April 23, 1974 T. F. BRIODY 3,806,360

METHODS FOR HEATING AND/OR COATING ARTICLES

Original Filed Dec. 15, 1966 2 Sheets-Sheet 1

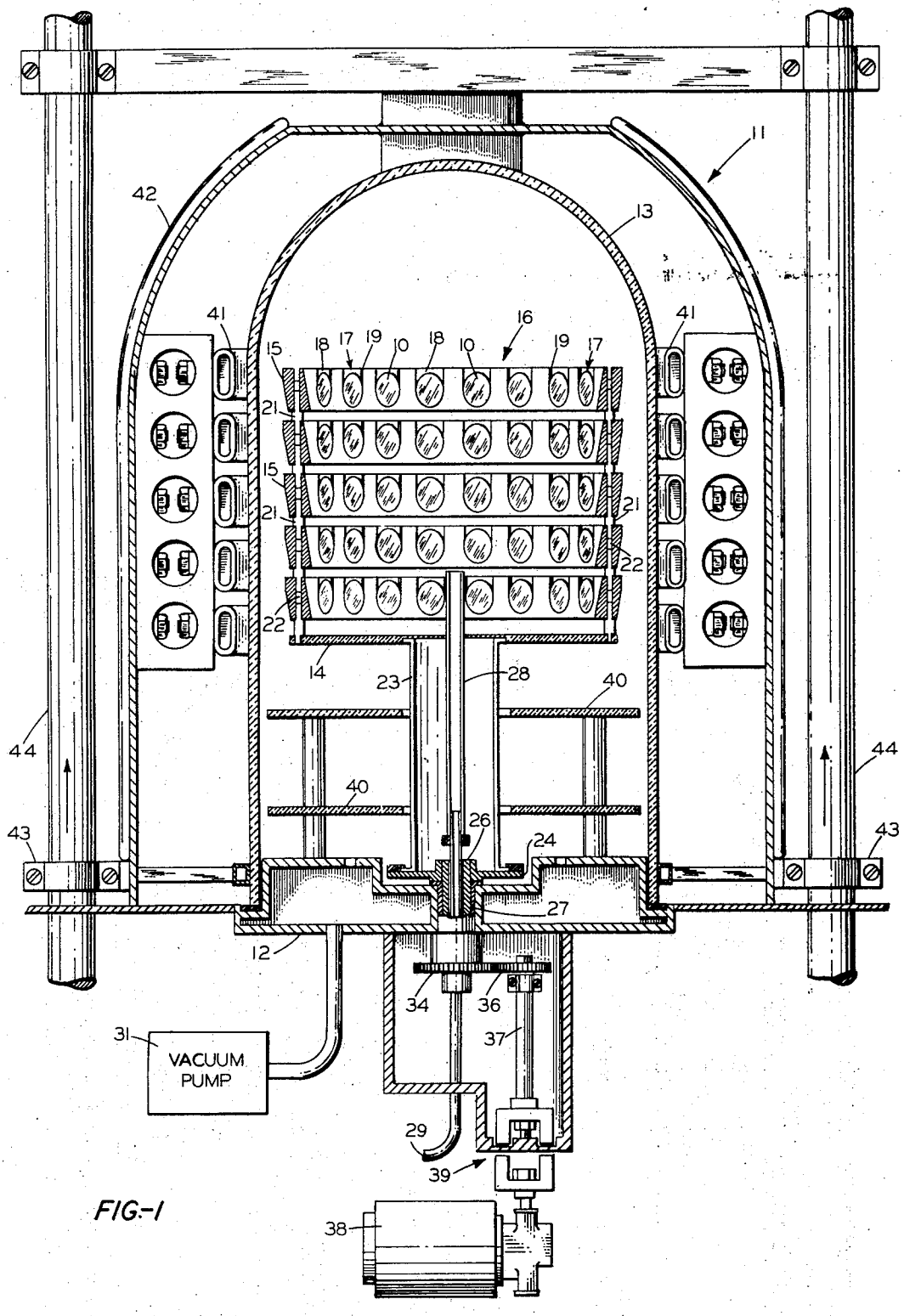


FIG-1

METHODS FOR HEATING AND/OR COATING ARTICLES

Original Filed Dec. 15, 1966 2 Sheets-Sheet 2

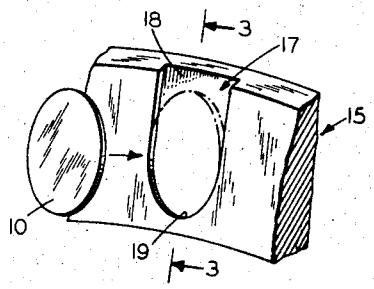


FIG.-2

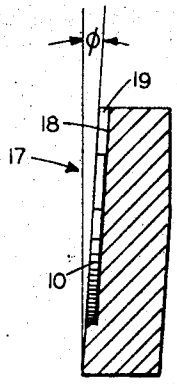


FIG.-3

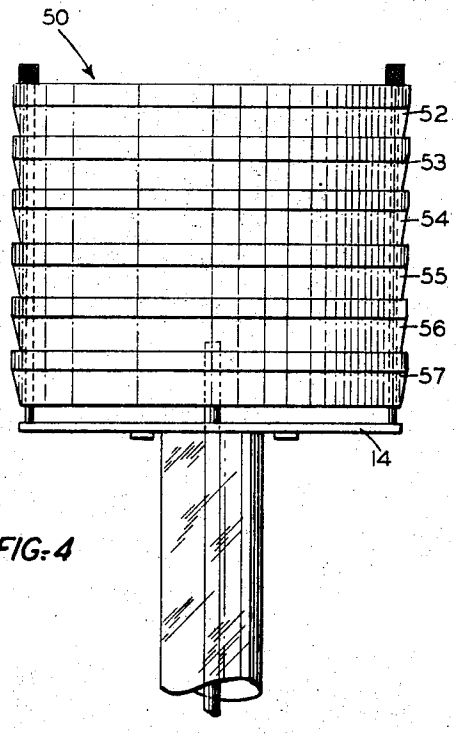


FIG.-4

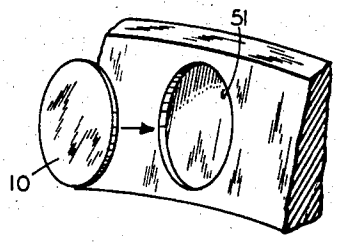


FIG.-5

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3,806,360

METHODS FOR HEATING AND/OR COATING ARTICLES

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Original application Dec. 15, 1966, Ser. No. 601,885, now Patent No. 3,659,552. Divided and this application Dec. 23, 1971, Ser. No. 211,465
Int. Cl. C23c 11/00; B05b 3/02
U.S. Cl. 117—201 18 Claims

ABSTRACT OF THE DISCLOSURE

A method of heating an article, such as a silicon slice, includes centrifugally forcing the article against a surface of a support of a heat-conducting material, and heating the support, while the article is so forced against the support, to heat the article by conduction through the support. In order to coat the article, a coating substance, such as a mixture of silicon tetrachloride and hydrogen gas, is impinged against the article.

This is a division of application Ser. No. 601,885 filed Dec. 15, 1966, now U.S. Pat. No. 3,659,552.

This invention relates to methods for heating and/or coating articles and, in particular, to methods for epitaxially depositing coatings of semiconductor material onto slices of such material. Accordingly, the general object of this invention is to provide new and improved methods of such character.

The production of epitaxially deposited semiconductor slices, such as silicon, requires special techniques to insure uniformity of deposited material. For example, in a commonly assigned copending application of James T. Hartman et al., Ser. No. 287,051, filed June 11, 1963, now abandoned methods and apparatus are described that teach the placing of silicon slices onto a horizontal, circular heating plate and the rotation of the plate about its principal axis. The plate is placed within a bell jar and heated inductively from within the bell jar by a pancake-shaped R.F. coil adjacent to the plate. A carrier gas (e.g., hydrogen) saturated with a halide of the semiconductor involved (e.g., silicon tetrachloride) is introduced from below and passes upwardly along the axis of the rotating heating plate through a central orifice thereof. Hartman et al. provide epitaxially deposited semiconductor slices of high quality and high uniformity. However, the number of slices that can be uniformly treated by the Hartman et al. apparatus is limited (for example, to the neighborhood of 20 one and one-quarter inch diameter slices).

Other commercial machines, capable of treating larger quantities (e.g., the neighborhood of 60 to 70 one and one-quarter inch diameter slices), generally suffer nonuniform epitaxial deposits among the slices.

In forming an epitaxial layer of silicon on silicon slices, it is imperative for large commercial production that the silicon be deposited uniformly thereon. In the past, epitaxial reactors have been incapable of handling large quantities of silicon slices while providing deposition in a uniform manner.

Therefore, it is an object of this invention to provide new and improved methods for heating and/or coating, in a uniform manner, large quantities of articles.

It is a specific object of this invention to provide novel methods for uniformly producing epitaxial deposits onto large numbers of semiconductor slices.

The foregoing and other objects are accomplished in accordance with certain features of the invention by providing a hollow cylindrical drum adapted to house a plurality of articles, such as semiconductor slices, within its inner surface. The drum is constructed of material to facilitate inductive heating thereof, such as graphite. The drum may have a plurality of recessed portions, within its inner surface, with flat surfaces inclined at a small acute angle (e.g., 3 degrees) with the principal axis for contact with flat slices. The recessed portions can be oriented circumferentially in one or more rows. The drum is rotated within a bell jar. A vapor for providing an epitaxial deposit of semiconductor material onto the slices may include a carrier gas, such as hydrogen, saturated with a halide of the semiconductor involved, as silicon tetrachloride.

Means are provided for rotating the hollow drum about its principal vertical axis during the deposition. An induction coil encloses the bell jar in order to heat the drum inductively. The apparatus is covered by a Faraday shield to prevent undesired electrostatic interference.

In accordance with other features of the invention, articles (such as semiconductor slices) can be heated and/or coated with a suitable material (such as by epitaxially applying deposits thereto) by centrifugally forcing the articles against the surface of a support that is inductively heated in order that the articles be conductively heated by the support. The articles can be placed against internal corresponding inclined recessed surfaces of a hollow rotatable drum; the drum rotated with its principal axis oriented in the vertical direction so that centrifugal force causes the articles to better contact the drum; the drum covered by a bell jar; and the drum inductively heated from without the bell jar to heat the articles by conduction. The bell jar is enclosed by a Faraday shield and raised and lowered along a guided path to permit an operator to remove the heated and/or coated articles from the drum and to replace those articles with other articles to be heated and/or coated. The vapor, such as hydrogen saturated with silicon tetrachloride, which is used for coating the articles, is directed along the principal axis toward the bell jar causing dispersion thereof in a manner to substantially uniformly affect the exposed surfaces of the slices.

Other objects, advantages and features of the invention will be apparent from the following detailed description when read in conjunction with the appended drawings in which:

FIG. 1 is an elevational view, partly in section, of deposition apparatus including a work holder in accordance with the invention;

FIG. 2 is a perspective view, in section, of a portion of the work holder shown in FIG. 1, in accordance with the specific embodiment of the invention, illustrating how a slice is oriented within a recessed portion therein;

FIG. 3 is a side view, in section, of the work holder taken along the line 3—3 of FIG. 2;

FIG. 4 is an elevational view of a different work holder suitable for use with the embodiment of the invention shown in FIG. 1; and

FIG. 5 is a perspective view, in section, of a recessed portion of the work holder shown in FIG. 4, illustrating how a slice fits therewithin.

GENERAL ARRANGEMENT

Referring now in detail to the drawings, and particularly to FIGS. 1, 2, and 3, the illustrative embodiment of the invention concerns methods and apparatus for heating and/or coating articles, including, for example, the epitaxial deposition of semiconductive coatings onto a plurality of silicon slices **10**—**10**, one of which is shown in FIG. 2. A typical slice may measure 1¼" diameter with a thickness of 5½ to 6½ mils. The apparatus includes a high capacity epitaxial reactor **11**, as shown in FIG. 1. The reactor **11** includes a base member or hous-

ing 12 upon which a bell jar 13 mates therewith. The bell jar 13 is constructed of inert, heat-resistant material, such as quartz. Within the bell jar 13, a rotatable horizontal base plate 14, preferably of quartz, holds a hollow drum-like work holder 16 having tapered or inclined recessed portions 17—17 (FIG. 2), each of which portions holds one of the slices 10. In the embodiment of FIG. 1, the drum 16 includes a plurality of removably mounted annular members, such as graphite rings 15—15, which are adapated to be heated inductively. As illustrated in FIG. 3, a recessed portion 17 includes a flat face 18, inclined at a small angle ϕ, preferably less than 15° and desirably in the neighbood of 3°, from the vertical. Each of the recesses 17—17 has a U-shaped wall 19 surrounding the face 18, forming pockets for holding the silicon slices 10—10. Spacers 21—21 are inserted within holes 22—22 disposed around the rims of the graphite rings 15 to couple the rings together as a single drum 16, as shown in FIG. 1.

The support plate 14 is affixed to one end of a quartz support tube 23. The other end of the support tube 23 is coupled to a flanged end 24 of a hollow shaft 26 that is adapted to rotate within a bearing 27 which provides an air tight seal within the housing 12. A quartz gas tube 28, concentric within the shaft 26, extends from below the center of the drum 16, along its principal axis, down through the tube 23 and hollow shaft 26 to an inlet 29 to permit gas to be introduced therethrough into the bell jar 13, of the reactor 11.

A vacuum pump 31 is coupled to the housing 12 in order to remove air from the bell jar 13.

A spur gear 34 is fitted onto the hollow shaft 26 so as to mesh with a second spur gear 36 affixed to a drive shaft 37. A motor 38, from without the reactor 11, is coupled to drive the shaft 37 by a magnetic coupler 39. Hence, the motor 38 causes the drum-like work holder 16 to rotate.

Several horizontal quartz members 40—40 are oriented between the base member 12 and the rotatable support plate 14 to reflect heat toward the drum 16 so that the temperature gradient throughout the drum 16 stays relatively uniform.

An induction heating coil 41, concentric with the graphite rings 15—15, circumferentially surrounds the outer periphery of the bell jar 13. Radio frequency (R.F.) energy is applied to the induction coil 41 so that the graphite rings 15 are heated by induction. Hence, the slices 10—10 held within the pockets 17—17 can be heated by conduction from the drum 16. By way of illustration, the graphite rings can be heated by R.F. energy in the neighborhood of 100 kilowatts at a frequency of 10 kilohertz. A Faraday shield 42, cooled by a fluid, such as water, surrounds the induction coil 41 to limit undesired electrostatic interference caused by outgoing radiation due to the energy provided to the induction coil 41. Also, the Faraday shield 42 serves to lessen radiating heat from the reactor so that surrounding work area is not uncomfortable.

The Faraday shield 42, induction coil 41, and bell jar 13 are joined together by a clamping member 43 so that they can be lowered or raised in unison along suitable guides 44—44.

STRUCTURE OF GRAPHITE RINGS 15—15

A perspective section of one graphite ring 15 is shown in FIG. 2. The recessed portion 17 includes a flat face 18 inclined at the small acute angle ϕ (e.g., 3 degrees) with the principal axis, with a U-shaped wall 19 rimming the face 18 to form a pocket of sufficient dimension to house a slice 10. As illustrated in FIG. 3, the face 18 is inclined in order to hold the slice 10 both at rest and when centrifugal force is radially applied.

ARRANGEMENT OF THE SLICE IN THE RINGS 15

In one embodiment, each of the rings 15—15 of eleven inch diameter has a single row of pockets 17 around its internal periphery for holding a plurality of slices 10—10, for example, twenty in number. A plurality of rings 15 can be stacked, one upon another, to form a single drum 16, in the manner shown in FIG. 1, so that, with five rings, one hundred slices can be treated at one time.

METHOD OF OPERATION

Initially, the bell jar 13, the induction coil 41, and the Faraday shield 42 are raised in unison along the guides 44—44. Each of the graphite rings 15—15, forming the drum 16, can be easily removed by an operator to enable easy insertion of semiconductor slices 10 within each of the recessed pockets 17—17 of the rings 15. The bottom graphite ring 15 fits onto spacers 21 coupled to the base plate 14. Each succeeding graphite ring 15 fits onto similar spacers 21—21 that are inserted into corresponding holes 22 of its preceding graphite ring.

In lieu of removing one or more of the graphite rings, inserting the slices into the rings, and reinstalling the graphite rings onto the base plate, an operator, instead, can place the silicon silces 10—10 into the pockets 17—17 of the graphite rings 15 directly without intermediately removing the rings.

The bell jar 13, the R.F. coil 41 and the Faraday shield 42 then are lowered into place, along the guides 44—44, so that the bell jar 13 makes intimate contact with the base member 12. The vacuum pump 31 is actuated so that the air within the bell jar 13 is removed. Inert gas, such as nitrogen or helium, is then introduced to atmospheric pressure. Radio frequency energy is then supplied to the induction coil 41 to inductively heat the graphite rings 15 while hydrogen is introduced into the bell jar 13, creating an environment suitable for epitaxial deposition. Meanwhile, suitable fluid, as water, flows through the Faraday shield 42 to limit the excessive heat which may radiate.

The motor 38 is started to rotate the graphite rings 15—15 as a unit through the magnetic coupler 39 and the gears 36 and 34. Rotation of the rings 15—15 is effected without moving the slices 10—10 parallel to the faces 18—18 of the rings 15—15. Also, the speed of rotation is sufficient to produce centrifugal force that causes the slices 10—10 to fixedly engage the faces 18—18 of the rings 15—15 so that the slices 10—10 have more intimate contact with said faces 18—18.

Typically, the motor speed is set to rotate the graphite rings 15 from about 10 to about 200 r.p.m.

A carrier gas, such as hydrogen, saturated with a halide of the semiconductor involved, such as a one percent mixture of silicon tetrachloride, is introduced into the inlet 29. The vapor is dispersed through the quartz gas tube 28, which does not rotate.

A complete deposition cycle takes approximately two hours to heat, to stabilize, and to deposit. The deposition time to produce epitaxial deposits (typically from 7 to 14 microns) is relatively short; the rate of deposition preferably is one micron per minute.

ALTERNATE ARRANGEMENT

FIG. 4 shows an alternate embodiment of a workholder adapted to be inductively heated—hereinafter termed "susceptor." The susceptor 50 is an integral unit and is a hollow drum having pockets 51 therewithin for holding a plurality of semiconductor slices 10. The pockets 51 are oriented in a plurality of rows 52 to 57. Each of the rows 52 through 57 contains equally circumferentially spaced pockets 51 about the internal periphery of the susceptor 50. For example, in FIG. 4, six rows of twenty pockets each hold a total of 120 slices for treatment at one time.

FIG. 5 illustrates a circular pocket for housing a slice 10. The circular pocket 51 is one of several possible configurations. Other suitable choices, such as the U-shaped pocket 17 shown in FIG. 2, can be used. The pocket 51, shown in FIG. 5, in a similar manner, is inclined at a

small acute angle with the principal axis, such as three degrees.

The susceptor **50** can be loaded and unloaded with slices **10** without removing the susceptor from its base plate **14**.

A susceptor that was constructed with pockets inclined at a 15° angle produced, upon testing, deposits which were not so uniform as the susceptors having 3° inclined pockets.

In a preferred commercial operation, two epitaxial reactors **11** are operated side by side with common electrical control apparatus so that, when slices are treated in one unit, the other unit can be loaded and unloaded.

While several specific embodiments of the invention have been described in detail hereinabove, it will be obvious that various modifications can be made from the specific details described without departing from the spirit and scope of the invention. In particular, while the invention is particularly advantageous for use in the epitaxial deposition of coatings on the semiconductor slices, or in vapor plating articles, the invention may be practiced for depositing coatings onto articles and/or heating articles in general.

As used in the claims, the term "drum" is to be construed broadly to include both the one-piece drum and the multipiece drum as taught herein.

What is claimed is:

1. A method of coating an aritcle with a substance comprising:
- placing a flat side of said article against an internal surface of a rotatable supporting drum;
- rotating said drum without moving the article parallel to the internal surface of said drum, and rotating said drum about an axis at a speed such that centrifugal force causes said article to fixedly engage the surface of said drum and to have more intimate contact with said surface; and
- impinging said coating substance against an exposed surface of said article, the centrifugal force also causing said substance, upon contact with said article, to more intimately adhere to said exposed surface of said article.

2. The method of claim **1**, further comprising heating said article so that the article has a greater affinity, during deposition, for said coating substance.

3. The method of claim **2**, wherein the heating of said article is caused by inductively heating said supporting drum to heat said article by conduction through said drum.

4. A method of coating an article with a substance, comprising:
- placing said article against the internal surface of a hollow, rotatable drum of heat-conductive material;
- introducing said substance, in vapor form, to an exposed surface of said article;
- rotating said drum without moving the article parallel to the internal surface of said drum, and rotating said drum about its principal axis at a speed so that centrifugal force causes said article to fixedly engage the surface of said drum and to have more intimate contact with said drum surface; and
- heating said drum during rotation thereof to heat said article by conduction through said drum.

5. The method of claim **4**, further comprising orienting said principal axis in the vertical direction.

6. The method of claim **5**, for coating an article having a flat surface, wherein the article is placed against the internal surface of the drum and the surface is inclined at an angle to the vertical.

7. The method of claim **4**, wherein the substance, in vapor form, is introduced to said exposed surface of said article by initially directing a flow of the vapor substance along said principal axis of said drum.

8. The method of claim **4**, wherein:
- said drum is covered with a bell jar;
- said drum is inductively heated from without the bell jar to heat said article by conduction through said drum; and
- the substance, in vapor form, is directed along said principal axis toward said bell jar to introduce said substance to said exposed surface of said article.

9. A method of coating a plurality of articles with a substance, comprising:
- placing said articles against the internal surface of a vertically positioned, hollow, rotatable drum of heat conductive material;
- covering said drum with a bell jar;
- rotating said drum without moving the articles parallel to the internal surface of said drum, and rotating said drum about its principal axis at a speed so that centrifugal force causes said articles to have more intimate contact with said drum;
- directing a flow of the substance, in vapor form, along said principal axis toward said bell jar to introduce the substance to exposed surfaces of said articles; and
- inductively heating said drum from without said bell jar to heat said articles by conduction through said drum.

10. A method of epitaxially depositing a coating of semiconductive material onto a slice of like material, comprising:
- placing said slice against a surface of a rotatable supporting drum of heat-conductive material;
- introducing a carrier gas containing a heat-decomposable compound of the semiconductor involved to an exposed surface of said article;
- rotating said supporting drum without moving the slice parallel to the internal surface of said drum, and rotating said drum about an axis at a speed such that centrifugal force causes said slice to have more intimate contact with said surface of said drum; and
- heating said drum during rotation thereof to heat said slice by conduction through said drum.

11. The method of claim **10**, wherein said heating of said drum is caused by induction.

12. A method of epitaxially depositing a silicon coating onto a silicon slice, comprising:
- placing said slice against an inclined suface of a rotatable supporting drum of heat-conductive material;
- introducing a carrier gas of hydrogen saturated with silicon tetrachloride to an exposed surface of said slice;
- rotating said drum without moving the slice parallel to the inclined surface of said drum, and rotating said drum about an axis at a speed such that a major component of centrifugal force causes said slice to have more intimate contact with said surface of said drum; and
- inductively heating said drum during rotation thereof to heat said slice by conduction through said support.

13. A method of epitaxially depositing coatings of semiconductive material onto slices of like material, comprising:
- placing said slice against the internal surface of a vertically positioned, hollow, rotatable drum of heat-conductive material;
- covering said drum with a bell jar;
- rotating said drum without moving the slices parallel to the internal surface of said drum, and rotating said drum about its principal axis at a speed so that centrifugal force causes said slice to have more intimate contact with said drum;
- directing a carrier gas containing a heat-decomposable compound of the semiconductor involved along said principal axis and toward said bell jar, to cause dispersion of said gas in a manner to substantially equally affect exposed surfaces of said slices; and
- heating said drum during rotation thereof to heat said slices by conduction through said drum.

14. The method of claim 13, wherein the slices are placed against the internal surface of the drum with their flat surfaces inclined at an angle to the vertical and abutting inclined flat walls of the pockets.

15. The method of claim 13, wherein said drum is inductively heated from without the bell jar.

16. A method for vapor plating articles, comprising the steps of:

positioning the articles in generally radially, inwardly facing article engaging portions of an electrically conductive support that is hermetically sealed within a chamber;

rotating said support without moving the articles parallel to said portions of said support, said rotation being without said chamber whereby articles positioned against said portions are at least partially held in place by centrifugal force;

inducting heating current in said support with an electric coil to heat the articles; and

directing vapor over the heated articles through vapor outlet means positioned within said chamber.

17. The method of claim 16, wherein the support is rotated about a vertical axis and the articles are slices

that are placed at an angle of approximately 3° to the vertical.

18. The method of claim 16, wherein the support is rotated at a speed in the range of about 10 to about 200 r.p.m.

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U.S. Cl. X.R.

117—101, 106 R, 106 A

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,806,360 Dated April 23, 1974

Inventor(s) THOMAS F. BRIODY

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Claim 16, line 15 (Amended claim 41, line 7)

"without" should be --within--.

Signed and sealed this 22nd day of October 1974.

(SEAL)
Attest:

McCOY M. GIBSON JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents