

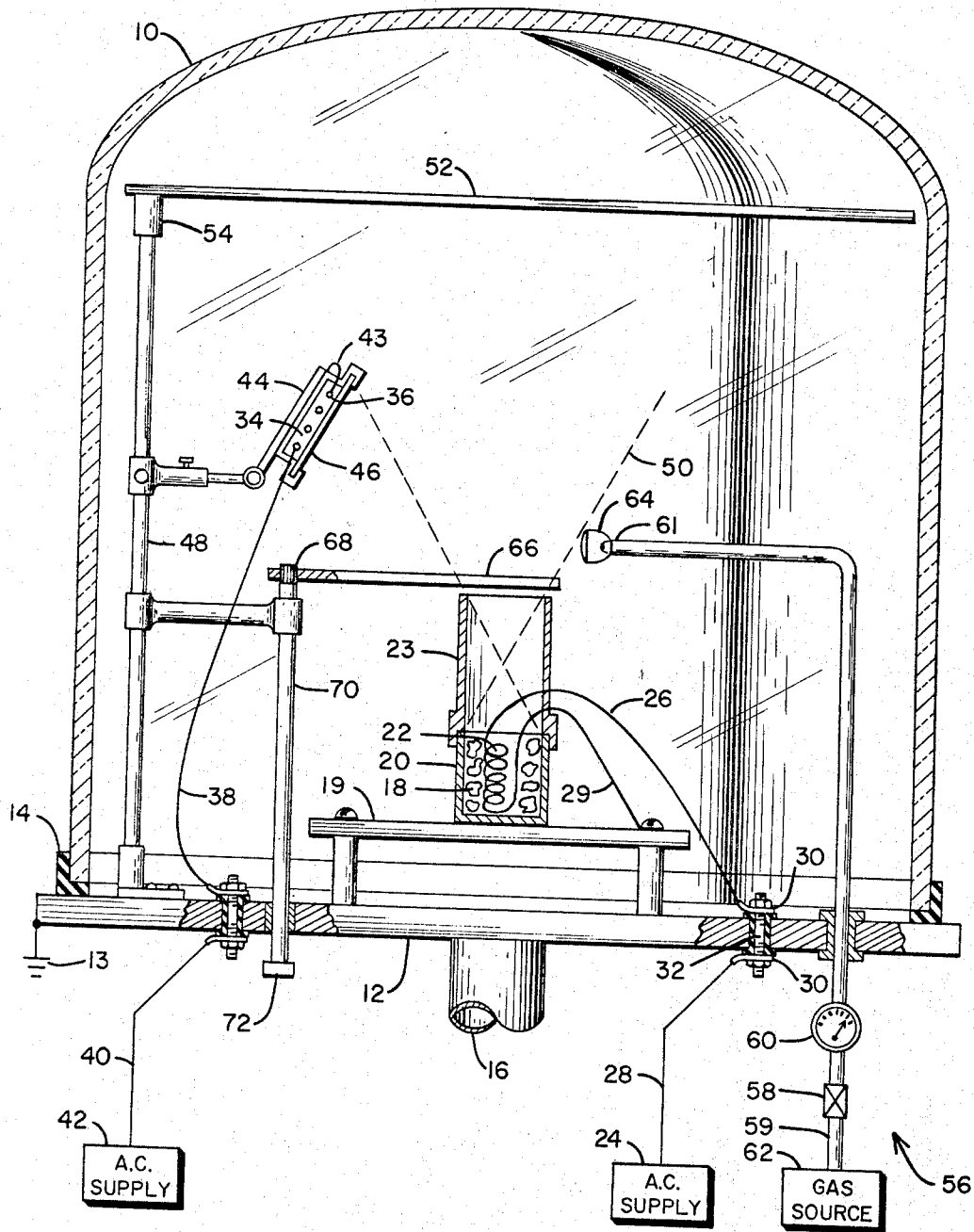
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DEFLECTING PARTICLES IN VACUUM COATING PROCESS

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DEFLECTING PARTICLES IN VACUUM
COATING PROCESS

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ABSTRACT OF THE DISCLOSURE

The use of a jet of inert gas in a thin film vacuum deposition apparatus to separate unduly large particles which may spatter from the melt from the evaporant to be deposited on the substrate.

The present invention relates generally to the evaporative fabrication of thin films in an evacuated chamber and more particularly to a method and apparatus for causing an evaporated material to deviate from its expected evaporant path.

The evaporative fabrication of a film of material within an evacuated chamber is a technique that has recently been employed in the electronics industry for fabricating electrical apparatus, such as, integrated circuits. Conventional integrated circuits usually are composed of several layers of electrically conductive films which in most instances are insulated from one another. Conventionally, the electrically conductive films and the electrically insulating films are formed within the evacuated chamber by vapor deposition techniques. Generally successful circuit design depends, in some degree, in each of the deposited layers having desirable uniform characteristics.

It has been found that when certain materials are employed to perform one or the other of the conductive or insulating functions of these layers it becomes difficult to consistently attain films exhibiting the desired uniformity. A major difficulty results from the low thermal conductivity of certain non-conductive materials, such as silicon monoxide, which, when heated develops unevenly hot areas which result in spattering. Spattering is characterized by the sudden ejection from the material being evaporated of solid or liquid particles. Although metals do not generally exhibit the problem of spattering during their thermal evaporation, some difficulty has been experienced when working with certain types of metals, for example, cadmium, zinc and magnesium. These large particles tend to cause "pin-hole" flaws in the deposited layer.

In the past, in order to reduce spattering, evaporation sources have been adapted to provide a structural arrangement for causing an evaporant to ascend to a substrate member by following a diverse path from its source. For effecting such diverse path, baffle plates have been interposed between the material in the evaporating container and the substrate upon which the evaporant is intended to be deposited. The baffle is effective to prevent the free flow of vapor between the baffle and the substrate by cutting off straight line paths between the material and the substrate. Spattering particles are trapped on the baffle surface whereupon they may be retained or subsequently re-evaporated therefrom. A baffle arrangement, however, for preventing the formation of nonuniform films, is often undesirable in that it can cause a reduction in evaporation rate and can become clogged by condensing vapor.

The formation of certain films such as metal oxides must often be accomplished by depositing the metal film and simultaneously or subsequently heating the entire sub-

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strate and any previously deposited films on the substrate to a high temperature in an oxygen atmosphere—a process which can lead to possible harmful effects on the substrate or on previously deposited films.

It is therefore an object of the invention to provide an improved method of evaporatively fabricating thin films of materials.

It is another object of the present invention to provide apparatus for reducing the effect of source spattering upon the deposited film layers.

It is a still further object of the present invention to provide apparatus which is effective to reduce the effect of source spattering upon the deposited film without impeding the evaporant path.

It is a still further object of the present invention to provide a method for separating mixed material in an evaporant.

It is yet another object of the invention to provide an improved apparatus for permitting the evaporative fabrication of both metallic and non-metallic films.

Yet another object of the invention is to provide a means of depositing films without influencing their properties by thermal effects caused by direct heat radiation onto the substrate from the vapor source.

Still another object of the invention is to provide a means of depositing certain homogeneous films of different composition from that of the vapor source without subjecting the substrate and/or any previously deposited films to possible undesirable treatment.

The above and other objects are accomplished in accordance with the method and apparatus of the present invention wherein there is provided vapor deposition apparatus which includes a gas introduction means for permitting the introduction of selected gas or gases into the evacuated chamber. The gas introduction means is arranged such that the gas emanating therefrom may be directed toward the evaporant path. Particles of evaporant are caused to be deflected from their normal path by the gas molecules. A vapor or evaporant particle rising from the source is deflected by a particle or gas molecule of the gas stream in accordance with the velocities, masses, and directions of the particles. In the case of vapor deposition of such materials as silicon monoxide, for instance, spattered off particles have a greater mass than the evaporated particles, and the spattered off particles are not deflected to the same degrees as the evaporated particles. By properly disposing of the substrate member within the apparatus, the deposition of undesirable particles or spattered-off pieces of the evaporant can be obviated. Accordingly, the resultant deposit would be free from the larger spattered particles and thus would exhibit a relatively smooth, homogeneous surface.

The novel features of the invention, as well as additional objects and advantages thereof, will be understood more fully from the following description when read in connection with the accompanying drawing, in which:

The figure describes a vertical section of the bell jar enclosure together with system components.

The apparatus shown in the figure sets forth an embodiment of the present invention, and includes a base plate 12 supporting the bell jar 10, which with gasket member 14 form an enclosure or chamber adapted to be evacuated by any suitable vacuum pump (not shown) coupled to the apparatus via pipe 16. The base plate is in a preferred manner constructed of stainless steel and grounded as shown at 13. Conventionally, the base plate is constructed of rigid material and the bell jar of glass. The material to be evaporated and subsequently condensed, that is, for example, the silicon monoxide melt 18, is contained in the crucible 20 which is provided with a helically wound tungsten resistance heating coil 22 for

the mouth of the source 20 so that the silicon monoxide vapor cannot be deposited directly onto the substrate and so that heating of the substrate by radiation from the source is minimized. As observed from the figure, the substrate is positioned out of the line of sight of the melt 18. The crucible and shield are preferably constructed of tantalum which, as a refractory material is capable of withstanding high temperatures as well as exhibiting low vapor pressure characteristics essential in a vacuum chamber to prevent contamination. The crucible and heating coil cooperate to form a treating means. Containers other than crucibles may be employed to hold the material to be melted and heating means other than a resistance heated filament may be employed to heat the melt. Current for the heating element may be supplied by an external source 24 by way of conductors 26 and 28 secured to conductive elements 30 in turn secured to a bolt-like conductor plug 32 sealed to the base plate 12 by appropriate sealing means. Conductor 29 is a ground return from the resistance coil and is connected to table 19. It is not intended, however, that the conductor member 32 (plug) is to be limited to the particular configuration illustrated inasmuch as a wide variety of arrangements are suitable. The power output of the heater filament 22 is controlled either automatically or manually and the output may be continuously varied, for example, from 5% to 90% of maximum power. Crucible 20 is located on a table member 19 suitably positioned on the base plate 12. Disposed to the left of the melt material 18 and in the upper portion of the bell jar is a substrate heater 34 provided with heating elements 36, preferably made of tungsten, connected to leads 38 and 40 and power supply means 42 via a bolt-like plug as aforementioned. Beneath the heater and adjacent thereto is a holder 44 for holding a substrate 46 in a predetermined position. Conductor 43 connected to holder 44 provides a ground through support member 48. Various substrate materials may be used, the instant embodiment employing glass, and upon this glass substrate, or upon layers previously formed upon this glass substrate, the evaporated material is allowed to condense in a designed manner. A mask (not shown) may be used for defining one or more films and may be held in contact with, or at a predetermined distance from the substrate 46 by holder 44. The holder which supports the substrate 46, may be held in any convenient manner, as by the support 48. The substrate holder may be designed to be adjustable to a variety of positions and is preferably constructed of stainless steel.

The normal evaporant path may be defined by a cone 50 projected from a point slightly above the melt surface to the slag deposit shield 52. The slag shield may be secured to the post 48 such as by bracket means 54 or by any other suitable means.

On the right-hand side of the vapor source and between the slag shield 52 and the crucible, there is provided a gas introduction means 56. The gas introduction means 56 includes a variable leak valve 58 in suitable tubing 59, pressure gauge means 60, and jet means 61 attached to a gas source 62. As representative of dimension, no limitation intended, the distance between the upper edge of crucible 20 and the slag shield 52 may be 18 inches, and the gas introduction jet is disposed in the bell jar such that the stream of the gas particles intercepts the evaporant cone at a predetermined distance above the vapor source to produce the desired deflection. The jet of gas emanating from the introduction means is similarly described as or represented by a cone projected from the orifice of the introduction means through nozzle element 64 which functions to direct the gas in a desired manner toward the evaporant. The nozzle configuration may be designed in accordance with one offering the desired result. The substrate 46 is angularly disposed with respect to the crucible 20 and located outside of the normal evaporant path and accordingly evaporant is normally precluded from depositing onto the substrate.

It is also evident that by suitably adjusting the substrate by the support 48, spattering onto the substrate may be precluded. Normal evaporant travels in straight lines, and as observed in the figure, the evaporant cannot see the substrate because of the shield 23. However, it is still possible for spattering to occur, since a spattered particle travels in a trajectory due to gravity and hence may travel in an arc-like trajectory to the substrate. Hence a suitable adjustment of the substrate holder prevents the spattered particles from striking the substrate. The slag shield 52 is effective to prevent evaporant from haphazardly coating the upper portion of the bell jar and other apparatus contained therein. In operation, the material 18 in the evaporation source 20 is heated by applying power from the supply means 24 to heating element 22 and raising the temperature of the material above its vaporization point, for example, in the case of silicon monoxide, to a temperature of approximately 1200 degrees centigrade. This is done while maintaining the pressure in the bell jar at approximately 1 times 10 to the minus 6 millimeters of mercury. When the desired evaporation temperature is reached, and it appears that such temperature has reasonably stabilized throughout the melt, a shutter member 66 above the melt is rotated outside of the evaporant cone area and the melt or evaporant is permitted to deposit on the slag shield 52. The shutter member 66 is mounted at one end upon a serrated gear portion 68, such gear portion being integral with the shaft 70 which is turned by applying a force to knob 72. Since any other suitable arrangement for the shutter may be used, there is no intention to limit the configuration to the specific one illustrated. At this time the gas introduction means 56 is permitted to cause a flow of gas to intercept the evaporant. In the preferred embodiments, argon gas is employed, although other inert gasses may be used in lieu thereof, such as xenon, krypton, neon, helium, or radon. Although the mass of the argon atom is slightly less than that of the silicon monoxide molecule, there is sufficient momentum exchange for deflection of silicon monoxide molecules to occur. Depending upon the velocity of the argon and silicon monoxide streams, the deflected silicon monoxide emerges at an angle between the horizontal and vertical, but preferably strikes the substrate at a right angle to prevent angle of incidence effects during deposition.

Silicon monoxide films or layers deposited while utilizing the method of the present invention are free from spattered particles and are not influenced by heat radiated directly from the source or by varying deposition rates caused by clogging baffles which can be a problem when depositing in the normal manner.

Additionally, an active gas such as oxygen can be used as the deflecting beam resulting in the deposition of a different vapor such as SiO_2 instead of SiO . The process of deflecting the original vapor beam automatically assures that a majority of the deflected particles come into contact with the deflecting beam particles resulting in a homogenous deposited film of different composition from the original vapor beam. The chemical reaction between the deflecting beam and the evaporant beam in the vapor before it reaches the substrate aids in the assurance that the resulting deposit upon the substrate is one of a homogeneous nature. It is not necessary to heat the substrate excessively in order to promote the chemical reaction.

It is understood that suitable modifications may be made in the structure as disclosed provided that such modifications come within the spirit and scope of the appended claims. Having now, therefore, fully illustrated and described my invention, what I claim to be new and desire to protect by Letters Patent is:

What is claimed is:

1. The method of indirect vacuum deposition of an evaporant upon a substrate comprising the steps of:
 - (a) heating a material within an evacuated enclosure to produce an evaporant,

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- (b) introducing a gas against the evaporant to thereby create a momentum exchange between the evaporant and the gas causing only evaporant particles below a predetermined size to deflect and deposit upon said substrate.
2. In a method of vacuum depositing a smooth homogeneous layer of evaporant free of larger particles of evaporant, the method comprising the steps of:
- (a) locating a substrate within an evacuated enclosure above a treating means and to one side of a conical path extending above an upper surface of said treating means,
- (b) heating a deposition material to cause evaporation thereof as evaporant,
- (c) introducing a gas stream against the evaporant at a predetermined point along a path thereof to thereby cause a deflection of only the evaporant particles which are below a predetermined mass path a predetermined amount to cause deposition upon the substrate the particles above said predetermined mass failing to impinge on said substrate.
3. The method defined in claim 1 wherein said evaporant intercepts the substrate at an angle of substantially 90° with respect thereto.
4. The method of claim 1 wherein the evaporant is silicon monoxide.
5. The method of claim 1 wherein the gas is selected from a class which does not react chemically with said evaporant.
6. The method of claim 1 wherein the gas reacts chemically with said evaporant.
7. A method of forming thin film layers on a substrate which is substantially free from defects comprising the steps of

- (a) positioning a source of material to be vaporized within a vacuum deposition chamber;
- (b) positioning a substrate onto which the material is to condense out of direct line of sight of said source such that vapor particles produced by the heating of said material normally fail to impinge on substrate;
- (c) heating said source to a temperature causing said material to vaporize; and
- (d) introducing a suitable gas in a well defined jet into said chamber at a location where said gas ballistically deflects said vapor particles below a predetermined size onto said substrate, the method being such that particles above said predetermined size fail to impinge on said substrate.
8. The method as in claim 7 wherein said gas is chosen from the class including argon, xenon, krypton, neon, helium and radon.
9. The method as in claim 7 wherein said gas is oxygen.

References Cited

UNITED STATES PATENTS

2,831,784	4/1958	Gastinger	117—106
2,920,002	1/1960	Auwarter	117—106 X
2,996,418	8/1961	Bleil	117—106 X
3,113,040	12/1963	Winston	117—106 X
3,208,873	9/1965	Ames et al.	117—106
3,237,508	3/1966	Keller et al.	
3,243,363	3/1966	Helwig	117—217 X

OTHER REFERENCES

IBM Technical Disclosure Bulletin, vol. 4, No. 6, December 1961, p. 6 relied upon.

ALFRED L. LEAVITT, *Primary Examiner*.

A. GOLIAN, *Assistant Examiner*.