

United States Patent

Harrison et al.

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[54] METHOD OF CRYSTAL GROWTH BY VAPOR DEPOSITION

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[51] Int. Cl. B01J 17/26
[58] Field of Search 23/273 SP, 300, 301 SP, 223.5, 23/294; 117/200, 201, 106, 107.1

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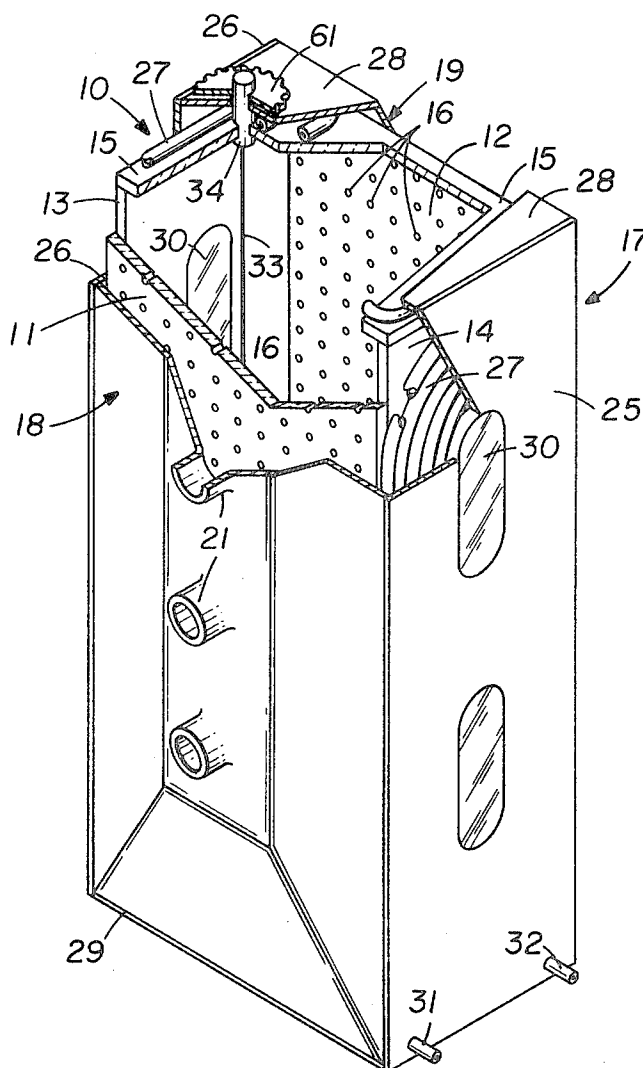
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[57] ABSTRACT

A method for growing elongated rods of silicon or the like by positioning an elongated silicon filament within a reaction chamber, directing the gas from which material is to be deposited over the filament in a direction transverse to the longitudinal axis of the filament at a relatively uniform flow rate along the length of the filament, and while the gas is being directed transversely to the filament, rotating the filament so that all the parts along the surface will be uniformly exposed to the gas.

6 Claims, 5 Drawing Figures



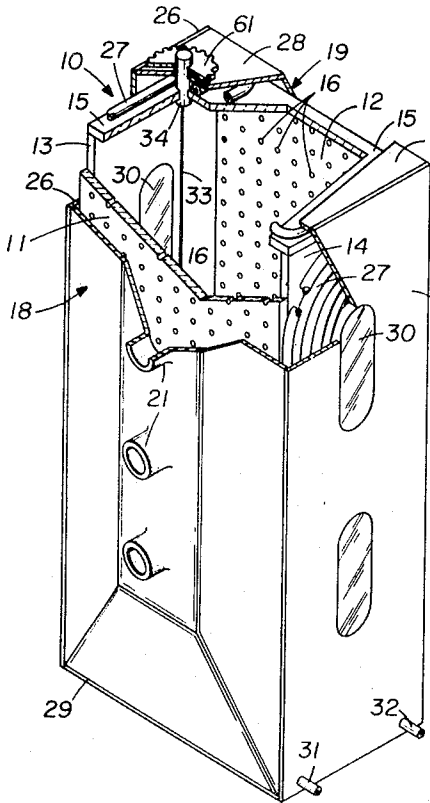


FIG. 1

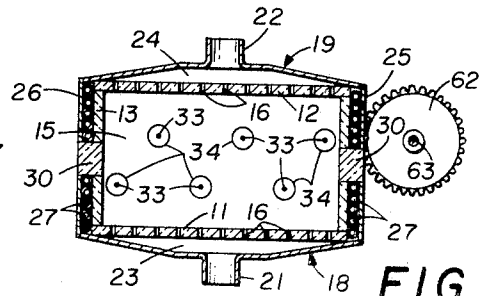


FIG. 3

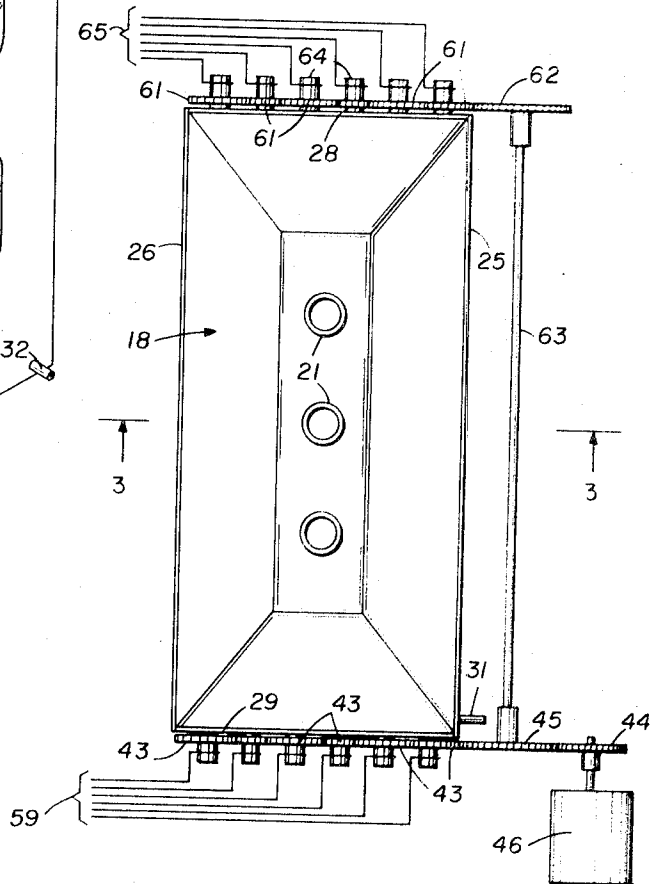


FIG. 2

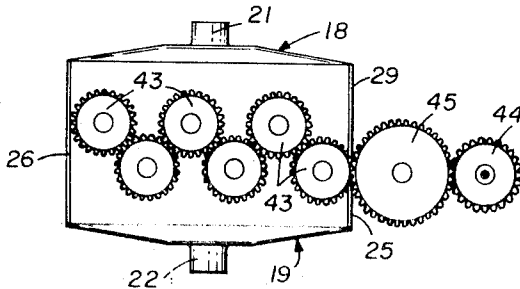


FIG. 4

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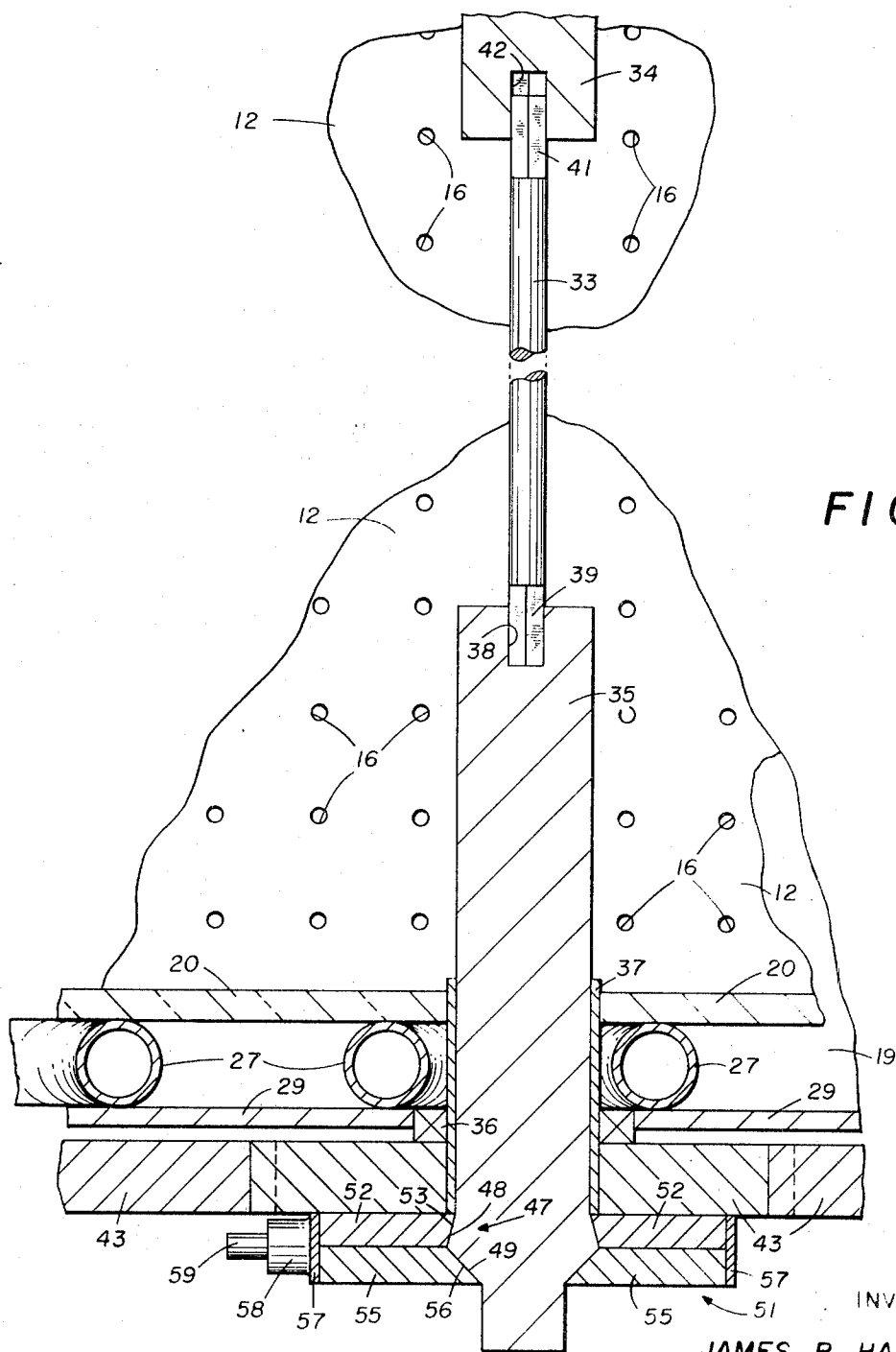


FIG. 5

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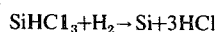
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METHOD OF CRYSTAL GROWTH BY VAPOR DEPOSITION

This invention relates to chemistry, and more particularly, but not by way of limitation, to methods and apparatus for depositing material from a vapor or gas onto a substrate.

In the semiconductor industry, it is common to deposit material from a gas onto a substrate for the purpose of forming various electronic components. In some applications the material deposited from the gas is the same material as that from which the substrate is formed, while in other instances it is a different material from that which the substrate is formed. As an example of the former, in the growth of silicon by vapor deposition techniques, it is common to position an elongated silicon filament between two chucks each of which extend through the end of a quartz container within which the filament is placed. A potential is impressed across the graphite chucks causing a current to flow through the filament. The resistance of the filament to current flow elevates the temperature of the filament to a temperature generally in excess of about 1,100° C. A gas stream, which may comprise a mixture of trichlorosilane and hydrogen, is introduced into the quartz chamber and after flowing along the longitudinal axis of the filament is withdrawn from the chamber. The gas stream, upon contacting the hot surface of the silicon filament, will react to deposit silicon on the filament, thus increasing the diameter of the filament. The reaction of the trichlorosilane and hydrogen may be generally illustrated by the following simplified formula:



Gas flow through the quartz cylinder or reaction chamber is usually continued for several hours to increase the diameter of the filament, which may be one-fourth inch in diameter upon commencement of the deposition, to the diameter in excess of 1 inch. When the silicon rod has reached a desired diameter, flow is terminated and the rod is removed from the reaction chamber. Usually the material deposited on the silicon filament will be polycrystalline and therefore must be zone melted to produce a single crystalline material which is then sliced and further treated to produce transistors, diodes and the like. Alternatively, the polycrystalline rod may be melted in a crucible and a large single-crystal rod "pulled" from the melt by any of a variety of apparatus, such as a "Czochralski" puller.

The reaction vessel, usually quartz, within which the deposition is effected can assume various shapes. The vessel may be cylindrical in shape, and the filament mounted concentrically therewithin. The reaction vessel may also be dome shaped with the filaments being supported therewithin in a hairpin fashion as illustrated in U.S. Pat. No. 3,053,638.

For many applications, it would greatly decrease the time and expense of preparing final semiconductor components from silicon slices if the slices were all of a uniform diameter. Before the slices can be of uniform diameter, the rod from which they are cut must be of uniform diameter which requires that either the rod as removed from the reactor after deposition be uniform or that the rod after removal be ground to a uniform diameter. Using the deposition techniques, such as described above, it is virtually impossible to obtain rods having uniform diameter during the deposition process. More particularly, it is common that the silicon rods, after removal from the reactor have a center diameter which is about 1.25 times the diameter of the ends of the rod. The rod must then be ground to remove the excess material between the ends of the rod which not only increases the expense of producing semiconductor components but involves a waste of silicon material.

Since, for various reasons, the silicon rods formed in the deposition reactors must be of a minimum diameter, it is necessary to continue deposition for extended periods of time after the intermediate portion of the rods has reached the required diameter to permit those portions of the rod near the ends to reach the required size. Thus, more time is consumed and more reactants are required than would be necessary if the deposition could be conducted in an environment which

was conducive to the formation of a rod of uniform diameter throughout its length. The purity of the finished material would also be improved, as grinding of a rod having nonuniform diameter necessarily introduces contaminants which are undesired.

Silicon rods having nonuniform diameters are believed to be created in various of the deposition techniques presently being used because the gas from which the silicon is being deposited onto the substrate will change in composition as it traverses the length of the filament. The end of the filament positioned near the end of the reaction chamber through which the gas is admitted will be exposed to a gas having one concentration, while the end of the filament at the opposite end of the reactor will be exposed to a gas of a different composition, since a portion of the gas will have reacted intermediate the ends to deposit silicon along the length of the filament. Also, various countercurrents of the gas can be created in the reactor during the deposition causing the gases to be exposed for longer periods of time to certain portions of the filament than they are to other portions of the filament. In dome-shaped reaction vessel having a gas flow pattern which causes the gas to flow somewhat transverse to the longitudinal axis of the filaments supported therewithin, such as in U.S. Pat. No. 3,053,638, the filaments can grow with an irregular diameter since the concentration of reactants in the gas will vary along the length of the filament and around the circumference. Attempts have been made to avoid these problems by alternating the points at which the gas is introduced into the reaction chamber. For example, the gas for a given period of time will be introduced through one end of a cylindrical reactor and discharged through the other, after which the flow direction will be reversed for a like period of time. However, such technique does not solve the problem as the resulting silicon rods continue to be nonuniform in diameter requiring that they be ground before zone melting and slicing.

The method of the present invention may be generally described as an improved method for growing rods of material by depositing material onto an elongated filament from a gas being circulated over the filament. The method includes the steps of suspending the filament within the enclosure and directing the gas over the filament in a direction transverse to the longitudinal axis of the filament at a flow rate which is relatively uniform along the length of the filament. During passage of the gas over the filament, the filament is rotated so that all points along the surface will be uniformly exposed to the gas.

The apparatus of the present invention may be generally described as one for the growing of rods of material by deposition onto an elongated filament from gas being circulated over the filament which apparatus includes a reaction chamber having parallel, foraminous front and back panels through and between which a gas may flow. A manifold is positioned over a front foraminous panel for distributing a gas admitted into the manifold evenly over the surface of the front panel. Chucks extend into the reaction chamber in registering relationship so that an elongated filament can be received therebetween, and the chucks are rotatably mounted relative to the chamber so that the filament may be rotated with its longitudinal axis parallel to the front panel. Means are provided for rotating at least one of the chucks relative to the reaction chamber and thereby the filament. The apparatus preferably also includes a foraminous back panel which is also provided with a manifold so that the pressure across the back panel will be fairly uniform over the entire panel.

For a more complete understanding of the present invention, reference is here made to the drawings, in which:

FIG. 1 is a perspective view, partially cut away, of one embodiment of the apparatus of the present invention;

FIG. 2 is a cross-sectional, elevational view of the apparatus illustrated in FIG. 1;

FIG. 3 is a cross-sectional view taken along line 3—3 of FIG. 2;

FIG. 4 is a bottom plan view of the apparatus illustrated in FIGS. 1, 2 and 3; and

FIG. 5 is a partial, cross-sectional view of electrodes and a filament supported thereby.

The present invention provides a method of growing rods of a material such as silicon, which permits the growth of rods having uniform diameters throughout their length, thus eliminating the necessity of grinding rods of nonuniform diameter. Apparatus suitable for the deposition from a gas onto a filament for obtaining uniform diameter rods is illustrated in FIGS. 1-5, to which reference is here made.

With particular reference to FIG. 1, a reaction chamber generally indicated by the reference numeral 10 comprises parallel, foraminous front panels 11 and 12 which are joined at their lateral edges to side panels 13 and 14 and at their ends with top and bottom plates, only top plate 15 of which is illustrated in FIG. 1. Foraminous panels 11 and 12, side panels 13 and 14, top plate 15 and the bottom plate are preferably constructed of quartz. The apertures 16 in foraminous front and back panels 11 and 12 are preferably about one-eighth inch in diameter. Reaction chamber 10 is covered by a metal jacket, generally indicated by the reference numeral 17. Jacket 17, over foraminous front and back panels 11 and 14, forms manifolds 18 and 19. Manifolds 18 and 19 are provided with headers 21 and 22, respectively. As particularly illustrated in FIG. 3, manifolds 18 and 19 define with foraminous front panels 11 and 12, respectively, chambers 23 and 24.

Disposed between side plate 25 of jacket 17 and side plate 14 of reaction chamber 10 is a cooling coil 27, turns of which are also disposed between top plate 28 of jacket 17 and top plate 15 of reaction chamber 10. Coil 27 also extends along the opposite side and end of the apparatus. Specifically, coil 27 is disposed between side plate 26 of jacket 17 and side plate 13 of reactor 10, and as illustrated partially in FIG. 5, coil 27 also is disposed between bottom plate 29 of jacket 17 and bottom plate 20 of reaction chamber 10. Coil 27 is provided with inlet and outlet connectors 31 and 32, respectively, through which a cooling fluid, such as water, may be circulated for cooling side plates 13 and 14 and top plates 15 and 20 of reaction chamber 10.

As illustrated by FIG. 3, a plurality of elongated filaments 33 are supported within reaction chamber 10 parallel to the foraminous panels 11 and 12. The filaments 33 are supported within reaction chamber 10 by a set of top chucks 34, each of which is in registering alignment with one of the corresponding set of bottom chucks 35. With reference to FIG. 5, which illustrates one of the bottom chucks 35 and the manner in which it is supported within the apparatus of FIG. 1, each of the chucks 35, which may be constructed of graphite or the like, passes through bottom plate 29 of jacket 17 and bottom plate 20 of reaction chamber 10. Chucks 35 are rotatably mounted relative to jacket 17 and reaction chamber 10 by a circular bearing 36 which engages a teflon insulating sleeve 37 surrounding a portion of chuck 35. Each of the chucks 35 is provided with a square recess 38 which receives there within the square shaped end 39 of filament 33 so that rotation of chuck 35 will cause rotation of filament 33. The opposite end 41 of filament 33 is also provided with square shoulders for receipt within the square recess 42 of the upper registering chuck 34. Chucks 34 and 35 are spaced from each other a sufficient distance to leave sufficient room within recess 42 for filament 33 to expand in a longitudinal direction when heated. To permit rotation of graphite chucks 35, each has a filled teflon ring gear 43 press fit around sleeve 37.

As particularly illustrated in FIG. 4, each of the gear rings 43 mounted on chucks 35 is engaged with an adjacent gear 43 so that rotation of a driven gear wheel 44 will effect simultaneous rotation of gears 43 through a coupling gear wheel 45 which engages one of the gears 43 and drive gear 44. As illustrated in FIG. 2, drive gear 44 is driven by a conventional electric motor and transmission assembly 46.

To permit communication of a current through each of the filaments 33, each of the chucks 35 is provided with a radially enlarged portion 47 which forms a top beveled circumferential surface 48 and a bottom beveled circumferential surface 49. Engaging the radially extending portion 47 of gra-

phite chuck 35 is an electrode, generally indicated by the reference numeral 51. Electrode 51 comprises a top ring 52 having a radially relieved surface 53 at its inner diameter which mates with the top beveled circumferential surface 48. Electrode 51 also includes bottom ring 55 having a radially relieved surface 56 at its inner diameter which engages bottom beveled circumferential surface 49 of chuck 35. The top ring 52 and bottom ring 55 are maintained in abutment and supported by a mounting sleeve 57. Mounting sleeve 57 is provided with a conventional adjustment, not illustrated, for radially enlarging or compressing ring 57. Ring 57 is also provided with a coupling 58 for attachment of electrical cable 59 thereto.

As illustrated in FIG. 5, top circumferential surface 48 and radially relieved surface 53 define an included angle with the centerline of chuck 35 which is less than the included angle which bottom circumferential surface 49 and radially relieved surface 56 define with the same line. Thus, when rings 52 and 55 are radially compressed by mounting sleeve 57, the bottom ring will exert a greater resultant force upwardly on beveled surface 49 than ring 52 will exert downwardly on surface 48. The net upward force tends to maintain chuck 35 in the position illustrated in FIG. 5 and therefore assure that an effective gas seal is maintained between sleeve 37 and bottom plate 20 of reactor 10.

The structure of the top of the apparatus illustrated in FIG. 1 is identical to the structure of the bottom but, of course, oriented in a different direction. Specifically, each of the graphite chucks 34 which, like chucks 35, will be urged inwardly by the electrodes attached thereto is mounted to one of a series of ring gears 61, which, as illustrated in FIG. 2, intermesh identically to gears 43 and are driven by a coupling gear 62 mounted on shaft 63 which also carries coupling gear 45. Coupling gears 62 and 45 are of the same diameter as are gears 61 and 43, thus chucks 34 and 35 are rotated at the same speed. Each of the top chucks 34 is also, through an electrode 64, in electrical communication with one of the electrical cables 65, and the bottom chucks 35, through an electrode 51 is in contact with one of the cables 59. Each of the registering sets of chucks 34 and 35 is, through the respective electrodes 64 and 51, connected across a common electrical energy source so that substantially the same identical current flow will be established through each of the filaments 33 within the reaction chamber 10.

In operation, the apparatus described above may be used to practice the method of the present invention. More particularly, silicon filaments 33 may be positioned between graphite chucks 34 and 35 by removing the brace, not illustrated, supporting bottom electrodes 51 permitting graphite chuck 34 to be removed from the apparatus 10. Silicon filaments 33 are then inserted in the chucks 35 and the chucks reinserted into the apparatus. An operator can assure that the filaments 33 are engaged with the top chucks 34 by viewing filaments 33 as they are inserted through clear quartz sight glass 30 provided in side plate 25 of the jacket 17. Once the filaments are inserted, and the brace supporting the electrodes 51 is reconnected, reaction chamber 10 is flushed with nitrogen and subsequently with hydrogen gas admitted to manifold 18 through headers 21. Hydrogen entering headers 21 will be distributed within chamber 23 formed by manifold 18 after which it will flow through apertures 17 in foraminous panel 11, transverse to the filaments 33. The gas will then exit through apertures 16 in back foraminous panel 12, and will exit the chamber 24 formed by manifold 19 behind back panel 12 through header 22. After reaction chamber 10 has been flushed with hydrogen, the temperature of the filament 61 is elevated to the desired temperature, for example 1,100° C., if the filament is silicon, by impressing a potential between the top electrodes 64 and bottom electrodes 51. When the filaments 33 have reached the desired temperature, a gaseous stream containing reactants is circulated through headers 21. If the filament is silicon, for example, a mixture of trichlorosilane and hydrogen in a ratio of 5:95 (by volume) could be admitted to headers

21. In chamber 23, the gas is distributed over the foraminous panel 11 to maintain a fairly uniform pressure across the face of panel 11 so that the gas flow through the apertures 16 will be at a substantially uniform rate thus exposing each of the filaments 33 to approximately the same amount of gas along the entire length of the filaments 33. The gas, since it traverses reaction chamber 10 transverse to the longitudinal axis of each of the filaments 33 exposes the entire length of each of the filaments 33 to a gas of the same concentration, thus avoiding creation of segments of the filaments which are larger in diameter than those of other portions of the filament. The gas, which as described, may be trichlorosilane and hydrogen reacts in reaction chamber 10 to deposit silicon on filaments 33, and after reacting is discharged through foraminous back panel 12 into chamber 24 and out through headers 22. Due to the manifold 19, the gas pressure across the foraminous back panel 12 will be approximately equal at all points, thus avoiding channeling of gas flow through reaction chamber 10, further insuring that the entire lengths of each of the filaments 33 are exposed to approximate uniform quantities in a gas which is of a fairly constant concentration along the entire length of filament 33.

To insure that the filaments 33 grow in a uniform manner, the filaments are rotated by actuation of motor and transmission assembly 46, which rotates the elements at the rate of 1 revolution per minute or less for most application. Since the filaments may become quite plastic under the influence of the high temperatures, the gear assemblies provide an equal and simultaneous rotary force to both ends of filaments 33, thus minimizing any stress which may be created by rotation by imparting rotary force to only one end.

When the desired amount of material has been deposited on filament 33, which may be determined by inspection through sight glasses 30, the gas flow may be terminated and the rods of material removed in a reverse manner from which filaments 33 were installed.

With the present invention, it is possible to grow rods of uniform diameter throughout their length, thus avoiding the problems created by the necessity of grinding away excess material from rods of nonuniform length.

The invention may be used in the growing of various materials including either single crystal or polycrystalline silicon or germanium, or in forming epitaxial layers of various materials on substrates of yet another material. Various types of foraminous panels may be used to distribute the gas flow evenly, including a quartz frit material, and the expression "foraminous" is used to include various porous materials through which gas flow may be effected.

While rather specific terms have been used to describe an embodiment of the apparatus of the present invention and the method of the present invention, these expressions are not intended, nor would they be construed as limitation upon the invention as defined with the following claims.

What is claimed is:

1. An improved method for the chemical vapor deposition of a material onto an elongated hot filament from a decomposable gas passed over the steps of:

suspending the filament within an enclosure;

directing the gas into the enclosure and over the filament in a direction transverse to the longitudinal axis of said filament at a flow rate and in a manner which is substantially uniform along substantially the entire length of said filament;

withdrawing the resulting off gases from said enclosure in a direction transverse to the longitudinal axis of said filament at a flow rate and in a manner which is substantially uniform along substantially the entire length of said filament; and

rotating the filament so that all points along the surface of the filament will be uniformly exposed to the gas.

2. The method of claim 1 which is further characterized by the filament being suspended in a vertical position and the gas being directed transversely over said filaments being introduced through a first foraminous panel parallel to the longitudinal axis of the filament.

3. The method of claim 1 which is further characterized by the impartation of simultaneous rotary force to both ends of said filament thereby minimizing the creation of stresses in the filament.

4. The method of claim 1 wherein the gas flowing transversely over said filament is introduced through a first foraminous panel and after passing by said filament passes through a second foraminous panel.

5. An improved method for growing rods of semiconductor material by depositing said material onto an elongated hot filament from a decomposable gas circulated over the filament, which comprises the steps of:

introducing the gas into a first manifold to distribute the gas within the manifold and thereby maintain a substantially uniform pressure within the manifold,

passing the gases from said first manifold through a first foraminous panel positioned parallel to the longitudinal axis of the filament to thereby direct the gas transversely of the longitudinal axis of the filament, and

rotating the filament so that all points along the surface thereof will be uniformly exposed to the gas being introduced through the first foraminous panel, passing said gas after contact with said filament through a second foraminous panel also generally parallel to the longitudinal axis of said filament, and directing the gas passing through the second foraminous panel into a second manifold for maintaining a fairly uniform pressure across the second foraminous panel.

6. The method of claim 5 in which a simultaneous rotary force is imparted to both ends of said filament to minimize creation of stresses in said filament.

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