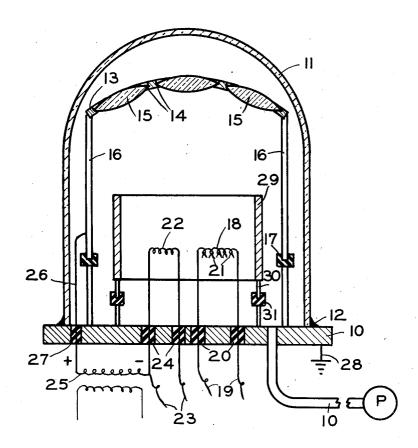
METHOD FOR DEPOSITING THIN FILMS

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## METHOD FOR DEPOSITING THIN FILMS

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This invention relates to the deposition of thin films of materials on a surface of a body in a vacuum and more particularly it has reference to a method for treating the surface prior to, during, and after deposition of the material.

Thin films of various kinds of materials, such as aluminum, rhodium, magnesium fluoride, cryolite, and zinc sulfide, are now widely used in the optical industry. Metallic films are demirrors while thin layers of magnesium fluoride, cryolite, and zinc sulfide are used on the surfaces of optical elements in order to decrease the reflection of light and thereby increase the transmission of light through the elements. It is 15 necessary that the surfaces be treated before the deposition of such materials in high vacuum in order to remove the adsorbed gases, water vapor, and other material in order to provide a durable, tenacious film. This treatment is generally ac- 20 complished either by subjecting the surface to a glow discharge in a vacuum having a pressure greater than about 10 microns or by heating the surface by means of suitable heating elements positioned within the evacuated chamber. Another method of treating the surfaces prior to the deposition of the material involves the bombardment of the surface by a stream of electrons which is carried out in a vacuum under pressures tion for U.S. patent, Serial No. 555,097, filed by Richard M. Rice, and issued on May 20, 1947 as Patent No. 2,420,724.

When the glow discharge methods are used, some of the positive ions collide with the metal 25 parts within the chamber and so act to sputter off some of the metallic material which then becomes deposited on the surface which is being treated so that the subsequent deposited layer of material exhibits an undesirable graininess. As 40 the pressures are reduced, the amount of such sputtering is diminished. With the electronic bombardment method as described in said application, some sputtering also occurs so that the final layer of material has a grainy structure. 45 Such a grainy structure is, of course, undesirable in the case of a mirror because the graininess tends to scatter the light and so affects the image forming characteristics of the mirror.

electronic bombardment methods is due to the fact that an electron may collide with a wandering gas molecule and form a positive ion which would be attracted to the metal table or other and so act to sputter off material from these parts.

Accordingly, it is an object of this invention to provide a method whereby the foregoing disadvantages may be eliminated when a surface is treated by electronic bombardment prior to the deposition of material thereon. A further object is to provide an improved method for depositing thin films of material on a surface of a posited on glass or plastic surfaces to provide 10 body in a vacuum. Another object is to provide a method whereby improved films of material may be deposited on a commercial scale on various types of surfaces in a vacuum. These and other objects and advantages will be more fully described and pointed out in the appended claims.

> The drawing is a diagrammatic view of an apparatus which can be used for practising our method.

Our method may be carried out by the apparatus shown in the drawing wherein a metal base plate 10 supports the glass bell jar 11 with the juncture therebetween sealed by suitable vacuum-tight gasket means 12. Mounted within 25 the chamber is the work support which comprises a metal plate 13 having seats 14 for holding the work such as lenses 15. The plate 13 is supported by metal rods 16 which are electrically insulated from the plate 10 by means of the insuof about 0.01-10 microns as disclosed in applica- 30 lators 17. The bell jar may be evacuated by means of a vacuum pump P connected to the chamber by means of conduit 19'.

A suitable heating coil 18 having leads 19 passing through insulating bushings 20 in plate 10 is adapted to support the material which is to be vaporized, such as the loops of aluminum wire 21. The leads 19 are connected to a suitable source of electric current so that the material 21 may be heated and vaporized.

In order to provide a source of electrons in the chamber for the bombardment of the lens surfaces prior to the evaporation of the material, there is provided a tungsten wire filament 22 which may be heated by a suitable electric current passing through leads 23 which are insulated from the plate 10 by bushings 24. A high voltage transformer 25 has one of its secondary terminals connected with the rod 16 by means of a lead 26 passing through the bushing 27 in plate The sputtering action which is present with 50 10. The other secondary terminal is connected to one of the leads 23 which supply current to the electron emitting filament 22. The transformer or rectifier 25 delivers from 2,000-15,000 volts pulsating filtered D. C. or A. C. The positive negatively charged metal parts in the chamber 55 terminal is connected to the rod 16 so that the

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work holder 13 and the lenses 15 are at a positive potential. The base plate 10 is connected to the ground by lead 28. A cylindrical metal shield 29, supported on rods 30 and insulated from the base 10 by insulators 31, prevents the deposition of material on the rods 16 and bushings 17.

In one method of practising our invention, the lenses 15 are mounted on the work holder 13 and the chamber is evacuated to a pressure of about 10 0.1 micron of mercury. The transformer 25 is energized so that a potential of about 2,000-15,000 volts is applied to the work holder 13. The electron emitting filament 22 is also energized so that electrons therefrom bombard the 15 surfaces of the lenses 15. After the filament 22 has been energized for about eight to ten minutes, the filament 18 is energized so as to evaporate the material 21. When the material has been evaporated and condensed to the de- 20sired thickness on the lenses is, the current is shut off from the filament 18 but the emission of electrons from the filament 22 is preferably continued for two or three minutes longer. All current supplies are then disconnected, the vacuum 25 released, and the coated lenses removed.

It will thus be apparent that the electrons which are emitted by the filament 22 under high velocity will pass upwardly to the work holder 13 and the lenses 15. If an electron collides with 30 a gas molecule and forms a positive ion, the latter will be attracted to the filament 22 since it is the only negatively charged part in the chamber when D. C. is used. The electrons from the filament 22 will collide with the positive ions and 35will drive them away from the filament or will combine with them and form a gas molecule which, having no charge, wanders off into the system. When A. C. is used, the plate 13 is positive for a half cycle during which electrons  $^{40}$ from filament 22 pass towards the plate 13. During the other half cycle, the plate 13, being negative, does not attract the electrons from the filament 22 and hence there is less chance for collisions between electrons and gas molecules 45 and so less chance for the formation of positive ions which would cause sputtering. Since the positive ions will thereby be substantially prevented from striking the exposed metal parts in the chamber, there will be little or no sputtering 50 of such metal parts and hence such metal will not be deposited on the lens surfaces. As a consequence, the layer of material which is subsequently evaporated and condensed onto the lens surface 15 will be homogeneous in character 55 and substantially free from the undesired grainy

Our improved method has been used successfully in the commercial production of metallic mirrors which are not only hard and durable but 60 are also free from graininess so that they will provide specular reflection of light rays rather than diffused reflection. Such types of aluminum mirrors have, for example, been commercially used in television sets. Our method 65 has also been successfully used for the produc-

tion, on a commercial basis, of anti-reflection films of magnesium fluoride, cryolite, and zinc sulfide on lens surfaces.

From the foregoing, it will be apparent that we are able to attain the objects of our invention and provide an improved method for depositing thin films of material on surfaces of a body in a vacuum. By means of our method, durable, corrosion-resistant films may be deposited and such films will have the further advantage that their structure is more homogeneous than that of the films deposited by prior art methods. Various modifications can obviously be made without departing from the spirit of the invention as pointed out in the appended claims.

#### We claim:

1. A method of depositing a film of material on a surface of a body in an evacuated chamber having metal parts exposed therein which comprises the steps of bombarding the surface with electrons from a source, maintaining the metal parts at a potential which is positive relative to said source during the bombardment of the surface whereby positive ions formed in the chamber are substantially prevented from striking the metal parts and depositing a film of material on the surface by thermal evaporation immediately after the bombardment of the surface.

2. A method of depositing a thin film of material on an optical surface of a body in an evacuated chamber having metal parts exposed therein which comprises the steps of bombarding the surface with a stream of electrons from a source while maintaining the pressure in the chamber at about 0.01–10 microns and maintaining said metal parts at a potential which is positive relative to the potential of the source whereby positive ions formed in the chamber will be substantially prevented from sputtering material from said parts onto the surface and thereafter depositing the film of material on the surface by thermal evaporation in the evacuated chamber.

3. The invention as set forth in claim 2 wherein the thin film of material is formed of magnesium fluoride.

4. The invention as set forth in claim 2 wherein the thin film of material is formed of aluminum.

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### REFERENCES CITED

The following references are of record in the file of this patent:

## UNITED STATES PATENTS

+	Number	Name	Date	
	2,256,771	Berghaus et al	Sept. 23, 19	)41
	2,383,469	Colbert	Aug. 28, 19	45
	2,391,595	Richards et al	Dec. 25, 19	<del>)</del> 45
	2,420,724	Rice	May 20, 19	<del>)</del> 47
	2,447,979	Hansel et al	Aug. 24, 19	)48
	2,456,708	Kellogg	Dec. 21, 19	)48