowing the shape of the meniscus and the heat relationship, a heater having a contour similar to that of the meniscus may be provided to supply the heat necessary to maintain equilibrium conditions. Under these conditions, then, the crystal may be pulled at a constant rate and the process is self stabilizing.

In certain instances, it may be desirable to first grow a crystal by conventional methods and then to employ the method and apparatus of the invention to grow an elongated crystal having predetermined small cross sectional area and contour. A suitable apparatus is schematically shown in Figure 9. Thus the large crystal 61 is placed in a large oven schematically illustrated at 62. The temperature of the oven is such that the crystal is near its melting point but is still in the solid state. Near the upper region of the crystal, a local heater 63 is provided. This heater serves to maintain the upper region of the crystal at such a temperature that it is in a liquid state. The size of the molten region is such that the surface tension serves to hold the molten region stable.

The small elongated crystal of predetermined diameter 64 is withdrawn from the molten region. A suitable heat exchange means, for example a heat source 66, is disposed in the growing region 65 and serves to control the size of the crystal as previously described.

It is apparent, of course, that rather than a heat source, a heat sink of the type previously described may be employed. Suitable guides 67 may be provided for guiding the crystal as it is withdrawn. Also, to reduce the temperature gradients along the grown crystal 64, a suitable heater may be provided along the length of the same. The heater acts to reduce the temperature gradients along the crystal as it is pulled from the melt.

In the growing technique shown in Figure 9, it may be desirable to blow out the impurities from the molten

zone at periodic intervals. This might be accomplished by moving the heat source 63, employing a gas jet to blow out the melted zone with the impurities collected therein. The grown crystal 64 is then lowered and a new melt formed and the crystal continues to be grown until sufficient impurities have again collected at which time the crystal is again flushed out.

The processes described lend themselves very nicely to growing a crystal which may subsequently be mechanically stressed to break it up into short sections to form wafers suitable for forming semiconductive devices. Thus, as illustrated in Figure 10A, the crystal 71 may have regions 72 of different concentration gradients formed therein by doping the melt or by rate growing the crystal. The crystal may then be etched by a selective etch which will etch out these regions to form notches 73, as illustrated in Figure 10B. By subjecting the crystal to suitable mechanical strain, it will break up into short sections to form wafers of semiconductive material suitable for forming semiconductive devices.

The structure illustrated in Figure 10B may also be arrived at directly by varying the heat exchange in the crystal growing region. Thus, by momentarily supplying a large amount of heat, the crystal will be reduced in cross section to form the notches 73. Similarly, by decreasing the gas pressure, the temperature will increase to again reduce the crystal cross section to form the notches 73.

The method and apparatus described lends itself nicely to a continuous process for forming a crystal from a melt. Thus, referring to Figure 11 which carries like reference numbers, the crystal 30 is withdrawn from one side of the molten pool. Simultaneously, pure semiconductive material can be added to the melt. For example, a tube 81 may extend out through the apparatus and the material may be added to the melt through the tube. The crystal may be continuously withdrawn from the melt and the level of the pool maintained constant.

In Figures 12 and 13 another embodiment of the invention is shown. Most semiconductive materials have a high coefficient of surface tension. As a result, a hole 82 may be formed in the bottom of the crucible 19 and the molten material will not flow out. However, a crystal will form on a seed inserted into the material. Thus, a crystal may be pulled from the bottom. The hole 82 is formed to have a predetermined size and contour whereby the grown crystal will have the desired cross sectional area and shape. The crucible 19 will effect a heat exchange in the growing region and serve to control the size. If desired, an additional heat source may be employed. Material may be continuously added to the melt through the tube, as previously described, for a continuous process.

The apparatus illustrated is similar to that of Figure 12. In this embodiment, the lip or shell 83 is formed on the crucible 19 and a hole 82a is formed therein. The crystal is pulled down through the hole, as previously described. An additional heat source may be provided. Additional material is supplied through the tube 81.

In the embodiment shown in Figure 13, molten material flows over the lip. The material flowing outward over the lip may be employed to control the temperature of the melt. Thus, the end of the lip may be cooler than the remainder of the crucible whereby when the melt is at the desired temperature the material solidifies near the ends of the lip. Any increase in the solid zone means the temperature of the melt is too low, while a decrease means the temperature is too high. Means for controlling the temperature are described in copending application Serial No. 601,815, filed August 2, 1956, entitled "Crystal Growing Method and Apparatus."

It is apparent from the foregoing description that other types of heat sinks and sources may be employed. For example, suitable hot gases may be blown over the growing region, or the region may be suitably irradiated.

Although the description has been devoted to the growing of crystals from melts of semiconductive material, it is apparent that the method and apparatus may be employed to grow other elongated structures of predetermined cross sectional area. For example, it may be employed to grow wires, candles, etc.

It is seen that I have provided a crystal growing apparatus in which an elongated crystal having a cross sectional area of small predetermined contour may be formed. The apparatus lends itself readily to being controlled to form a crystal of uniform cross sectional area. Crystals can be grown which may subsequently be mechanically stressed to form wafers for semi-conductive devices. Time consuming and material wasting operations such as slicing and dicing are eliminated.

I claim:

In a crystal growing apparatus of the type adapted to grow a crystal of semiconductive material from a melt of the same, means serving to form a melt, means serving to pull a crystal from said melt, a chimney surrounding said melt and said crystal, a tube disposed within said chimney surrounding said pulling means and a portion of said crystal, said tube having a closure at one end thereof with an opening of predetermined contour surrounding the crystal in the crystal growing region, and means for supplying an inert gas at constant pressure to the other end of said tube whereby the gas flows outwardly from said tube between the crystal and the sides of the opening, said gas flow serving to automatically control the size of the growing crystal.

References Cited in the file of this patent

UNITED STATES PATENTS

1,892,806	Pedersen	Jan. 3, 1933
2,545,271	Gartner	Mar. 13, 1951
2,683,676	Little et al.	July 13, 1954
2,686,864	Wroughton	Aug. 17, 1954
2,809,136	Mortimer	Oct. 8, 1957

OTHER REFERENCES

Hoyem: "Physical Review," vol. 33, January 1929, pages 81-85.