Oct. 29, 1968 E. R. CAPITA 3,407,783

VAPOR DEPOSITION APPARATUS

Filed Aug. 31, 1964 3 Sheets-Sheet 1

Fig. 1.

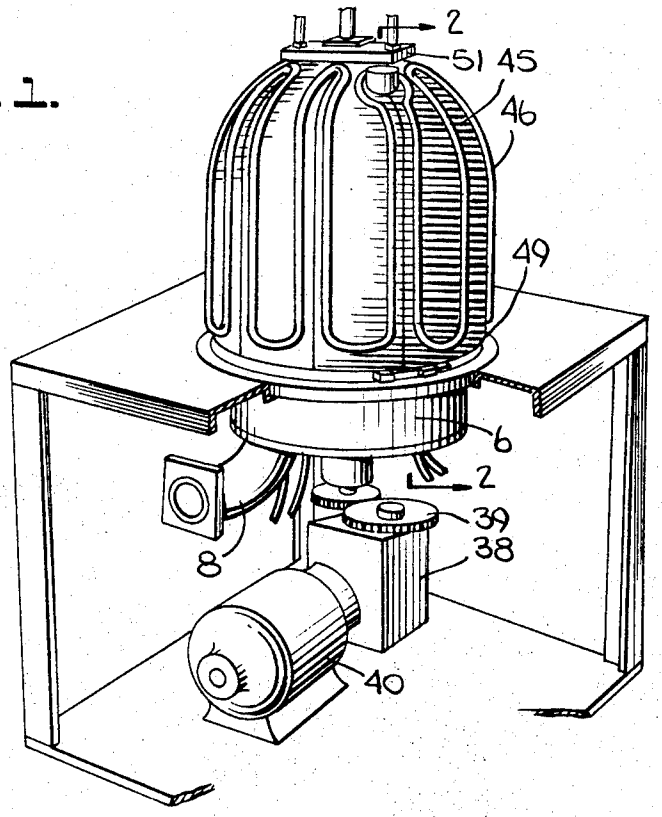


Fig. 6.

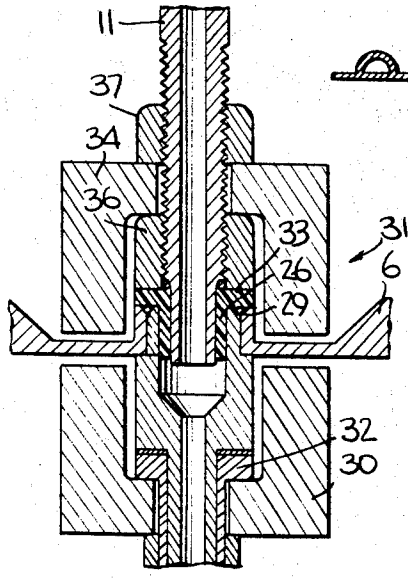
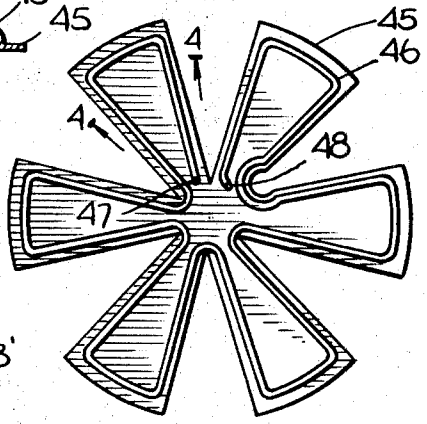


Fig. 4.



Fig. 3.



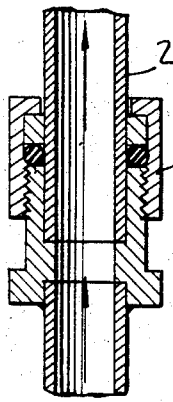


Fig. 5.

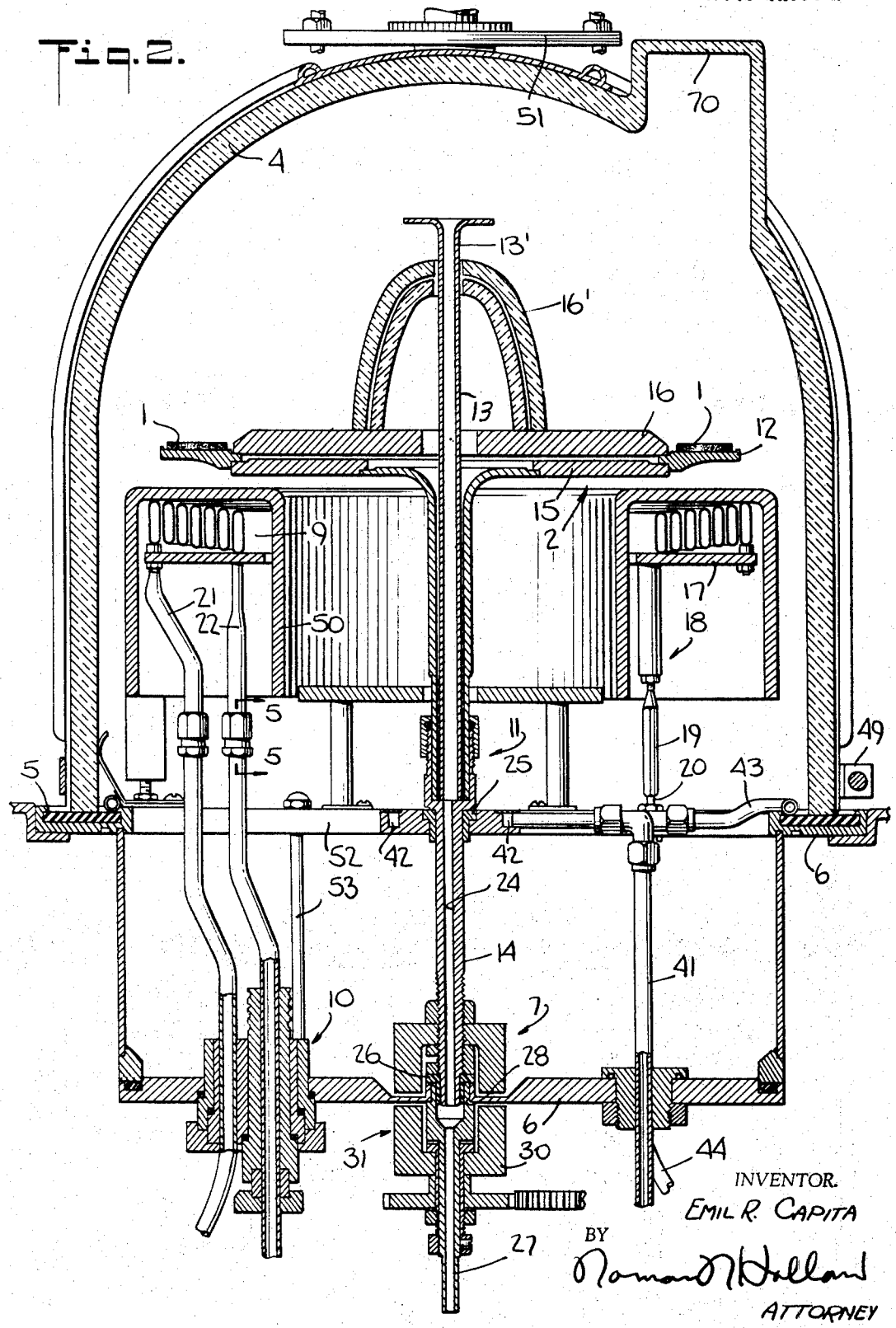
INVENTOR.
EMIL R. CAPITA
BY
Norman J. Holland
ATTORNEY

Oct. 29, 1968 E. R. CAPITA 3,407,783

VAPOR DEPOSITION APPARATUS

Filed Aug. 31, 1964 3 Sheets-Sheet 2

Fig. 2.



INVENTOR.
EMIL R. CAPITA
BY
Norman J. Holland
ATTORNEY

Oct. 29, 1968 E. R. CAPITA 3,407,783

VAPOR DEPOSITION APPARATUS

Filed Aug. 31, 1964 3 Sheets-Sheet 3

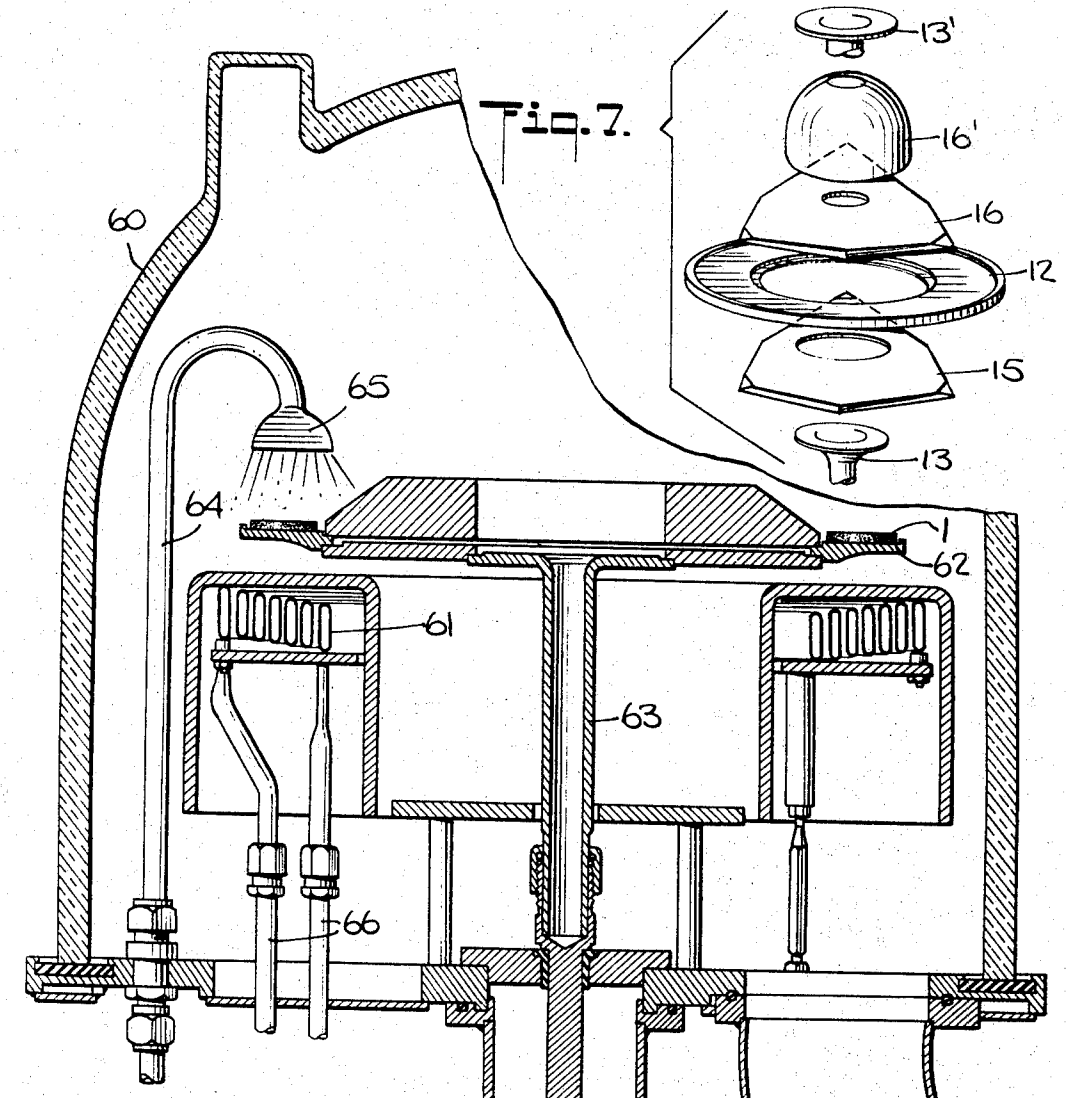


Fig. 7.

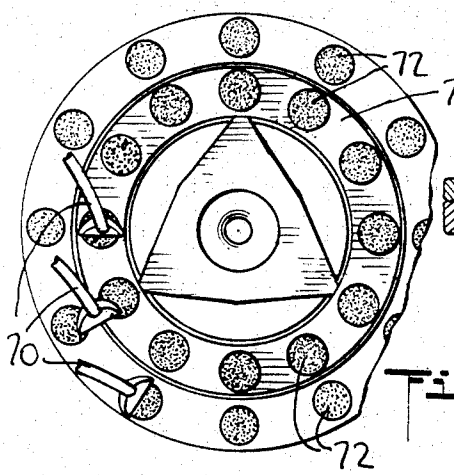


Fig. 9.

Fig. 8.

INVENTOR.
EMIL R. CAPITA

BY
Norman N. Holland
ATTORNEY

United States Patent Office 3,407,783
Patented Oct. 29, 1968

3,407,783

VAPOR DEPOSITION APPARATUS

Emil R. Capita, Hudson, N.J.
(7020 Hudson Blvd., North Bergen, N.J. 07047)

Filed Aug. 31, 1964, Ser. No. 393,253
5 Claims. (Cl. 118—49.5)

The present invention relates to a means for vapor plating and more particularly to an improved vapor coating furnace of the type adapted to form a coating of a precisely controlled and uniform thickness. The present invention is particularly characterized by its improved coating thickness and uniformity, its reduction in any possible contamination, and its improved and more useful structure which simplifies operation and decontamination.

The present invention represents an improvement in this general type of vapor coating apparatus wherein a uniformity of the coating thickness, an improved depth control, and a degree of purity of the coating are obtained which have not been heretofore achieved. In particular, the apparatus of the present invention is an improvement upon my copending application Ser. No. 189,365, filed Apr. 23, 1962, now Patent No. 3,233,578.

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 nonvolatile reaction product.

An important use of vapor deposition is in applying silicon coatings to silicon discs or slices such as are used in the manufacture of transistors. A silicon slice comprises the N-type and a silicon coating on the slice comprises the P-type. The coating purity, the uniformity of the coating thickness, and the control of the coating depth are of critical importance in transistors. The apparatus of the present invention obtains these results in a simpler and more reliable manufacturing operation to give a better product. The invention will now be described 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 other coatings on other objects in a similar way.

Accordingly, an object of the present invention is to provide improvements in vapor plating.

Another object of the present invention is to provide an improved apparatus for vapor plating combining improved coating control and contamination elimination with simplicity of furnace manufacture and operation.

Another object of the present invention is to provide an improved vapor plating furnace which is safely and efficiently operated by relatively inexperienced personnel.

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 partially cut away of a preferred embodiment of the vapor plating furnace of the invention;

FIG. 2 is a vertical sectional view of the vapor plating furnace of FIG. 1 taken along line 2—2 of FIG. 1;

FIG. 3 is a top plan view of a Faraday shield for the apparatus;

FIG. 4 is a sectional view of the Faraday shield taken along line 4—4 of FIG. 3;

FIG. 5 is a sectional view of a heating coil lead disconnect taken along line 5—5 of FIG. 1;

FIG. 6 is an enlarged detailed sectional view of the connection between the rotating article support and the source of gas and vapor;

FIG. 7 is an exploded perspective of the support of FIG. 2;

FIG. 8 is a vertical sectional view of another embodiment of the vapor plating furnace; and

FIG. 9 is a plan view of another embodiment of the furnace of FIG. 8 for polycrystal deposition.

The improved coating apparatus or furnace will first be described generally with particular reference to FIGS. 1 and 2. As discussed above, the process will be described in connection with the formation of a silicon coating upon silicon slices in the manufacture of P-N type transistor elements wherein the slices comprise the N-type and the silicon coating comprises the P-type. The silicon slices to be coated are illustrated at **1** arranged in spaced relation on the top of a susceptor **12** on a rotatable support **2**.

An air-tight chamber is provided surrounding the support **2** and comprising a removable cover **4** formed of a heat and corrosion resistant quartz. The cover **4** is removably mounted by means of an air-tight seal **5** on a hollow and air-tight base **6**. The gases used in the process to purge the air-tight chamber and the vapor plating mixture are admitted to the chamber through a gas and power coupling **7** in base **6** and an exhaust outlet **8** (FIG. 1) is provided to evacuate the enclosure and to withdraw the purging gases and the spent gases of the vapor coating mixture from the furnace.

The slices **1** are raised to the reaction temperature before the vapor plating operation by a pancake-type induction heating coil **9** mounted beneath the susceptor **12** and coupled to a high frequency power source through the coupling **10** in the base **6**. The ring-like susceptor **12** is preferably formed of molybdenum or pure graphite which is heated by the field of the induction heating coil **9** and which transfers the heat energy to the slices **1**. During the heating and the plating operations, the entire support **2** including the susceptor **12** is rotated. The support **2** includes a hollow support shaft **11** having an upper flanged portion **13** and a lower drive portion **14**.

The coating mixture of gas and vapor is directed upwardly through the hollow shaft **11** so that it flows radially outwardly over the slices **1** which are being rotated on the susceptor **12** of the support **2**. While the coating mixture may be passed directly out of the flared outlet at the top of the upper shaft portion **13**, a preferred embodiment is illustrated in FIG. 2 wherein a feed tube **13'** is mounted in the upper portion **13** of the shaft **11** having a flanged outlet at its top so that the coating mixture is released centrally but above the slices **1**. The uniformity of the coating thickness has been found to be improved by the use of a rounded dome **16'** positioned on support **2** below the feed tube **13'**. This dome **16'** is also formed of a heat resistant material such as quartz and a preferred embodiment as illustrated is made up of an inner and outer shell. As illustrated in FIG. 2, the ring-like susceptor **12** is removably held in place on a quartz support plate **15** and is held down on the support plate by a quartz hold-down plate **16**. As seen in FIG. 7, these plates **15** and **16** preferably have a generally triangular shape to further facilitate the even flow of the coating mixture about the slices **1**.

In order to further insure the uniform heating of the slices **1**, the induction heating coil **9** is mounted on an adjustable support plate **17** of a suitable heat resistant insulating material such as quartz. The relative position of the coil **9** with respect to the rotating support **2** is ad-

justed by changing the lengths of the supports **18** which connect the coil support plate **17** to the base **6**.

It will be seen that a fine adjustment can be made of the length of each of the supports by rotating the nut portion **19** on the threaded portion **20**. The slice heating may be controlled by this adjustment of the spacing between the coil **9** and the susceptor **12** through a suitable adjustment of the length of the supports **18**. In order to prevent corrosion and contamination within the chamber, the heating coil **9** and the connecting leads **21** and **22** between the coil **9** and the coupling **10** are silver or silver coated. Both the leads **21** and **22** and the coil **9** are hollow tubes to permit a coolant to be passed through them during the coating or epitaxial deposition operation.

The upper portion **13** of the shaft **11** for the support **2** is preferably made of a material which is heat resistant and corrosion resistant such as quartz. This upper portion **13** is threadedly coupled and sealed with an O-ring to a lower shaft **14** which also has a hollow center **24** communicating with the hollow center of the upper portion **13** and which communicates with the coupling **7** through bearing **26** as will be further described below. The rotatable shaft **11** is supported by thrust bearing **26** and axial bearing **25**.

In order to insure an absolutely tight furnace chamber for the coating operation and to maintain a contamination-free coating zone by preventing the entrance of impurities, a novel coupling plan for the detachable shaft **11** is provided in the improved furnace as is illustrated in detail in FIG. 6. The inlet line **27** for the coating vapor is welded to the bottom of the furnace chamber at a suitable aperture **28** as illustrated at **29**. This stationary vapor inlet line **27** has the lower portion **30** of the coupling magnet **31** rotatably mounted thereon on a suitable bearing **32**. The radial flange portion **33** of the thrust bearing **26** supports the weight of the support **2** together with bearing **25** and fixes the height of support **2** in respect to the heating coils **9**. In order to transmit the rotational drive of the lower magnet **30** portion to the support **2** and to simultaneously provide a hold-down force to improve the seal of the thrust bearing the upper portion **34** of the magnet **31** is fixedly attached to the support shaft **11** by the gripping nuts **36** and **37**. The lower portion **30** of the magnet **31** is coupled to a variable speed drive motor through the reduction box **38** and gear train **39** illustrated in FIG. 1 so that this portion of the magnet rotates about the stationary vapor inlet **27** at a predetermined speed. This motion is transmitted to the support **2** by the magnetic coupling between the upper and lower portions of the magnet **30** as the upper portion **34** rotates in synchronization with the lower portion **30**.

Since the vapor inlet **27** is welded to the furnace base **6**, it is clear that the only possible source of vapor leakage in this gland is around the surfaces of the thrust bearing **26**. This leakage is eliminated or effectively minimized due to the weight of the support **2** and the magnetic force which holds this bearing in place in the furnace base **6**. Any leakage that does occur, however, will be seen to pass the vapor into the furnace enclosure rather than into the surrounding atmosphere. Since this inlet is located in the lower portion of the furnace enclosure adjacent to the vapor outlet **8**, the vapor pressure on the inside of the bearing and within the shaft **11** will be greater than the pressure surrounding the outside of the coupling **7** so that any leakage which does occur will be outwardly so that there is no entry of vapor through the bearing **26** to the center of the hollow shaft **11**. It is thus clear that the above described vapor entry and drive coupling provide an absolutely leak proof and contamination proof vapor and power coupling for the furnace.

The top of the base **6** is cooled by the inlet **41** which connects a source of coolant to cooling channel **42** and cooling tube **43** at the outer edge of the base **6**. The channel **42** and tube **43** connect to outlet **44**.

As described above, the support **2** is continuously rotated during the vapor plating operation. The variable speed drive motor **40** is adjusted during the vapor plating operation to rotate the support **2** at speeds of from about 5 to 30 r.p.m. depending upon the thickness of the coating being applied and other operating conditions.

The quartz jar **4** is preferably shielded by a Faraday shield **45** which is cooled by the tube **46** coupled to a coolant source by inlet **47** and outlet **48**, FIG. 3. The shield **45** is clamped to the jar **4** by an insulated and open circuited ring-like clamp **49** (FIGS. 1 and 2).

Preferably, a cover **50** is positioned between the heating coil **9** and the support **2**. This cover **50** which is formed of a heat resistant nonconducting material such as quartz reduces heat transfer from the susceptor **12** to the water cooled heating coil and prevents the deposition of silicon on the heating coil **9**.

The base **6** and lower portion **14** of the shaft **11** are preferably made of corrosion resistant metal such as stainless steel.

In the vapor coating process described above it is particularly important that absolute purity be maintained for the slices **1** prior to the coating operation as well as for the surrounding elements of the furnace and it is equally important that this purity be maintained in the vapor coating formed during the coating process. The above described structure accomplishes this result with a high degree of efficiency, however, it has been found essential also to periodically perform a thorough clean-out of the interior of the furnace and of the various elements therein.

The improved furnace of my present invention has a novel arrangement of its elements to permit such a decontamination including the provision of readily removable supporting elements for the electrical coils and for the support elements for the slices including the vapor distributing and directing elements.

Referring to FIG. 2, the jar **4** is removably maintained on the base **6** with its lower edge engaging the air tight seal **5**. The jar or cover **4** is tightly clamped in this position by a movable top clamp **51**. When the clamp **51** is removed, the jar **4** together with its Faraday shield **45** is lifted off so that the interior of the furnace is exposed. The supporting and vapor distributing support **2** is now easily removed since, as described above, this support **2** is rotatably mounted in the furnace with the vertical shaft **11** supported in the base **6** by the two bearings **25** and **26**. The support **2** may therefore be removed by being simply lifted vertically from the bearings **25** and **26**. The top plate **52** of the base **6** is lifted from the furnace at the same time by removing the spaced disconnect bolts **53** which hold the plate **52** onto the lower portion of the base **6**. The support **2** and the base **6** may be removed as a unit. If it is desired to further disassemble these elements, the threaded couplings between the upper and lower portions of the column **11** and the threaded connection between the top magnet portion **34** and the lower portion of the shaft **11** may be disconnected. Prior to the lifting off of the plate **52** the heat cover **50** for the induction heating coil **9** may be lifted from its supports by the temporary raising of the support **2** and the heating coils **9** themselves are detached at the two disconnect couplings **53'** (FIG. 5). As indicated above both the slices **1**, the susceptor **12** and the related hold-down weight **16** are readily removable since they are held in place only by gravity in their normal operating positions.

FIG. 8 illustrates another embodiment of the improved vapor coating furnace. In the embodiment already described above, the vapors used in the coating operation are distributed in an outwardly flaring pattern from the hollow center **15** of the rotating support **2**. The embodiment illustrated in FIG. 8 has a similar furnace enclosure or jar **60** with a pancake-type induction heating coil **61** and an annular susceptor **62** mounted on the rim of a rotating support member **63**. In this embodiment, however, which is effective for extremely thin coatings the

incoming vapor is applied to the moving slices from a stationary vapor distributor **64** having a vapor directing nozzle **65** positioned directly above one portion of the circular path of the moving slices **1**.

The slices **1** are continuously heated by moving in a circular path immediately above the circular induction heating coil **61**. The slices **1** pass directly beneath an intense concentration of the coating vapor during a portion only of their rotation as they move beneath the vapor distributing nozzle **65**.

The interior of the coating furnace is kept tight and secure against contamination by having tight seals for the entry of the electrical conduits **66** and the vapor conduit **64** and by using a magnetic clutch **67** to transmit power to the rotating slice support **63**.

A typical operating cycle for the above described coating apparatus of FIGS. 1–7 will now be described.

With the quartz jar **4** removed from the base **6** by releasing clamp **51**, the silicon slices **1** which are to be coated are first carefully placed on a cleaned susceptor **12** and the susceptor **12** is placed on the support **2** and held down by the plate **16**. The quartz jar **4** is now placed on the seal **5** on the base **6** and is clamped into position by clamp **51** to form an air-tight chamber. A vacuum is now drawn in the air-tight chamber surrounding the slices **1** of the order of about one micron. The chamber is next purged with nitrogen or an inert gas such as argon, neon, helium and the like by passing it through the chamber between the coupling **7** and the outlet **8**.

A high frequency voltage source is connected to the induction heating coil **9** ranging from about 10 k.c. to about 450 k.c. adjusted to provide power in the range required for the various coating processes. A typical temperature range for applying silicon coatings is about 1190° C. to 1450° C. The induction heating coil **9** now heats the molybdenum or graphite susceptor **12** and the silicon slices **1** arranged around the edges of the susceptor **12**. The temperature of the slices **1** is observed by means of an optical pyrometer through a viewing surface **70** provided on the jar **4**. During the heating, the support **2** is rotated at speeds of from 5 to 30 r.p.m. or higher to insure a uniform heating of the several silicon slices **1** and pure hydrogen is passed through the chamber between coupling **7** and outlet **8**. A continuous supply of coolant is passed through the coil **9** and the cooling channel **42** and tubes **43** and **46** for the base and the Faraday shield **45** during the operation of the induction heating coil **9**. When the silicon slices **1** have reached a temperature of between 1190 and 1450 degrees C. the vapor plating is commenced by the admission of hydrogen gas containing silicon tetrachloride vapor through coupling **7**. This mixture enters through the above described conduits to the center of the rotating shaft **11** and it then flows outwardly and over the heated slices **1** on the rotating susceptor **12** in a uniform pattern. When the mixture of hydrogen and silicon tetrachloride vapor contacts the heated surfaces of the slices **1**, it reacts at the heated surface to deposit an adherent coating of silicon on each of the slices **1**. In a typical silicon coating operation, the pressure in the chamber at the slices **1** is maintained at about 1 to 2 p.s.i. above atmospheric pressure and the spent gases flow downwardly through the jar **4** to an exhaust zone within the base **6** adjacent to outlet **8** which is kept at about atmospheric pressure by the continuous evacuation of the spent gases through the exhaust outlet **8** to the atmosphere. This provides for a continuous flow of the mixture past the heated slices **1**. 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 **9** 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 **4** to provide access to the coated discs **1** after a suitable cooling period.

The embodiment illustrated in FIG. 8 and described above is operated in a generally similar manner including the loading and purging steps. During the actual coating process itself, however, the vapor is directed downwardly onto the moving slices **1** in a concentrated zone determined by the vapor nozzle **65**. The heated slices **1** move in a circular path above the induction heating coil **61** and continually pass through this area of concentrated vapor at a constant speed as they turn on the rotating support **63**.

As described above, the coating is done with various mixtures of vapors and gases with the composition depending upon the coating being applied. FIG. 9 illustrates another embodiment of the general type of furnace illustrated in FIG. 8 which differs by having a plurality of nozzles **70** positioned at the edge of a susceptor plate **71** for coating slices **72**. Different vapors or gases may be applied from the various nozzles **70** in performing a coating operation known as polycrystal deposition. This furnace thus provides a further degree of flexibility of operation by permitting this control of gas or vapor control through the various spaced nozzles **70** in addition to the slice temperature and rotational speed control as described above. While three nozzles **70** are illustrated in FIG. 9, it is clear that any number of two or more may be used as required for particular coatings.

FIG. 9 also illustrates double rings of slices **72** being coated on the susceptor **71** and one nozzle **70** is positioned over each ring and the third nozzle **70** is positioned in an intermediate position between the two rings of circularly arranged slices **72**. This provides a coating means of even greater capacity and flexibility of operation.

It will be seen that significant improvements have been provided in a means for vapor plating whereby a unique coupling is provided for the entry of vapor and for the rotational forces which cooperate with the article heating elements to provide for a uniform and pure vapor coating of objects. The elements of the improved furnace are combined in a particularly effective and novel manner to provide for improved coating characteristics combined with an improved overall coating operation wherein the decontamination, loading and purging 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.

I claim:

1. Apparatus for vapor plating articles comprising the combination of a sealed chamber; an induction heating coil in said chamber; an electrically conductive article supporting susceptor mounted on a rotatable hollow shaft adjacent said coil, said shaft adapted for connection to a source of said vapor located externally of said chamber and having a vapor outlet disposed substantially equidistant from and above said susceptor; a vapor guide member disposed uniformly about said hollow shaft and, from a location intermediate the vapor outlet and the article supporting surface of the susceptor, having a uniform wall portion extending from said shaft, downwardly and outwardly to said susceptor surface; a vertical thrust bearing for said shaft within said sealed chamber for supporting the shaft, and a non-conducting heat resistant shield member intermediate said coil and said support.

2. The apparatus as claimed in claim **1** in which said susceptor comprises a ring-like member, said susceptor being removably mounted on said shaft intermediate a support plate and a hold down plate, and said plates being shaped to permit the passage of a portion of the vapor

downwardly through the center of the ring-like susceptor.

3. The apparatus as claimed in claim 1 which further comprises a Faraday shield at least partially surrounding said chamber.

4. The apparatus as claimed in claim 3 in which said shield includes liquid cooling means.

5. The apparatus as claimed in claim 1 wherein means to drive said hollow shaft comprises a magnetic couple.

References Cited

UNITED STATES PATENTS

1,821,530	9/1931	Spire	219—10.49
2,398,382	4/1946	Lyon	118—49.1 X
2,532,971	12/1950	Van Leer et al.	118—49.1 X
2,828,225	3/1958	Goetzel et al.	
2,997,979	8/1961	Tassara	118—49
3,023,727	3/1962	Theodoseau et al.	117—107.1 X
3,045,642	7/1962	Auzolle	118—49
3,099,579	7/1963	Spitzer et al.	118—48 X
3,226,254	12/1965	Reuschel	118—49.5 X
3,233,578	2/1966	Capita	118—49.1

CHARLES A. WILLMUTH, *Primary Examiner.*

MORRIS KAPLAN, *Assistant Examiner.*