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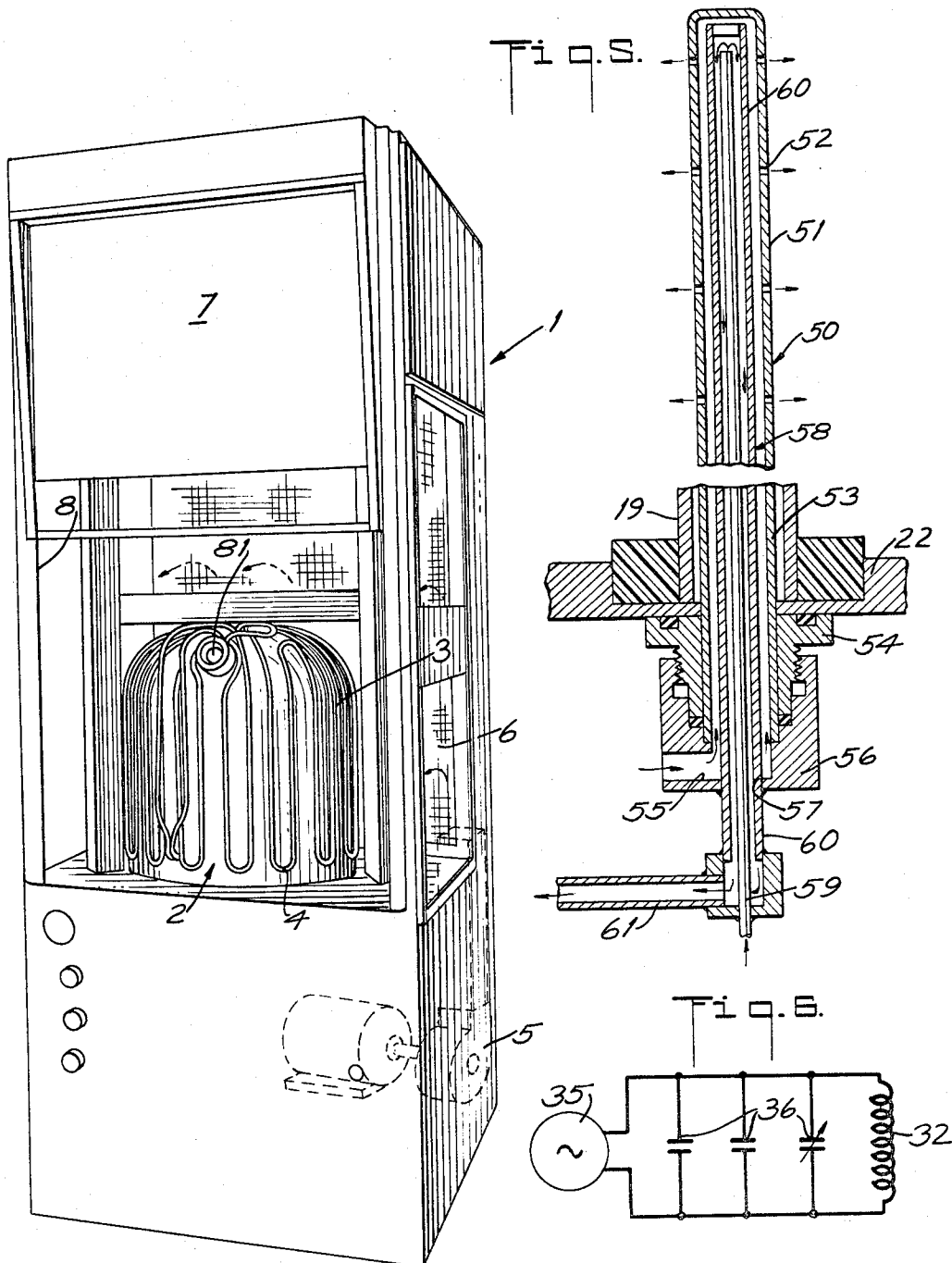
E. R. CAPITA

3,384,049

VAPOR DEPOSITION APPARATUS INCLUDING CENTRIFUGAL
FORCE SUBSTRATE-HOLDING MEANS

Filed Oct. 27, 1966

3 Sheets-Sheet 1



INVENTOR
EMIL R. CAPITA

BY
Nathan N. Holland
ATTORNEY

May 21, 1968

E. R. CAPITA
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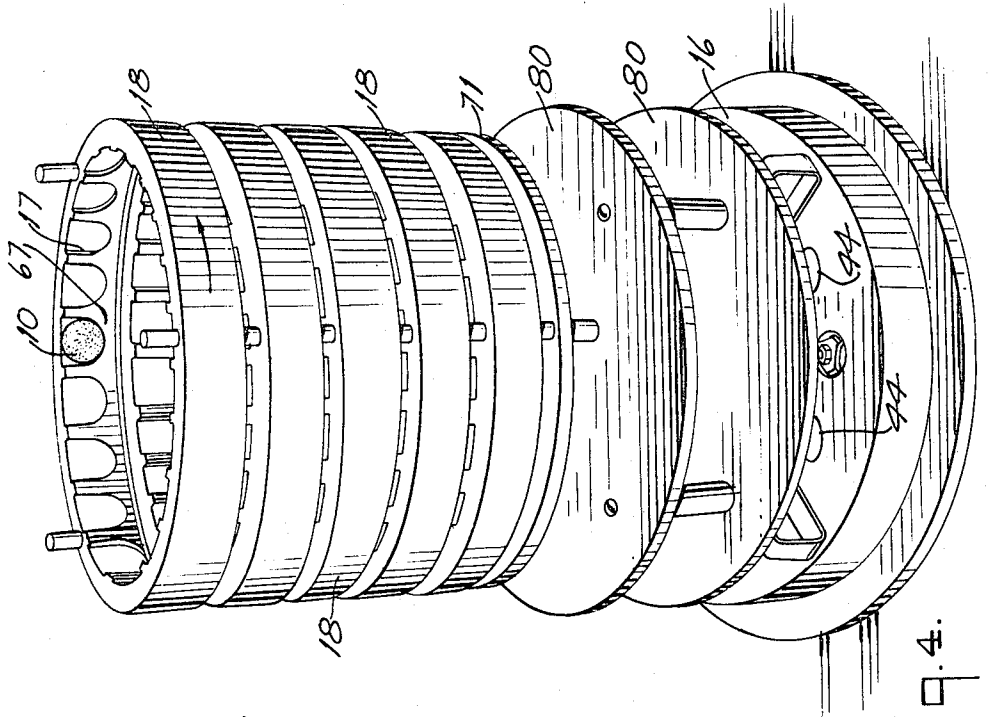


Fig. 4.

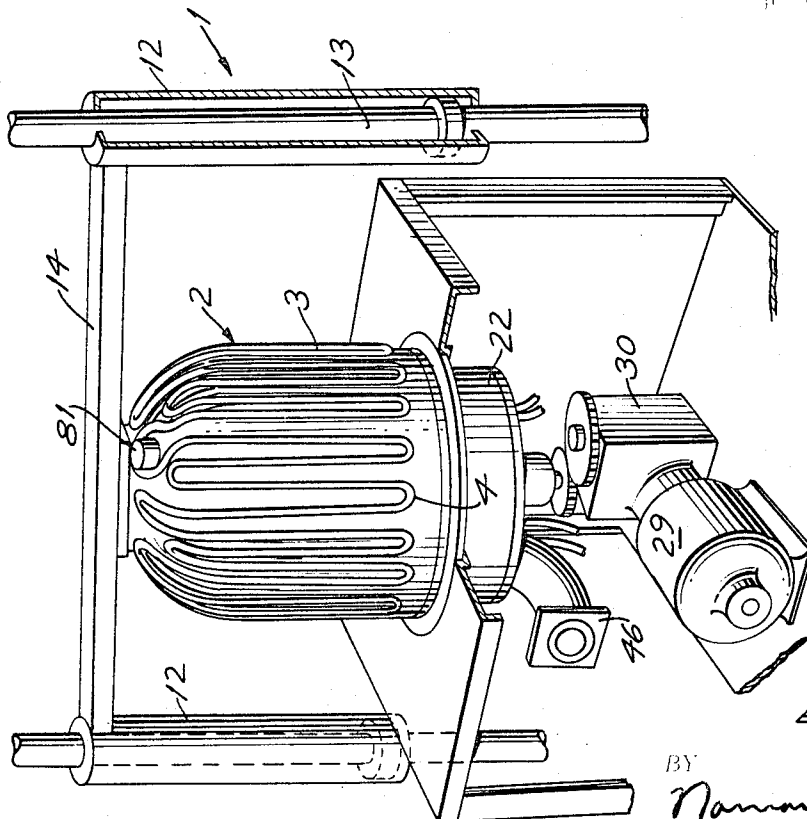


Fig. 2.

INVENTOR
EMIL R. CAPITA

BY
Namont Walburn
ATTORNEY

May 21, 1968

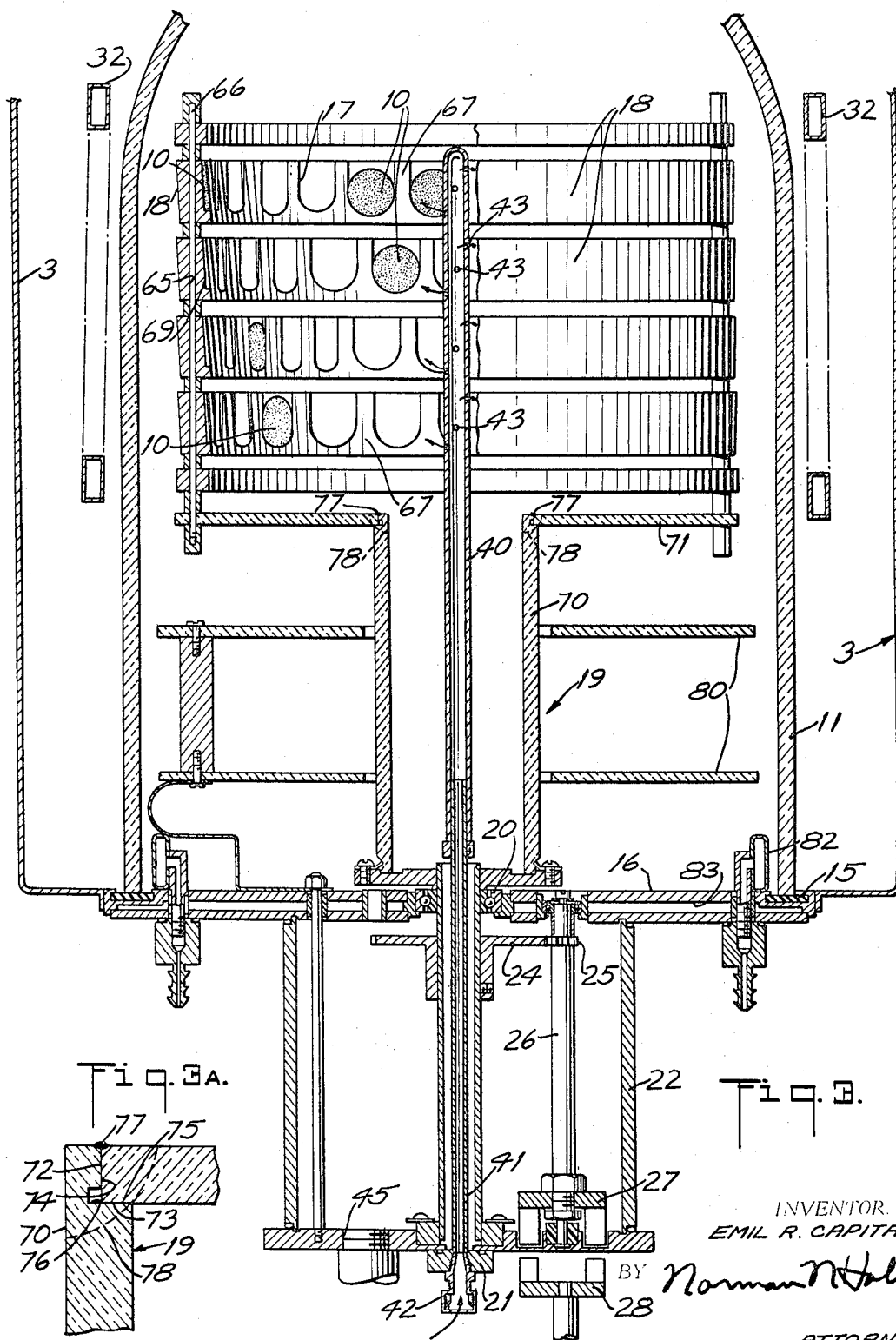
E. R. CAPITA

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3 Sheets-Sheet 3



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VAPOR DEPOSITION APPARATUS INCLUDING CENTRIFUGAL FORCE SUBSTRATE-HOLDING MEANS

Emil R. Capita, 7020 Hudson Blvd.,
North Bergen, N.J. 07047
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The present invention relates to a means for vapor coating or plating and more particularly to an improved vapor coating furnace of the type for forming a coating of a precisely controlled and uniform thickness. The present invention is particularly directed to providing an improved coating thickness control and better coating uniformity and an improved and more useful structure which both simplifies the operation and increases the output capacity.

The present invention represents an improvement in vapor coating apparatus wherein the uniformity of the coating thickness, an improved depth control, and a high degree of purity of the coating are obtained at increased output rates which have not been heretofore achieved. In particular, the apparatus of the present invention is an improvement upon the vapor coating method and apparatus of my United States Patent No. 3,233,578 dated Feb. 8, 1966.

Known vapor deposition coating processes reduce or decompose a volatile compound on the heated surface of the object to be coated. The hydrogen-reduction process, for example, passes hydrogen over a heated liquid metal halide to provide a resulting mixture of hydrogen and the metal halide vapor. The mixture passes into a furnace coating chamber having a controlled pressure where it reacts at the heated surface of the object to be coated and deposits an adherent coating of the non-volatile reaction product.

A significant application for vapor deposition is in applying silicon coatings to silicon discs or slices such as are used in the manufacture of transistors. The silicon slice comprises the N-type and the reaction product coating on the slice comprises the N-type. The coating purity, the uniformity of the coating thickness, and the control of the coating depth are of critical importance in such transistors.

The improved apparatus of the present invention obtains these results in a relatively high capacity manufacturing operation. The apparatus and method will now be described for use in a silicon coating operation although it is to be understood that it is not limited to such an operation and may be used with the formation of other coatings on other objects in a similar way.

Accordingly, an object of the present invention is to provide an improved means for vapor plating.

Another object of the present invention is to provide an improved susceptor means for vapor plating apparatus.

Another object of the present invention is to provide an improved apparatus for vapor plating combining improved coating control and contamination elimination with a relatively high capacity operation.

Another object of the present invention is to provide an improved vapor plating furnace having a multiple susceptor ring support for higher capacity operation.

Other and further objects of the invention will be obvious upon an understanding of the illustrative embodiment about to be described, or will be indicated in the appended claims, and various advantages not referred to herein will occur to one skilled in the art upon employment of the invention in practice.

A preferred embodiment of the invention has been chosen for purposes of illustration and description and is

shown in the accompanying drawings, forming a part of the specification, wherein:

FIG. 1 is a perspective view of a preferred embodiment of a vapor coating or plating furnace in accordance with the present invention;

FIG. 2 is a detailed perspective view illustrating a preferred embodiment of the furnace drive system and the cover lifting apparatus;

FIG. 3 is a detailed vertical sectional view of the coating furnace including the rotating susceptor rings;

FIG. 3A is an enlarged fragmentary sectional view of the susceptor support shaft;

FIG. 4 is a detailed perspective view illustrating a preferred embodiment of the susceptor ring mounting arrangement for supporting the slices during the coating operation;

FIG. 5 is a detailed vertical sectional view illustrating a preferred embodiment of a water cooled vapor feed tube; and

FIG. 6 is a schematic diagram of the induction heating circuit.

As already discussed above, the basic principle involved in the furnace is the simultaneous exposure of small discs or slices of silicon or other material to an induction heating field and to a gas and vapor mixture whereby the discs are heated and the vapor is decomposed resulting in the formation of a coating of the non-volatile reaction product on each of the slices.

FIG. 1 illustrates the improved furnace cabinet 1 in accordance with the invention wherein the process is carried out in an atmosphere of extreme purity and at a relatively high production rate and where a relatively large number of slices are simultaneously handled using an extremely simple and easily manipulating slice loading, and removal operation. The actual vapor coating of the slices is done within a furnace enclosure 2 having a Faraday shield 3 and having a series of water cooling conduits 4 positioned on the shield 3 surface for maintaining the shield at a suitably low temperature during the induction heating of the slices and arranged to be electrically open circuited to prevent induced current flow. This enclosure 2 is mounted within the air-tight cabinet 1 which preferably includes an air blowing system with a blower 5 and filters 6 whereby the entry of unfiltered outside air into the cabinet 1 is prevented whether or not the cabinet door 7 is open by a continuous discharge of filtered air from the blower 5 and through the filters 6 at the rear of the cabinet 1 across the enclosure 2 and out of the front opening 8 which is normally closed by the sliding door 7 during furnace operation.

The loading and unloading of the furnace enclosure 2 to remove coated slices 10 (FIG. 3) and to insert uncoated slices is facilitated by a preferred lift means for simultaneously raising the Faraday shield 3 and a quartz furnace hood illustrated at 11 in FIG. 3 and mounted within shield 3 by means of hydraulic lift cylinders 12 mounted on stationary piston and support rods 13 and positioned to raise the shield 3 and the cover 11 with the cross beam 14.

FIG. 3 illustrates the details of the preferred vapor coating furnace enclosure 2. The furnace enclosure 2 comprises a quartz cover 11 suitable for high temperature operation and positioned to detachably engage a sealing gasket 15 on a stainless steel water cooled furnace base 16. The slices 10 which are to be heated and subjected to the gas and vapor mixture are preferably mounted in slots 17 formed on the inner wall of a plurality of susceptor rings 18 which will be further described below. A stack of the susceptor rings 18 loaded with slices 10 are removably mounted on a rotatable and hollow center support shaft 19 formed of heat resistant quartz material and rotatably mounted in a bearing 20 in the base and

an additional bearing 21 in the lower exhaust chamber 22 which depends from the base 16. Continuous rotation of the circular susceptor rings 18 is provided for during the vapor coating process by rotating the support shaft 19 through the intermediation of drive gears 24 and 25 and vertical drive shaft 26 mounted wholly within the enclosed exhaust chamber 22 and having one section 27 of a magnetic coupling attached to its lower end. The section 27 is engaged by a corresponding magnetic coupling section 28 positioned outwardly of the exhaust chamber 22 and driven at the desired speed by drive motor 29 and a speed reduction means 30 as illustrated in FIG. 2.

The heating of the rotating slices 10 is preferably done by an induction heating system for inducing heating currents in the graphite or molybdenum susceptor rings 18 and includes a water cooled heating coil 32 formed generally concentrically with the axis of support shaft 19. An alternating heating current is passed through this coil having a frequency of about 10,000 cycles per second, however, a frequency range of from 10 to 450 kc. may be used. FIG. 3 illustrates a preferred embodiment of the heating current circuit including a high-frequency alternator 35 coupled to a tank circuit where the heating coil 32 forms the inductance in combination with a series of capacitors 36 coupled in parallel with the coil 32 and conveniently mounted in the upper portion of the cabinet 1.

A coating furnace of the type of the invention using about four susceptor rings 18 to mount about 100 slices may employ a power input of 100 kw. with the tank power factor being about 8% and with about 1200 kva. in the tank resulting in a heating input into the susceptors of about 60 kw. This provides a temperature of about 1250° C. for the rotating slices.

The coil 32 is mounted between the shield 3 and the furnace hood 11 and preferably is connected to the shield 3 and hood 11 to permit the shield 3, the hood 11 and the coil 32 is to be raised as a unit by the lift cylinders 12.

The coating gas vapor mixture is preferably directed radially outwardly against the rotating slices 10 from a central vapor distributing feed pipe 40 passing through the hollow support shaft 19 and fixedly attached to the exhaust chamber 22 at its lower end 41 and coupled to a source of the gas vapor mixture being used through an inlet 42. Radially and axially spaced vapor outlets 43 in the upper portion of the feed pipe 40 direct the gas vapor mixture outwardly and over the moving slices 10. The gas vapor mixture which is not applied as a coating is drawn generally downwardly within the quartz 11 and through apertures 44 in base 16 (FIG. 4) to an exhaust outlet 45 provided at the bottom of the exhaust chamber 22 and is further removed through an exhaust pipe as illustrated at 46 in FIG. 2.

For certain high speed vapor coating operations which are best performed at extremely high temperatures, a water cooled vapor distributing feed pipe is preferred. A preferred embodiment of a water cooled vapor feed pipe 50 is illustrated in FIG. 5. This pipe 50 is seen to include an outer pipe 51 with the vapor directing outlets 52 and which has its lower end 53 fixedly attached to a mounting flange 54 on the base of the exhaust chamber 22. The gas vapor mixture is admitted to the interior of the outer pipe 51 through an inlet 55 in coupling 56 which is threadedly attached to the pipe mounting flange 54. The coupling 56 has an aperture 57 for receiving a jacketed water cooling tube 58 including a central water feed tube 59 and a surrounding water jacket 60 communicating with a water outlet pipe 61.

The preferred form of the slice 10 supporting susceptor 18 is illustrated in FIGS. 3 and 4. Each susceptor 18 includes a series of spaced mounting holes 65 for receiving molybdenum support rods 66 to releasably mount the susceptor rings 18 on the rotating support shaft 19 between dummy conductive end rings 68 which im-

prove the uniformity of the inductive heating effect. The inner walls 67 of each susceptor ring 18 are provided with adjacent open topped slots 17 each of which is proportioned to receive a slice 10 for the coating operation. A slight upward flare in the slot inner surface 69 tends to hold each slice 10 in position as the slices 10 are loaded into the susceptor rings 18 preferably by a vacuum tweezer or similar tool while the susceptor rings are in place on the support shaft 19. Thereafter it will be seen that the generally vertical position of each slice 10 within its positioning slot 17 on the susceptor ring wall 67 acts to hold each slice 10 in position during the vapor application through the centrifugal force resulting from the rotation of the susceptor rings 18 during the vapor plating. As already indicated these susceptor rings 18 rotate continuously as the gas vapor mixture is directed over them and a typical rotational speed for these susceptor rings 18 is about 120 r.p.m.

In order to assure the uniform coating of each of the slices 10 during the coating operation, it is important that the rotational path of each slice 10 be concentric with respect to the centrally located vapor distributing pipes as well as with respect to the center line of the heating coil. To obtain this result, it is necessary that the temperature resistant support shaft 19 for the susceptor rings 18 itself be formed to provide a concentric rotation of the slice supporting slots 68 around its own axis.

A unique method of attaching the vertical hollow cylindrical portion 70 of this shaft 9 to the circular ring support 71 is illustrated in FIGS. 3 and 3A. Since both the vertical portion 70 of the base and the circular support 71 are subjected to relatively high temperatures, it is preferable that they be formed of a heat and corrosion resistant material such as quartz. This being the case, it has been found difficult to provide an accurate means for attaching a cylindrical quartz portion 70 of the circular support 71 and to return the necessary alignment of these two portions to obtain the above described concentric rotation. The attaching means illustrated in these figures has solved this problem and provides an effective attachment between the two portions 70 and 71 which is formed by conventional grinding or shaping machinery on the quartz objects.

Precision turning machinery is used to form a precise bore 72 in the circular support 71 as well as a precision mounting surface 73 on the lower surface of the support 71 (FIG. 3A).

Cooperating and precisely located mounting surface 74 and 75 are similarly cut on the cylinder 70 and in addition a cut-out 76 is formed to prevent any disalignment from occurring in this critical area of the surfaces 72-75. The result provides a precise mounting of the quartz cylinder 70 with respect to the circular support 71 even though the quartz shaping instruments are limited as described above to precision cutting machinery capable of the rotary cutting steps as indicated. The ring 71 is preferably spot welded to the tube 70 as, for example, with four spot welds 77. In order to avoid distortion and excessive heat transfer during the welding, a slot 78 is cut through the tube 70 and ring 71 on both sides of the welds 77 by a saw or otherwise.

Preferably, quartz heat shields 80 are positioned between the susceptors 18 and the base 16.

A typical operating cycle for the above described coating apparatus of FIGS. 1-6 will now be described.

With the quartz jar 11 removed from the base 16 by the cylinders 12, the silicon slices 10 which are to be coated are first carefully placed on the susceptors 18 preferably while the susceptors 18 are in position on the support shaft 19. The quartz jar 11 and shield 3 are now lowered onto the seal 15 on the base 16. A vacuum is now drawn in the air-tight chamber surrounding the slices 10 of the order of about one micron. The chamber is next purged with hydrogen by passing it through the chamber between the inlet 42 and the outlet 46.

The high frequency voltage source is now connected to the induction heating coil 32. The induction heating coil 32 now heats the molybdenum or graphite susceptors 18 and the silicon slices 10 arranged around inner surfaces 67 of the susceptors 18. The temperature of the slices 1 is observed by means of an optical pyrometer through a viewing surface 81 provided on the jar 11. During the heating, the susceptors 18 are rotated to insure a uniform heating of the silicon slices 10 and pure hydrogen is passed through the chamber between feed pipe 40 and outlet 46. A continuous supply of coolant is passed through the coil 32 and the cooling channels 82 and 83 in the base 16 and the conduits 4 on the Faraday shield 3 during the operation of the induction heating coil 32. When the silicon slices 10 have reached a temperature of about between 1190 and 1450 degrees C. the vapor plating is commenced by the admission of hydrogen gas containing silicon tetrachloride vapor through feed tube 40. This mixture flows outwardly and over the heated slices 10 on the rotating susceptors 18 in a uniform pattern. When the mixture of hydrogen and silicon tetrachloride vapor contacts the heated surfaces of the slices 10, it reacts at the heated surface to deposit an adherent coating of silicon on each of the slices 10.

In a typical silicon coating operation, the pressure in the chamber at the slices 10 is maintained at about 1 to 2 p.s.i. above atmospheric pressure and the spent gases flow downwardly through the hood 11 to an exhaust zone within the base 22 adjacent to outlet 45 which is kept at about atmospheric pressure by the continuous evacuation of the spent gases through the exhaust outlet 46 to the atmosphere. This provides for a continuous flow of the mixture past the heated and moving slices 10. The thickness of the silicon coating on the discs is controlled by controlling the pressure and the flow rates of an incoming mixture as well as the proportions of hydrogen and silicon tetrachloride in the mixture and by continuing the flow of mixture for a predetermined time. When this time period has elapsed, the supply of the vapor mixture and the current to the heating coil 32 is cut off and the chamber is again purged with nitrogen or argon or another inert gas and is opened by the removal of the jar 11 to provide access to the coated discs 10 after a suitable cooling period.

It will be seen that significant improvements have been provided in a means for a high output vapor plating of objects such as silicon slices and other small objects. The elements of the improved furnace are combined in a particularly effective and novel manner to provide for improved coating characteristics combined with a high capacity overall coating operation and wherein the loading and unloading steps, which are an important part of the overall process, are made more efficient and more easily performed.

As various changes may be made in the form, construction and arrangement of the parts herein without departing from the spirit and scope of the invention and without sacrificing any of its advantages, it is to be understood that all matter herein is to be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim:

1. Apparatus for vapor plating articles comprising the combination of a hermetically sealed chamber, an electrically conductive article support, means for rotatably mounting said article support within said enclosure, said support having a generally radially inwardly facing article engaging portion, means for rotating said article support whereby articles positioned against said portion are at least partially held in place by centrifugal force, an electric coil positioned for inducing heating current in said support, and vapor outlet means positioned within said enclosure for directing vapor over the heated articles.

2. The apparatus as claimed in claim 1 in which said

support comprises a plurality of rings and the said portion comprises axially aligned inwardly facing surfaces on the rings.

3. The apparatus as claimed in claim 1 in which said support comprises a graphite ring having article positioning slots in said portion.

4. The apparatus as claimed in claim 1 which further comprises electrically conductive end members mounted on the opposite sides of said article support for movement therewith for increasing the uniformity of the induced heating of the support.

5. The apparatus as claimed in claim 1 in which said means for rotating said article support comprises a hollow quartz shaft member, a flat radially extending quartz flange member, said shaft and said flange member being attached at circularly cut portions each having a right angled corner shape in cross section, and the right angled corner of one member being cut away.

6. Apparatus for vapor plating articles comprising the combination of a hermetically sealed chamber, a plurality of electrically conductive ring-like article supports having generally axially aligned and inwardly directed article engaging portions, means for rotatably mounting said article supports on a generally vertical axis within said enclosure, drive means for rotating said article support whereby articles positioned against said portion are at least partially held in place by centrifugal force, an electric coil positioned for inducing heating currents in said supports, and vapor outlet means positioned within said enclosure for directing vapor over the heated articles.

7. The apparatus as claimed in claim 6 which further comprises electrically conductive ring members positioned above and below said article supports.

8. The apparatus as claimed in claim 6 in which said enclosure comprises an inner heat resistant hood, an outer Faraday shield, and said electric coil being positioned intermediate said hood and said shield.

9. The apparatus as claimed in claim 6 in which said enclosure comprises an inner heat resistant hood, an outer Faraday shield, and lift means for simultaneously raising the hood and shield.

10. The apparatus as claimed in claim 9 in which said lift means comprises movable cylinders and stationary pistons.

11. Apparatus for vapor plating articles comprising the combination of a hermetically sealed chamber, an annular electrically conductive article support having a generally axially aligned and inwardly directed article engaging portion, means for rotatably mounting said article support within said enclosure, means for rotating said article support whereby articles positioned against said portion are at least partially held in place by centrifugal force, an electric coil positioned for inducing a heating current in said support, and a vapor feed pipe positioned generally axially of said support having radially directed vapor outlets.

12. The apparatus as claimed in claim 11 which further comprises fluid cooling means for said vapor feed pipe.

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MORRIS KAPLAN, *Primary Examiner*.