May 29, 1962 H. L. CASWELL 3,036,933

VACUUM EVAPORATION METHOD

Filed Oct. 6, 1959

5 Sheets-Sheet 1

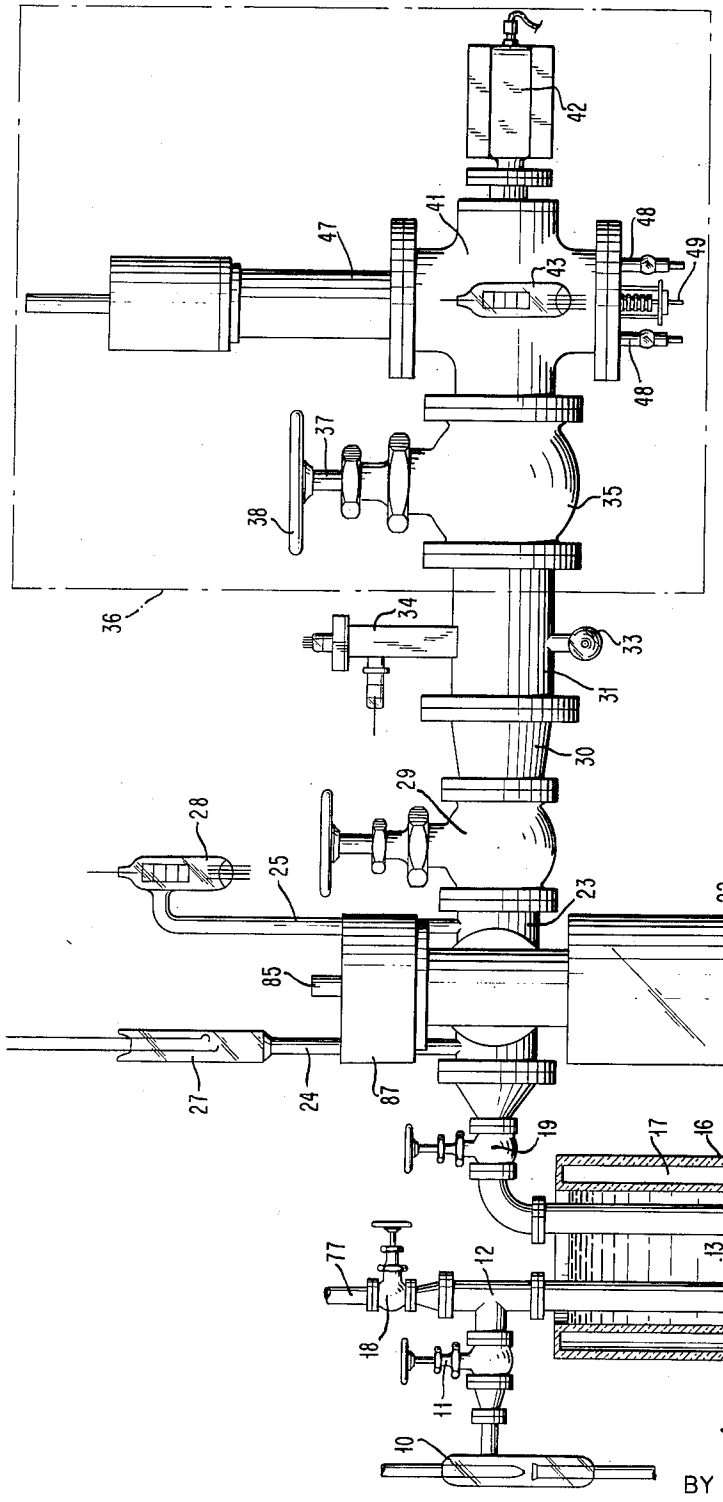


FIG. 1

INVENTOR
HOLLIS L. CASWELL

BY Frederick K. Smyth

ATTORNEY

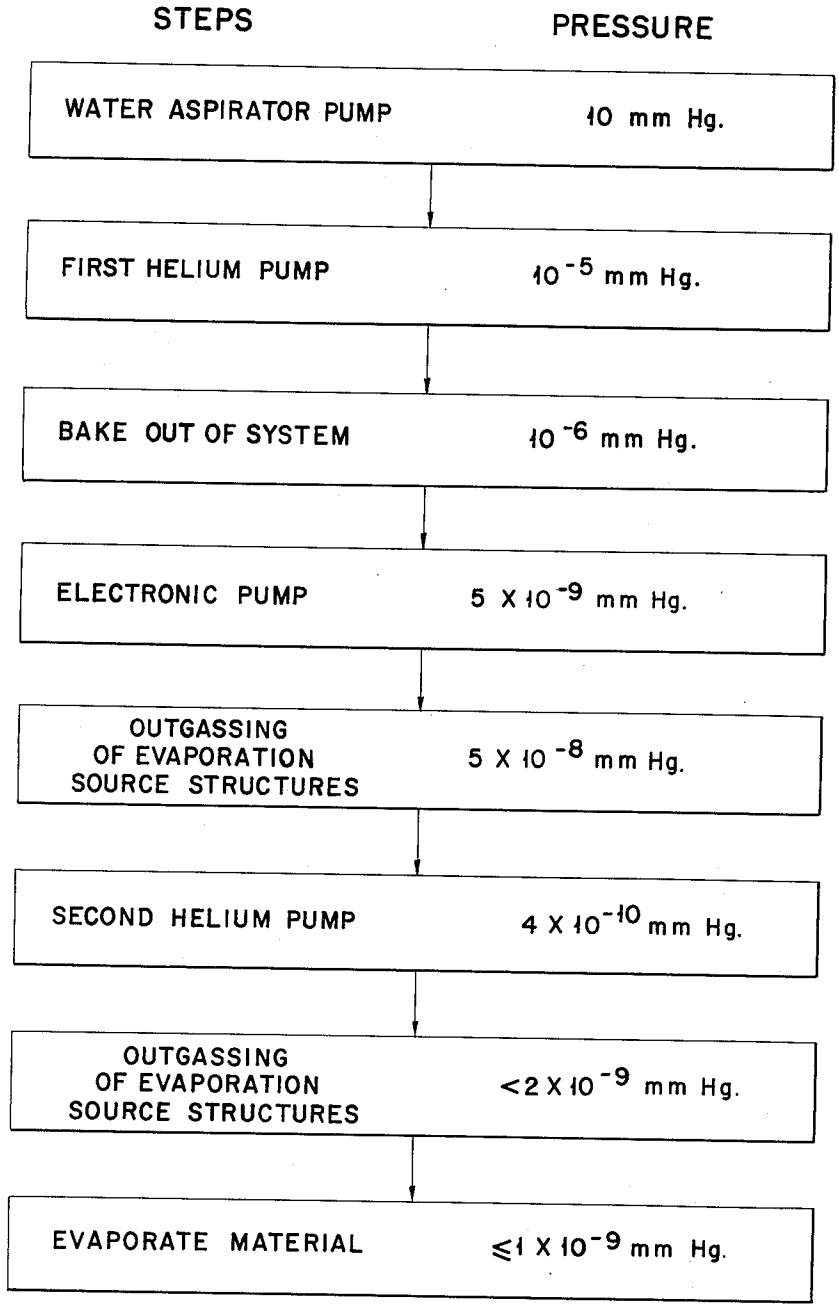

STEPS
PRESSURE
WATER ASPIRATOR PUMP
10 mm Hg.
FIRST HELIUM PUMP
10^{-5} mm Hg.
BAKE OUT OF SYSTEM
10^{-6} mm Hg.
ELECTRONIC PUMP
5×10^{-9} mm Hg.
OUTGASSING OF EVAPORATION SOURCE STRUCTURES
5×10^{-8} mm Hg.
SECOND HELIUM PUMP
4×10^{-10} mm Hg.
OUTGASSING OF EVAPORATION SOURCE STRUCTURES
$< 2 \times 10^{-9}$ mm Hg.
EVAPORATE MATERIAL
$\leq 1 \times 10^{-9}$ mm Hg.

FIG. 2

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FIG. 3a

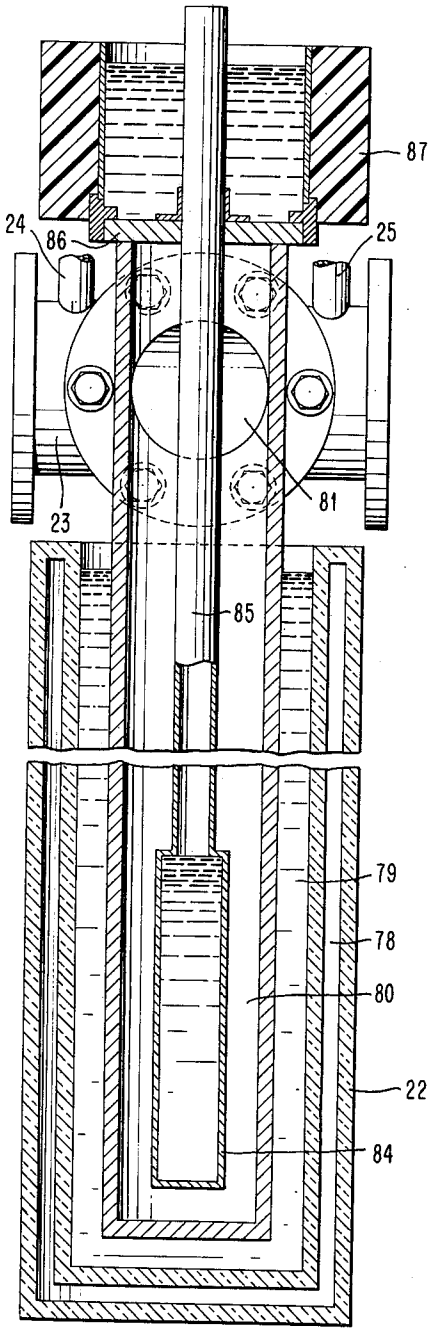
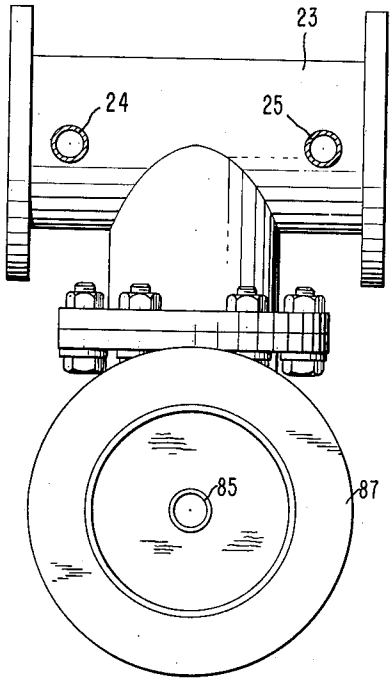


FIG. 3b



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5 Sheets-Sheet 4

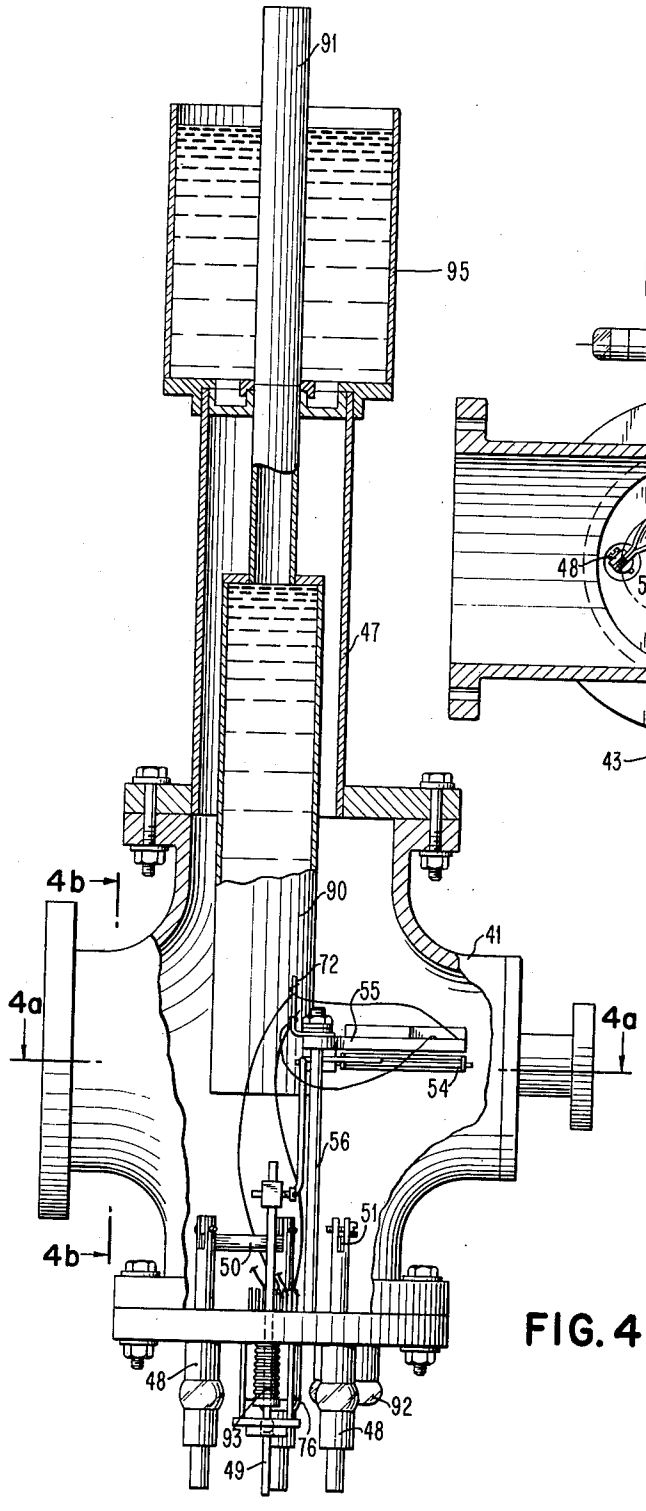


FIG. 4

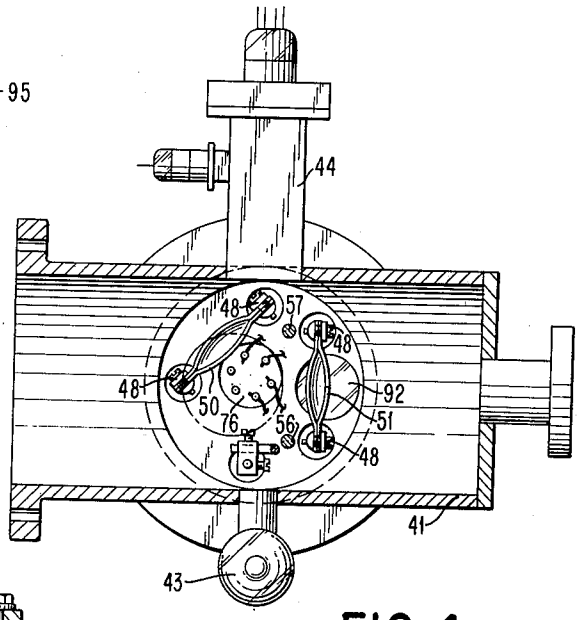


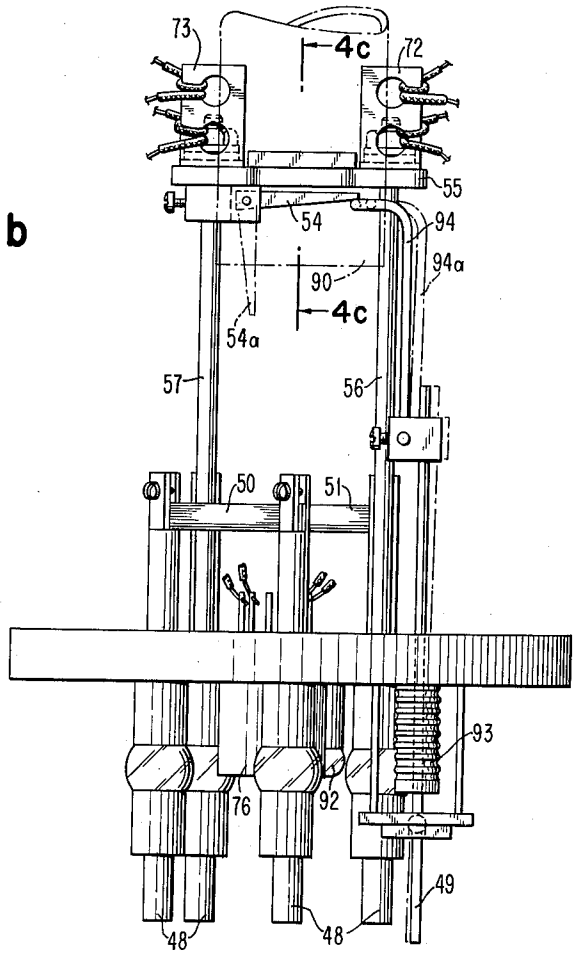
FIG. 4a

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FIG. 4b



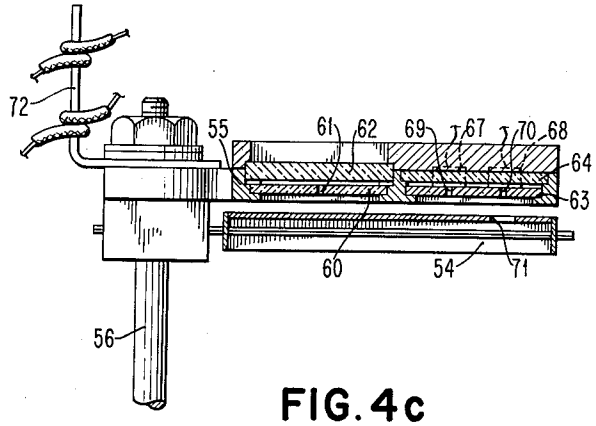


FIG. 4c

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VACUUM EVAPORATION METHOD

Hollis L. Caswell, Poughkeepsie, N.Y., assignor to International Business Machines Corporation, New York, N.Y., a corporation of New York

Filed Oct. 6, 1959, Ser. No. 844,754

6 Claims. (Cl. 117—107)

This invention relates to a method of evaporating materials within a vacuum, and more particularly, to a method of obtaining vacuum deposition of materials while maintaining an ultra high vacuum.

Vacuum deposition of materials onto a substrate has been employed in many fields to produce a large variety of articles. Generally, the methods of the prior art consist, essentially, of heating a coating metal in a vacuum and directing the vapors therefrom onto the article or substrate to be coated. Recently, because of advances of the state of the art in many fields, including, by way of example, magnetics and cryogenics, there has arisen the need of obtaining extremely thin coatings having a degree of purity and uniformity greater than previously possible.

It has been found that the electrical properties of evaporated coatings or films exhibit a lack of reproducibility in the results attained. Films evaporated under apparent identical conditions have exhibited a wide range of characteristics. The potentiality of utilizing evaporated thin film devices fabricated from magnetic or superconductive materials in high speed computers has stimulated further interest in evaluating the role various parameters cause in the characteristics of the evaporated thin film. It has been found that one important parameter in determining these characteristics is the magnitude and composition of the vacuum in which the evaporation occurs. In general, the majority of evaporations have been performed at pressures in the 10^{-5} to 10^{-6} mm. Hg range within systems evacuated by the well known oil diffusion pumps. In these systems, when conventional evaporation sources are employed, between one and fifty percent of the particles adhering to the substrate are residual gas molecules.

These gas molecules produce several detrimental effects in the electrical characteristics of deposited materials. By way of example, extremely small quantities of impurities in superconductive materials result in a disproportionately large variation in the following important electrical characteristics: the critical field, which is the magnetic field required to switch a superconductive conductor from the superconducting state to the resistive state; the critical temperature, which is the temperaure a which superconductivity appears in the absence of an applied magnetic field; the crtical self-current, which is the maximum current a superconducting conductor can carry before this current itself destroys superconductivity; the slope of the transition curve between the superconducting state and the resistive state; and the thermal and magnetic time constants. When thin film cryogenic devices of the type described in copending application Serial No. 625,512, filed November 30, 1956, on behalf of Richard L. Garwin and assigned to the assignee of this invention, are employed in large scale devices, such as computers or the like, it is desirable that each of the above characteristics be accurately controlled within close limits. As an example, each of the gate conductors of the above referenced cryogenic devices must have about the same value of critical temperature and critical self-current to ensure that all will be superconducting in the absence of an applied magnetic field. Additionally, each of the gate conductors must have about the same critical field value, to ensure selected gate conductors are in the resistive

state when subjected to the magnetic field applied by an associated control conductor.

What has been discovered is a novel method of evaporating materials in an ultra high vacuum, by which the contamination introduced by residual gas molecules is reduced to as much as one part in 10^{-6}, assuming that every gas molecule striking the substrate adheres thereto. The novel method of the invention consists, essentially, of obtaining an ultra high vacuum and, further, maintaining this vacuum during the actual evaporation of the coating material. Although the method will be described with particular reference to evaporation of superconductive materials as a particular example, it will be understood as the description proceeds, that the method may be employed whenever it is necessary to produce thin films by vapor deposition of high purity having reproducible characteristics.

More particularly, the method of the invention comprises evacuating a vacuum chamber through a series of steps to attain a vacuum therein of about 10^{-10} mm. Hg, which is relatively free of hydrocarbon vapor, subjecting the chamber, evaporation source structure, and coating material to a number of outgassing steps, and finally evaporating the coating material while maintaining the pressure at about 10^{-9} mm. Hg. By means of the novel combination of steps in the method of the invention, thin films having reproducible characteristics are obtained.

It is an object of the invention to provide an improved vacuum evaporation method.

Another object of the invention is to provide a method of obtaining vacuum deposition of materials while maintaining an ultra high vacuum.

A further object of the invention is to provide an improved method of fabricating superconductive devices.

Yet another object of the invention is to provide a method of fabricating thin films having a degree of purity and uniformity greater than previously possible.

Still another object of the invention is to provide a method of fabricating superconductive devices having reproducible characteristics.

A still further object of the invention is to provide a method of forming thin films through vapor deposition while maintaining a vacuum of about 10^{-9} mm. Hg.

A related object of the invention is to provide an improved method of attaining an ultra high vacuum.

The foregoing and other objects, features and advantages of the invention will be apparent from the following more particular description of a preferred embodiment of the invention, as illustrated in the accompanying drawings.

In the drawings:

FIG. 1 is a diagrammatical illustration of an apparatus useful in practicing the method of the invention.

FIG. 2 is a flow diagram illustrating the primary steps of the method of the invention.

FIG. 3*a* is a cross-sectional view of a helium pump useful in practicing the method of the invention.

FIG. 3*b* is a top view of the helium pump shown in FIG. 3*a*.

FIG. 4 is a cross-sectional view of a vacuum chamber useful in practicing the method of the invention.

FIG. 4*a* is a sectional view of the chamber illustrated in FIG. 4 along the lines 4*a*—4*a*.

FIG. 4*b* is a sectional view of the chamber illustrated in FIG. 4 along the line 4*b*—4*b*.

FIG. 4*c* is a sectional view of the substrate holder illustrated in FIG. 4*b* along the line 4*c*—4*c*.

The method of the invention may be practiced with various types of apparatus. FIG. 1 discloses, by way of example, an apparatus which has satisfactorily been employed to obtain the advantages of the method of

the invention. Each of the demountable sections illustrated therein is sealed by means of O rings fabricated of Teflon or gold rather than conventional O rings which might release hydrocarbon vapors. As shown in FIG. 1, the apparatus includes a conventional water aspirator **10** which may be isolated from the remainder of the apparatus by means of a valve **11**. Also connected to valve **11** through a T section **12** is a U-shaped member **13** immersed in liquid nitrogen. The liquid nitrogen is contained in a standard Dewar **16** having a vacuum insulating chamber **17**. This cooling of member **13** by liquid nitrogen prevents backstreaming of water vapor when aspirator **10** is operated. A valve **18** attached to section **12** is employed to fill the system with a helium and hydrogen free gas at atmospheric pressure after a partial vacuum has been attained by aspirator **10**, as an aid in reducing the amount of helium and hydrogen gases remaining in the final vacuum as will be further understood as the description proceeds. Both of valves **11** and **13** are isolated from the remainder of the apparatus by a valve **19**.

The next component of the apparatus illustrated in FIG. 1 is a liquid helium pump **22**. Helium pump **22** is similar to the helium pump disclosed in copending application Serial No. 809,409, filed April 27, 1959, on behalf of Irving Ames et al., and assigned to the assignee of this invention. Helium pumps are effective to rapidly reduce the pressure within a vacuum chamber without the use of pump fluids, which could otherwise introduce contaminating vapors into the vacuum system. Pump **22** is secured to the apparatus of FIG. 1 by another T section **23**. Secured to section **23**, are a pair of tubes **24** and **25**, the former providing support for a Pirani vacuum gage **27** and the latter providing support for a Bayard-Alpert ion gage **28**. Pirani vacuum gages are used to measure pressures down to about 10^{-3} mm. Hg and Bayard-Alpert ion gages are employed to measure pressures down to about 10^{-10} mm. Hg.

Section **23** is connected to the remainder of the apparatus by a valve **29**, a tapered section **30**, and a section **31**. Secured to opposite radii of section **31** is a second Bayard-Alpert ion gage **33** and a mass spectrometer tube **34**. As more particularly detailed hereinafter, the mass spectrometer is used in conjunction with an ion gage to determine the exact composition of the residual gases within the vacuum system. Section **31** is further connected to a bakeable all metal valve **35**, located within an oven **36** indicated by the dashed outline in FIG. 1. Valve **35** consists of a stainless steel seat into which a copper cone is driven by a shaft **37** under control of a handle **38**, and has a conductance of less than 10^{-11} liters per second when closed. The opposite end of valve **35** is secured to a vacuum chamber **41**. To the opposite end of chamber **41** is fastened an electronic ion pump **42** which may be, by way of example, of the type presently marketed by Varian Associates, Inc., Palo Alto, California, under the trademark "Vacion" as model VA–1402 High Vacuum Pump. Also secured to chamber **41** and oriented 90° with respect to both valve **35** and pump **42** are a Bayard-Alpert ion gage **43** and a mass spectrometer **44** (see FIG. 4*a* for the location of spectrometer **44**). Extending through the upper portion of chamber **41** is a second helium pump **47** and extending through the lower portion of chamber **41** are a number of electrical terminals **48**, shutter control **49**, and a viewing tube **92**, the operation of which will be hereinafter described in detail.

As the description proceeds, the cooperation of the various pumps and valves will be understood, and structural details of the above briefly described apparatus will be pointed out. As an aid in following the method of the invention, reference should now be made to FIG. 2 which indicates in the form of a flow diagram, the basic steps of the method. As there shown, the first step in the method is to reduce the pressure within the system to about 10 mm. Hg by use of the water aspirator pump. Next, the water aspirator pump is isolated from the system and the first helium pump is then operated to further reduce the system pressure to about 10^{-5} mm. Hg. The vacuum chamber is then heated while the helium pump is operating, to drive out absorbed gas molecules from the components within the vacuum chamber **41**, while the pressure reduces to approximately 10^{-6} mm. Hg. The first helium pump is next isolated from the vacuum chamber and the electronic ion pump is operated to reduce the pressure to about 5×10^{-9} mm. Hg. At this point, the evaporation source structures are outgassed until only a rise in pressure to less than 5×10^{-8} is observed. Next, the second helium pump is operated to reduce the pressure to 10^{-10} mm. Hg, and the evaporation sources are again outgassed. After the chamber components have cooled sufficiently, the second helium pump is again operated and the evaporation proceeds at a pressure that is maintained less than 10^{-9} mm. Hg, to minimize the number of residual gas molecules adhering to the substrate.

Although the method of the invention may be employed in the vacuum evaporation of many materials, a specific example will next be described in detail, by way of example, in which a tin conductive coating having a predetermined pattern is deposited upon a glass substrate. The use of tin as a coating metal has been chosen as an example because tin is a desirable metal for use as gate conductors in the hereinbefore referenced thin film cryogenic devices and for the reasons described above these films must have a high degree of purity.

Referring again to the drawings, FIG. 4 is a cross-sectional view of vacuum chamber **41**. Within the lower portion of chamber **41** and secured to electrodes **48** are a pair of evaporation source structures **50** and **51**, the location of which may further be understood with reference to FIG. 4*a*. For reasons which will be understood as the description proceeds, each of sources **50** and **51** are preferably fabricated of tantalum. Vertically aligned with source **51** is a removable evaporation shutter **54** and a substrate holder **55**, spaced from the base plate portion of chamber **41** by rods **56** and **57**.

To prepare for an evaporation operation, a pattern defining mask **60**, having an opening **61**, is positioned in holder **55** as shown in FIG. 4*c*, and a substrate **62**, upon which the tin pattern is to be deposited, is positioned immediately thereabove. Additionally, a monitor slide pattern mask **63** and a monitor slide **64** are also positioned in holder **55**. Monitor slide **64** is fabricated of electrically non-conductive material, as for example glass, having a pair of conductive lands secured to each opposite edge thereof, of which a first pair **67** and **68** may be seen in outline in FIG. 4*c*. Monitor slide **64** is employed during both an outgassing operation of the sources and the actual evaporation operation. It can be seen in FIG. 4*c* that monitor slide mask **63** includes a pair of transverse slots **69** and **70** and that shutter **54** includes an opening **71** in vertical alignment with opening **70** of monitor slide mask **63**. In this manner, during the time evaporation source structure **51** is being outgassed, vapors of the material contained therein will deposit on monitor slide **64** and provide an electrically conductive path between only land **68** and the corresponding land opposite thereof through openings **71** in shutter **54** and **70** in monitor slide mask **63**. Thus, the quantity of tin evaporated from source **51** can be evaluated during an outgassing operation as more fully explained below. Additionally, land **67** is employed together with a corresponding land opposite thereof to indicate the thickness of a deposit upon substrate **62** during an evaporation operation when shutter **54** no longer shields the substrate. The coating thickness of the material deposited upon monitor slide **64** is determined by measuring the resistance of the coating deposited between each opposite pair of lands through openings **69** and **70**. For this reason, electrical leads are secured to each land which are connected at a pair of junction posts **72** and **73** (see FIG. 4*b*), and thence to the

wires connected to a vacuum seal feed-through connector 76 located in the base of chamber 41 as shown in FIGS. 4, 4*a*, 4*b* and 4*c*. A resistance meter can be connected to selected terminals of connector 76 at atmospheric pressure to determine the resistance of a particular deposit on monitor slide 64 which is proportional to the thickness of the coating deposited thereon.

At this point metallic tin is inserted in source 51 only, and no material is placed in source 50 for reasons that will be understood as the description proceeds, and chamber 41, as well as the remainder of the components of the apparatus are vacuum sealed. Now, as the description of the method proceeds, FIGS. 1 and 2 should be referred to as necessary for background material, specific reference being made to the other figures as required. Next, valves 11, 19, 29 and 35 are opened and valve 18 is closed. Water is fed through aspirator pump 10, and pump 10 is effective to reduce the system pressure from atmospheric to about 10 mm. Hg. U-shaped member 13, cooled by the liquid nitrogen in Dewar 16, prevents water vapor from aspirator pump 10 from passing further into the system. When the system pressure is stabilized at about 10 mm. Hg, as indicated by Pirani vacuum gage 27, valve 11 is closed and valve 18 is then opened. Valve 18 is connected through a tubing 77 to a source of helium and hydrogen free gaseous nitrogen maintained at asmospheric pressure, and open valve 18 is now effective to fill the system with gaseous nitrogen and valve 18 is then closed. At this point, valve 11 is reopened and aspirator pump 10 is again effective to reduce the system pressure to about 10 mm. Hg. Depending on the amount of helium initially within the system, the steps of flushing the system with helium-free gaseous nitrogen is repeated several times by selectively operating valves 11 and 18. This step is included in the method of the invention since atmospheric air contains approximately five parts per million of helium and 100 parts per million of hydrogen, yielding a partial pressure of helium in the system of about 10^{-4} mm. Hg, and that of hydrogen about 2×10^{-3} mm. Hg. However, it is desirable to reduce the content of these gases to less than 10^{-7} to increase the efficiency of helium pump 47 as will be further detailed hereinafter. Successively flushing the system with nitrogen accomplishes this result.

The next step in the method is to close valves 19 and 11, and operate liquid helium pump 22. Referring now to FIGS. 3*a* and 3*b*, it can be seen that pump 22 includes three inner chambers; a first vacuum insulating chamber 78, a second chamber 79 for containing liquid nitrogen, and a third chamber 80 connected to the vacuum system through an opening 81. Centrally located within chamber 80 is a copper receptacle 84, which is filled with liquid helium, when pump 22 is to be operated, through a stainless steel tube 85. Tube 85 passes through a vacuum seal in a disk 86, and the upper portion of tube 85 is cooled by liquid nitrogen contained in a retainer 87, in order to minimize the thermal loss of the liquid helium. To operate helium pump 22, the following procedure is preferred. First, retainer 84 is filled with liquid nitrogen to condense out all water vapors in contact with the walls thereof. Next, retainer 87 is filled with liquid nitrogen as is also chamber 79. Valve 29 is then closed and the liquid nitrogen is removed from retainer 84 by blowing gaseous helium therein. When all of the liquid nitrogen within retainer 84 has been removed, retainer 84 is filled with liquid helium and pump 22 is operating.

With pump 22 operating, valve 29 is "cracked" after Bayard-Alpert gage 28 indicates that the pressure has fallen below 10^{-4} mm. Hg. Valve 29 is slowly opened in order that the system presure does not rise above 2×10^{-2} mm. Hg as indicated in Pirani vacuum gage 27. As valve 29 continues to open the system pressure drops to 10^{-5} mm. Hg or below within several minutes, as indicated by Bayard-Alpert ion gage 33. At this time mass spectrometer 34 may be employed to test for leaks in the vacuum system as a safety precaution using helium, argon, or other suitable tracer gas.

The next step in the method is to bake out the high vacuum portion of the apparatus. Oven 36 is operated to raise the surface temperature of vacuum chamber 41 to 430° C. and this temperature is maintained for a period of about five hours. During this time the system pressure raises above 10^{-5} mm. Hg, subsequently falling to 10^{-6} mm. Hg as nitrogen and water vapor are driven off the chamber walls, and removed from the system by pump 22. When the oven is turned off, the chamber begins to cool and when the temperature falls to 250° C., all metal bake out valve 35 is closed. Next, evacuation of chamber 41 is continued by operating electronic ion pump 42. After operating pump 42 for approximately 12 hours, the pressure within vacuum chamber 41 is about 5×10^{-9} mm. Hg, as indicated by Bayard-Alpert ion gage 43.

The next step in the method is to thoroughly outgas evaporation source structures 50 and 51 (see FIGS. 4 and 4*a*). Empty source 50 is heated to 2000° C. and then slowly allowed to cool. Next, source 51 containing the tin charge then is heated to about 1000° C. and also allowed to cool. Cyclic heating of sources 50 and 51 continues for about one-quarter hour at which time source 51 is further raised in temperature until about 1% of the tin therein deposits on monitor slide 64 through opening 71 in evaporation shutter 54 and opening 70 in monitor slide mask 63 (see FIG. 4*c*). At this point, the pressure within chamber 41 is about 5×10^{-8} mm. Hg and the second helium pump is now operated to reduce the pressure below 10^{-10} mm. Hg. This is accomplished by filling a copper retainer 90 of the second helium pump 47 with liquid helium through a stainless steel tube 91, after retainer 90 has been precooled with liquid nitrogen and container 95 filled with liquid nitrogen.

Next, the evaporation source structures 50 and 51 are again outgassed in a manner similar to that described above after the system pressure has fallen below 10^{-10} mm. Hg with about 10% of the tin charge being evaporated and the system is allowed to cool and stabilize for a period of about 12 hours. At the end of the 12 hour period, the pressure within the system is in the high 10^{-10} region due to gettering of the small amount of tantalum evaporated from empty source 50 during outgassing and to the mild bake out chamber 41 received during the outgassing procedure.

The next step in the method is to perform an evaporation operation. Liquid helium is again caused to fill retainer 90 and source 51 is heated to about 1500° C. When steady state evaporation of the tin has been attained which can be monitored by monitor slide 64, shutter control 49, operable through a bellows 93 is moved to the left pivoting a rod 94 to the position indicated at 94*a* in FIG. 4*b* and allowing evaporation shutter 54 to drop to the position shown at 54*a*. Thus, evaporated tin is now deposited on substrate 62 through opening 61 in pattern defining mask 60.

In the design of liquid helium pumps a compromise must be made between pumping speed and liquid helium consumption, since high vacuum conductance generally implies high heat conductance. For the apparatus shown in FIG. 1 each of the helium pumps were designed to operate from a single filling from a storage Dewar so that the total heat loss was determined by the liquid helium capacity. As an aid in understanding the method of the invention only the following specific valves are illustrative of the apparatus useful in providing the tin coating on a single substrate as described above. Helium pump 22 shown in FIG. 3*a* comprises a retainer 84 fabricated of a 9 inch length of 0.030 inch wall OFHC copper tubing 1⅝ inches in diameter, holding 340 cc. of liquid helium and having a surface area of 270 cm.^{2}. An average of 1½ liters of liquid helium is required to cool and fill the retainer when the system pressure is about 10 mm. Hg, the

pressure attained by water aspirator pump **10**. Retainer **84** is supported by a 16 inch length of 0.006 inch wall ¾ inch diameter stainless steel tubing which has the upper portion thereof cooled by liquid nitrogen. Thermal support losses are thus small, being of the order of 10^{-3} watts. Liquid nitrogen in chamber **79**, surrounding retainer **84** reduces radiation losses to the order of 10^{-3} watts. Thermal losses due to conduction by residual gas molecules in the vacuum system are a function of the pressure and composition of the gas. The presence of helium due to either a high initial concentration or diffusion into the system through glass components increases these thermal losses because helium is not effectively pumped by the helium pump. Further, helium has a high thermal conductivity so that a partial pressure of the order of 10^{-6} mm. Hg in the system is sufficient to make gaseous conduction losses comparable to support and radiation losses. Experimentally, a helium partial pressure of 10^{-4} mm. Hg, as measured in the room temperature portion of the system, increased the liquid helium consumption rate in retainer **84** to ten times the consumption rate when the helium partial pressure was less than 10^{-7} mm. Hg. This is the primary reason for the flushing of the system with gaseous nitrogen during the initial evacuation of the system.

The pumping speed of pump **22** is limited to 30 liters per second by the tubing connecting the pump to vacuum chamber **41**. This tubing must be long to reduce support conduction losses to retainer **84** and of relatively small diameter to fit into standard nitrogen Dewar **16**. Further, it might be expected that slight warming of the upper portion of retainer **84** as the level of liquid helium dropped would be sufficient to re-evaporate gases which had condensed thereon when the retainer was full. However, with the retainer fabricated of OFHC copper as pointed out above, the temperature difference between the upper and lower portion of retainer **84** is less than 1° Kelvin and little re-evaporation occurs.

The requirements of helium pump **47** differ from those of pump **22** primarily in that a higher pumping speed is necessary during an evaporation operation to maintain the pressure within chamber **41** in the 10^{-9} mm. Hg range in order to obtain a high purity coating upon the substrate. As shown in FIG. 4, liquid helium retainer **90** is inserted directly into vacuum chamber **41** so that the inherently high pumping speed is not limited by connecting tubing leading from the vacuum chamber to the pump. Retainer **90** is identical to retainer **84** of pump **22** and a pumping speed between 500 and 1000 liters per second is attained. Approximately one liter of liquid helium is required to cool and fill retainer **90** after precooling with liquid nitrogen. Since no radiation shielding is employed, radiation losses limit the pumping time to approximately 1½ hours. However, during an evaporation operation the pumping time is reduced to several minutes with a single filling of retainer **90** having the above specific dimensions.

What has been described is a method of vacuum evaporation wherein, through a novel combination of steps, the coating material is evaporated at a pressure of about 10^{-9} mm. Hg. Although pressures in this range have been attained heretofore, they have been attained in a static system, while an evaporation operation must be of necessity occur in a dynamic system due to gas evolution from the coating metal and the source structures. Thus, through a novel combination of pumping and outgassing steps a method has been illustrated wherein an ultra high vacuum can be maintained in a dynamic system.

While the invention has been particularly shown and described with reference to a preferred embodiment thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the spirit and scope of the invention.

What is claimed is:

1. A method of forming high purity coatings upon a substrate comprising the steps of mounting said substrate within a vacuum chamber in spaced relationship to an evaporation source structure; providing a removable shield adjacent said substrate to selectively shield a surface of said substrate from said source; placing a coating material within said source; attaining a vacuum within said chamber including the steps of employing a water aspirator to reduce the pressure within said chamber to a few mm. Hg, operating a first liquid helium pump for a time sufficient to further reduce the pressure within said chamber to about 10^{-5} mm. Hg, subjecting said chamber to a predetermined temperature to remove a portion of sorbed gases therefrom, cooling said chamber, isolating said first helium pump from said chamber, operating an electronic ion pump to still further reduce the pressure within said chamber, operating a second liquid helium pump located within said chamber to reduce the pressure therein to about 10^{-10} mm. Hg; outgassing said source by heating said source until about 10% of the material contained therein is evaporated; removing said shield from said substrate surface; and subjecting said source to a predetermined temperature at which said material vaporizes and deposits upon said substrate surface while said second helium pump maintains the pressure within said chamber in the range of 1 to 5×10^{-9} mm. Hg.

2. The method of claim 1 wherein said coating material is tin.

3. A method of forming a high purity coating upon a substrate comprising the steps of providing a pair of tantalum evaporation source structures within a vacuum chamber; mounting said substrate vertically above one of said sources; placing metallic tin in said source beneath said substrate; attaining a vacuum within said chamber including the steps of employing a water aspirator to reduce the pressure within said chamber to a few mm. Hg, isolating said chamber from said aspirator, filling said chamber with a helium and hydrogen free gas at atmospheric pressure, reoperating said water aspirator to again reduce the pressure within said chamber to a few mm. Hg, operating a first liquid helium pump to further reduce the pressure within said chamber to about 10^{-5} mm. Hg, isolating said first helium pump from said chamber, operating an electronic ion pump to still further reduce the pressure within said chamber, operating a second liquid helium pump located within said chamber to reduce the pressure therein to about 10^{-10} mm. Hg; outgassing each of said tantalum sources by subjecting said tin containing source to about 1000° C. and said non tin containing source to about 2000° C.; and subjecting said tin containing source to a predetermined temperature at which said tin vaporizes and deposits upon said substrate during the time said second helium pump is effective to maintain the pressure within said chamber at about 10^{-9} mm. Hg.

4. An improved evaporation method comprising the steps of positioning a substrate within a vacuum chamber in spaced relationship to an evaporation source structure; placing an evaporant material within said source; operating a series of oil free pumps to reduce the pressure within said chamber from atmospheric to about 10^{-9} mm. Hg; operating said source to subject said material to a temperature less than the temperature at which said material evaporates to remove a portion of the gases sorbed therein; and further operating said source to evaporate said material while simultaneously operating a liquid helium pump positioned within said chamber to maintain said vacuum at about 10^{-9} mm. Hg during the time said material is evaporated and deposited upon said substrate by capturing desorbed gas molecules from said heated evaporant material, to thereby reduce the contamination of said substrate to less than 1 part in 10^5.

5. An improved method of forming a high purity coating upon a substrate comprising the steps of providing an evaporation source structure containing material to be evaporated within a vacuum chamber; mounting said

substrate vertically above said source; reducing the pressure within said chamber to about 10^{-10} mm. Hg by means of sequentially operating a first liquid helium pump and an electronic ion pump; subjecting said source to a first predetermined temperature at which a portion of the gases absorbed by said material is outgassed; subjecting said source to a second predetermined temperature at which said material vaporizes and deposits upon said substrate; and operating a second liquid helium pump positioned within said chamber adjacent said substrate during the time said material is evaporated to maintain the pressure within said chamber at about 10^{-9} mm. Hg by intercepting further desorbed gas molecules from said source and said material.

6. An improved method of attaining an ultra high vacuum within a vacuum chamber employed for thermal evaporation of materials comprising the steps of operating a water aspirator pump to reduce the pressure within said chamber to a few mm. Hg; isolating said aspirator pump from said chamber; filling said chamber with a helium and hydrogen free gas at atmospheric pressure; reoperating said water aspirator pump to again reduce the pressure within said chamber to a few mm. Hg; operating a first liquid helium pump to further reduce the pressure within said chamber to about 10^{-5} mm. Hg; subjecting said chamber to a predetermined temperature to remove a portion of sorbed gases therefrom; cooling said chamber; isolating said first helium pump from said chamber; operating an electronic ion pump to still further reduce the pressure within said chamber; outgassing said materials; operating a second liquid helium pump located within said chamber to reduce said pressure to about 10^{-10} mm. Hg; and thermally evaporating said materials; said second liquid helium pump being further effective to maintain the pressure within said chamber at about 10^{-9} mm. Hg during the time said materials are thermally evaporated by intercepting gas molecules desorbed from said thermally heated materials.

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