Nov. 23, 1937. P. ALEXANDER 2,100,045

DEPOSITION OF METALLIC FILMS FROM METAL VAPORIZED IN VACUO

Filed Oct. 10, 1936

FIG. 1.

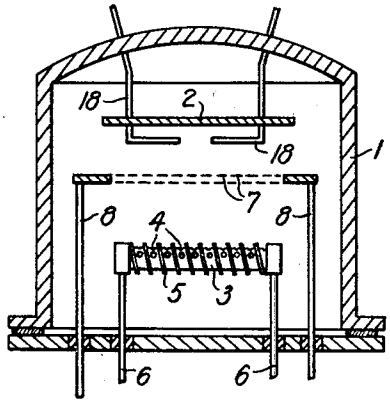


FIG. 3.

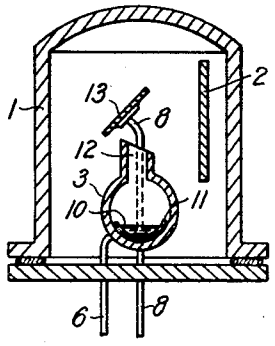


FIG. 4.

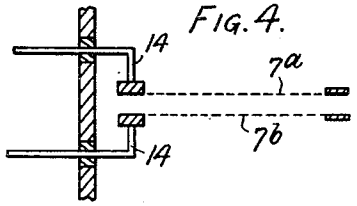


FIG. 2.

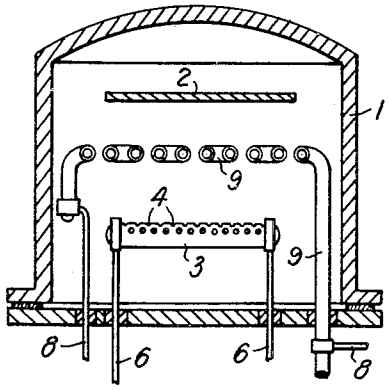


FIG. 5.

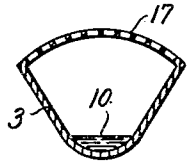
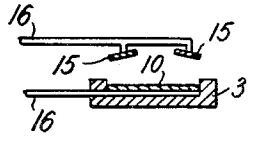


FIG. 6.



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DEPOSITION OF METALLIC FILMS FROM METAL VAPORIZED IN VACUO

Paul Alexander, Brussels, Belgium

Application October 10, 1936, Serial No. 105,091
In Germany October 12, 1935

19 Claims. (Cl. 91—12.2)

This invention relates to the deposition of metallic films from metal vaporized in vacuo.

In order to obtain a film which has the characteristics of the metal and adheres firmly to its support, it has heretofore been found necessary to effect the vaporization and deposition in an enclosure with high vacuum, of the order of 1/10,000 mm. or lower. To obtain and maintain so high a vacuum commercially presents great difficulties. If a lower vacuum be employed, the film obtained is subject to some or all of the following defects; it does not adhere well, it is porous, its reflecting power is small, its electrical conductivity is low, it is dark in color and, by transmitted light shows colors indicating a colloidal state.

According to the invention, films are deposited at substantially higher pressures than 1/10,000 mm. which have the characteristics of films hitherto deposited at this pressure, by heating the metallic vapor in its passage from its source to the support of the film.

It is believed that the reason why this heating of the vapor is effective in producing a good film at higher pressures is the following:—The partial pressure of the metal vapor close to the source of evaporation is higher than that of the residual gas in the vacuum chamber. The partial pressure of the metal vapor, however, diminishes rapidly with the distance from the source of evaporation. The metal atoms, in their passage from source to support, lose kinetic energy as the result of collisions with the molecules of the relatively cold residual gas. Metal atoms of low kinetic energy, colliding with each other, tend to coalesce to form particles of colloidal dimensions, which, when deposited on the support, form a defective film. When, according to the invention, heat is communicated to the metal vapor at an intermediate point of its passage from source to support, kinetic energy is imparted to the atoms, and coalesced particles are reevaporated into atomic form. In the further passage from this intermediate point to the support, the partial pressure of the metal vapor is much lower than it is from the source to the intermediate point, and is low in comparison with that of the residual gas; collisions of the metal atoms are then with gas molecules rather than with other metal atoms, and the metal vapor consequently completes the passage substantially in atomic form and is in this form deposited on the support.

In the accompanying drawing:—

Figures 1, 2 and 3 are vertical sections through alternative forms of apparatus for carrying out the invention, shown partly diagrammatically;

Figure 4 is a part section of an alternate form of vapor heating device;

Figure 5 is a section through an alternative form of heating vessel, and

Figure 6 is a section through an alternative form of heating device for the metal.

Referring to Figure 1, the vacuum chamber 1 contains the support 2 on which the metal film is to be deposited, and the heating vessel 3 contains the metal to be vaporized. The supports for the various parts in the chamber are not shown in the drawing. The heating vessel 3 is a refractory tube having holes 4 for the issue of the metal vapor. The vessel 3 is heated by current passed through the spiral 5 from the conductors 6. Between the heating vessel 3 and the support 2 is a device for heating the metal vapor, consisting of two wire mesh screens 7 connected with conductors 8 whereby current is passed through the screens to heat them to at least the vaporization temperature of the metal to be deposited. The metal vapor issuing from the holes 4, passes through the meshes of the screens 7 in its passage to the support 2, and is heated by the screens, and is deposited on the support 2 in the form of a film of good quality.

By means of the vapor heating screens 7, films have been deposited, at a chamber pressure of 1/100 mm. and even higher, which are of as good quality as films heretofore deposited at pressures of 1/10,000 mm. and lower.

Figure 2 shows an alternative form of heating vessel 3 and an alternative form of vapor heating device. The vessel here consists of a tube of metal of high melting point, such as tungsten or molybdenum, which itself forms the heating element through which current is passed by the conductors 6. The vapor heating device consists of a tube 9 bent in zig-zag, supplied with a gas inert to the metal vapor at its right hand end and closed at its left hand end. The tube 9 may itself form the heating element, as shown in the drawing, current being supplied to it by the conductors 8. In this case, the tube may be made of palladium, and the gas supplied may be hydrogen, to which the walls of the tube are porous. Alternatively, the tube may be made of a porous refractory material, and heated by a spiral wire heating element. The heated gas issuing through the walls of the tube 9 heats the metal vapor in its passage between the zig-zags of the tube. The quantity of gas introduced must not be so large as unduly to increase the vaporization temperature of the metal. Even without continuous evacuation, sufficient gas can be introduced to heat the metal vapor effectively

without substantially increasing the vaporization temperature.

Figure 3 shows an alternative form of apparatus, in which the metal 10 to be vaporized is contained in a heating element 11, supplied with current by conductors 6 (of which one is visible), contained in the vessel 3. Above the outlet 12 of the vessel 3 is a metal plate 13 which is heated to a temperature at least equal to the vaporization temperature of the metal to be deposited, by current passed through it from conductors 8 (of which one is visible). The metal vapor issuing from the outlet 12 impinges against the heated plate 13 in its passage to the support 2 and is heated thereby.

The heating of the vapor heating device, such as the wire mesh screen 7 may be effected as shown in Figure 4, in which the screen is in two parts 7a and 7b insulated one from the other and connected to high tension conductors 14, whereby a glow discharge is maintained between the two parts.

The metal may be vaporized by glow discharge, when its vaporization temperature is low enough, as shown in Figure 6, where the metal 10 is contained in a refractory vessel 3, while above it are located plates 15; the plates 15 and the metal 10 are connected to high tension conductors 16, whereby a glow discharge is maintained between the plates and the metal.

It is of advantage, in order that the partial pressure of the metal vapor may be kept low, that its concentration at its point of issue from the heating vessel be as low as possible. For this purpose a heating vessel such as that shown in cross-section in Figure 5 is of advantage, in which the cross-sectional area (that is to say, the space for the vapor) increases from the surface 10 of the metal to the outlet 17.

Another device for diminishing the concentration of the vapor from its source is to vaporize the metal from an alloy or mixture of the metal with another having a substantially lower vapor pressure. By way of example, cadmium may be vaporized from an alloy of 10% cadmium and 90% tin. At a pressure at which cadmium vaporizes at about 600° C., the vapor pressure of tin is negligibly small.

The residual gas in the chamber should be, as usual, inert with respect to the metal vapor, and it is of advantage that it be ionized. Thereby a good film is obtained at higher gas pressure or with more rapid vaporization. The ionization may be produced by electrodes 18 (Figure 1) within the chamber 1, to which electric potential is applied.

By means of the invention applicant has produced films of good quality with a gas pressure in the vacuum chamber exceeding 1/100 mm., but the maximum pressure which can be used depends on the rate of vaporization, the degree of heating of the vapor in its passage to the support, the vapor pressure of the metal used and whether the gas is ionized, and, for any given set of conditions, can best be ascertained by trial.

Having described my invention I declare that what I claim and desire to secure by Letters Patent is:—

1. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal and means located between the source of the vapor and the support adapted to heat the metal vapor.

2. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, a metallic member adapted to allow the metal vapor to pass through it, interposed between the source of the vapor and the support, and means for heating the metallic member to a temperature above the vaporization temperature of the metal.

3. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, a wire mesh screen located between the source of the vapor and the support, and means for heating the wire mesh screen to a temperature above the vaporization temperature of the metal.

4. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, a metallic member so located that the vapor issuing from the source impinges upon it in the passage of the vapor to the support, and means for heating the metallic member to a temperature above the vaporization temperature of the metal.

5. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, a metallic member adapted to allow the metal vapor to pass through it, interposed between the source of the vapor and the support, an electrode, and means for producing a glow discharge between the metallic member and the electrode.

6. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, two wire mesh screens located between the source of the vapor and the support and means for producing a glow discharge between the two wire mesh screens.

7. Apparatus as in claim 1, comprising two electrodes and means for producing an electric discharge between them adapted to ionize the gas in the chamber.

8. Apparatus as in claim 2, comprising two electrodes and means for producing an electric discharge between them adapted to ionize the gas in the chamber.

9. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising a vessel adapted to contain the metal to be vaporized and means for supplying heat to the metal to vaporize it, the vessel being so shaped that its cross-sectional area increases from the surface of the metal to the outlet, and means located between the outlet and the support adapted to heat the vapor.

10. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising a metallic vessel adapted to contain the metal to be vaporized, means for passing an electric current through the vessel to heat it to a temperature above the vaporization temperature of the metal, and means located between the outlet of the vessel and the support adapted to heat the vapor.

11. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising a vessel adapted to contain the metal to be vaporized, an electrode, means for producing a glow discharge between the metal and the electrode, adapted to heat the metal to vaporize it, and means located between the outlet of the vessel and the support adapted to heat the vapor.

12. Apparatus as in claim 9, comprising two electrodes and means for producing an electric discharge between them adapted to ionize the gas in the chamber.

13. Apparatus as in claim 10, comprising two electrodes and means for producing an electric

discharge between them adapted to ionize the gas in the chamber.

14. Apparatus as in claim 11, comprising two electrodes and means for producing an electric discharge between them adapted to ionize the gas in the chamber.

15. Process of depositing a metal film on a support in an evacuated chamber, comprising the steps of vaporizing the metal and heating the vapor in its passage from its source to the support.

16. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, a tubular member interposed between the source of the vapor and the support, means for heating the tubular member to a temperature above the vaporization temperature of the metal and means for supplying into the tubular member a gas inert to the metal, the walls of the tubular member being porous to the gas, the rate of introduction of the gas being so low that the vaporization temperature of the metal is not substantially increased by reason of rise of pressure in the chamber.

17. Process of depositing a metal film on a support in an evacuated chamber, comprising the steps of embodying the metal to be deposited in a comparatively large portion of another metal, having, at the pressure in the chamber, a vapor pressure low in comparison with that of the metal to be deposited, heating the combined mass of metal to vaporize the metal to be deposited, and without appreciably vaporizing the other metal, heating the vapor in its passage from its source to the support.

18. Apparatus for depositing a metal film on a support in an evacuated chamber, comprising means for vaporizing the metal, means for introducing a gas inert to the metal into the space between the source of the vapor and the support, the quantity of the said inert gas at any time in the chamber being insufficient to raise materially the vaporization temperature of the metal, and means for heating the gas.

19. Apparatus as in claim 18, comprising two electrodes and means for producing an electric discharge between them adapted to ionize the gas in the chamber.

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