

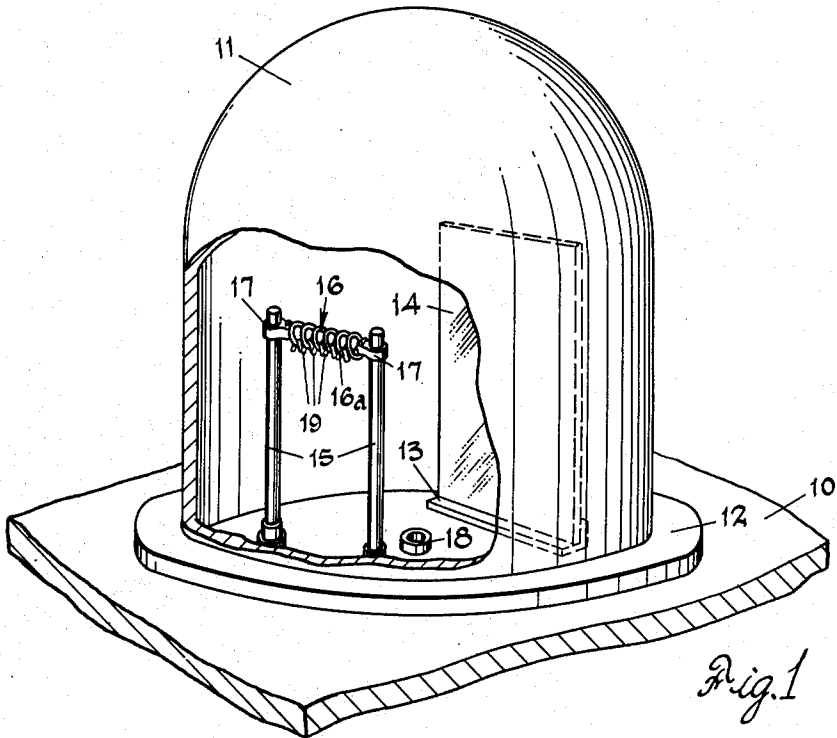
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A. R. WEINRICH

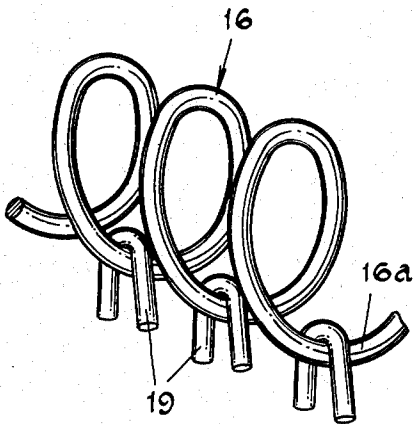
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METHOD OF VAPOR DEPOSITING COATINGS OF ALUMINUM

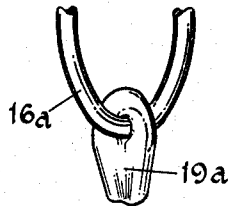
Original Filed Dec. 28, 1948



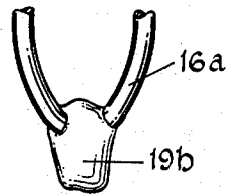
*Fig. 1*



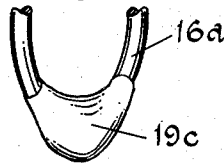
*Fig. 2*



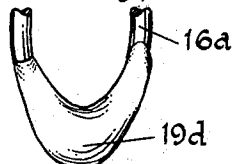
*Fig. 3*



*Fig. 4*



*Fig. 5*



*Fig. 6*

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## METHOD OF VAPOR DEPOSITING COATINGS OF ALUMINUM

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Original application December 28, 1948, Serial No. 67,610. Divided and this application June 21, 1951, Serial No. 232,700

2 Claims. (Cl. 117—107)

This invention relates broadly to improvements in metal evaporation and more particularly to the thermal evaporation of aluminum alloys in the forming of mirrors and other coatings.

The present application is a division of my copending application Serial No. 67,610, filed December 28, 1948, and entitled "Aluminum Base Alloy for Metal Evaporation," which has been abandoned.

In metal evaporation, the metal to be evaporated is usually hung on a filament of tungsten, molybdenum, tantalum, or columbium as small loops or pieces of wire. Electric current is then passed through the filament to heat it and the wire pieces are melted whereupon the fused metal clings to the filament and evaporates as the heating is continued.

In the application of mirror and other coatings by evaporation within a vacuum as known in the thermal evaporation art, considerable spoilage is encountered by small chunks or particles of metal which tend to spatter or fly off the electric filament and cause rough spots upon the mirror or other article being coated, rendering it unfit for commercial use. This highly objectionable condition is frequently encountered when using pure or commercially pure aluminum and is customarily referred to in the art as "spitting."

The primary object of this invention is to improve the thermal evaporation of aluminum and its alloys so as to minimize, if not entirely eliminate, objectionable spattering or spitting of the metal during evaporation.

Another object of the invention is to overcome spitting of the metal during evaporation by the provision of a special alloy of aluminum containing a relatively small amount of molybdenum and the balance substantially aluminum.

The use of such a special alloy of aluminum with molybdenum has been found to eliminate the spoilage from spitting or explosive boiling such as is found to a considerable degree when evaporating pure or commercially pure aluminum.

Other objects and advantages of the invention will be apparent from the following description and claims.

In the drawings:

Fig. 1 is a perspective view, partially broken away, of one type of thermal evaporation apparatus;

Fig. 2 is a perspective view of a portion of an electric filament showing the application of the metal alloy to several of the coils thereof; and

Figs. 3, 4, 5 and 6 are detail diagrammatic views illustrating the several phases of wetting or coating of the filament by the alloy of this invention.

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Suitable apparatus which may be employed in carrying out the invention is illustrated in Fig. 1 and comprises a supporting base 10 upon which is mounted a housing shown as a whole at 11. The housing 11 may be in the form of a bell-jar or the like having a dome-like or semi-spherical top portion or enclosed end and a bottom open end having a surrounding flange or projection 12 which is adapted to rest upon the upper surface of the supporting base 10.

Within the chamber provided by the housing 11 is a suitable work support 13 for supporting a work piece such as a plate or piece of glass, plastic, plaster, paper, porcelain, metal, or the like 14, in upright position.

Also located within the chamber and mounted upon the supporting base 10 is a pair of upright supporting posts 15 between which is carried in substantially horizontal position an electric filament 16. The filament, as shown, is in the form of a coiled wire made of tungsten, molybdenum, columbium, or tantalum, whose opposite ends are attached to brackets 17 mounted upon the supporting posts 15 and adjustable thereon so as to vary the position or location of the filament 16 with relation to the supporting base 10.

The chamber provided by the housing 11 may, if desired, be completely evacuated of air through outlet pipe or conduit 18 and have a high vacuum created therein by means of suitable air evacuating and vacuum creating means such as a pump (not shown).

In accordance with the invention, which is carried out within the chambered housing 11, short pieces or loops 19 of the special aluminum alloy herein provided and consisting of a relatively small amount of molybdenum with the balance substantially all aluminum, are bent and hung on the convolutions 16a of the filament 16 in the manner shown.

When pure or commercially pure aluminum such as 98.5% aluminum is applied to a thermal evaporation filament and the filament energized by applying a heavy current thereto, the small pieces of aluminum receive their heat by contact with the filament and by heat conduction from the filament down through the metal pieces. Under such circumstances, the filament is invariably at a much higher temperature than the aluminum at the time the aluminum reaches its melting point of 658° centigrade. Thus, depending upon the construction of the filament and the amount of electrical current applied thereto, the filament may be at a temperature in the range of 1000 to 2000 degrees centigrade, in order to transfer sufficient heat to the aluminum and to evaporate it in a reasonable time.

Pure and commercially pure molten aluminum wets by capillary attraction the metals tungsten, molybdenum, tantalum and columbium which are normally employed as evaporation filaments and such wetting occurs very rapidly. In fact, when the aluminum pieces applied to the filament become molten, they wet the filament so rapidly that the relatively cold molten aluminum flows out instantly over the much hotter filament surfaces originally not contacted by the aluminum pieces and as these hotter surfaces are above the temperatures required for forming aluminum vapor in the vacuum employed, the advancing molten aluminum is explosively vaporized. That portion of the advancing molten aluminum flowing by capillary attraction over and in direct contact with the very much hotter filament surfaces is the portion which

is immediately vaporized and which acts to blow away the overlying liquid aluminum by the explosive boiling conditions thus generated. This spitting results in aluminum masses being deposited upon the surface being coated as small lumps or chunks and such spitted aluminum leads to an unsatisfactory coating and a spoiled article. This action is similar to the explosive boiling and spitting which occurs when water is flowed over a hot stove.

However, when pieces of the special aluminum alloy of this invention containing molybdenum are applied to similar thermal evaporation filaments and the filaments similarly heated, the alloy, upon melting, supplies molten metal to the hot filament surfaces not originally contacted thereby at a relatively much slower rate. In other words, the inclusion of the molybdenum acts to retard the speed at which the aluminum alloy feeds out to the filament. While the alloy seems to be of the same surface tension and good wetting character for the filament metals as with pure or commercially pure aluminum, the alloy does not melt as a whole to a simple molten liquid as with pure or commercially pure aluminum, but gives a melt in which many small crystals of an unmelted material are present.

More particularly, the alloy melts first to a semi-pasty mass indicated at 19a in Fig. 3, and such pasty character retards the flow of molten metal by capillary attraction onto the hotter surrounding filament surfaces. This retardation also permits a longer time for heat to flow from the filament by conduction into the pasty mass 19a and its temperature is thus raised to a higher temperature so that the mass temperature and any small amounts of molten metal leaking out of the mass onto the hotter filament surfaces are relatively closer to the actual temperature of such surrounding filament surfaces. By reducing the temperature differential between the molten metal mass 19a and the surrounding portions of the filament upon which it later moves out upon, the danger of explosive boiling or spitting is minimized.

In Fig. 4, the pasty melted alloy mass 19b has become hotter than as shown in Fig. 3 and from such a pasty mass the molten phase begins to leak out upon the surrounding filament which it wets. The viscosity conditions in the pasty mass are sufficiently high, however, that the rate of leakage of the molten phase from the latter is controlled and relatively slow so that the amount of molten liquid moving out from the mass onto the surrounding filament surfaces results in only a very thin film of such liquid being present upon the filament surface, and the boiling action in such film as the liquid metal evaporates results only in completely vaporized metal and no unvaporized liquid metal is blown off or spitted.

As the temperature of the metal alloy mass is being continuously raised by heat flow from the surrounding filament, such heat movement from the filament into the mass is cooling the adjacent portions of the filament somewhat and the filament and mass are progressively rapidly approaching each other in temperature, or the temperature differential between the two is becoming less and less. As the mass becomes hotter, more and more of the alloy appears to become fluid and the wetting of the filament proceeds through stages such as indicated at 19c in Fig. 5, and 19d in Fig. 6. In this manner, the improved alloys herein provided slow down the rate of application of the molten metal to the hot filaments and spitting is avoided.

Aluminum forms with molybdenum intermetallic compounds which are of very high melting point, and in alloys of these materials the intermetallic compounds are present as fine crystals generally of a needle like nature. On heating such aluminum alloys containing small amounts of molybdenum, the excess aluminum over that needed to form such intermetallic compounds begins to become fluid as temperatures beyond the melting point of aluminum are reached. The intermetallic compound materials, however, do not melt until much higher temperatures are reached and the presence of these crystalline materials in

the molten aluminum phase results in the pasty or highly viscous nature of the molten alloys. As the temperatures are raised beyond the melting point of pure or commercially pure aluminum and the mass approaches the temperatures of the filament surfaces, the viscosity of the molten aluminum alloy decreases and finally the intermetallic compounds melt, and as the crystals disappear the pasty mass becomes more liquid and fluid.

As the fluid aluminum or mixture of melted aluminum and intermetallic compounds or molten alloy flows onto the surrounding filament surfaces, the aluminum is vaporized into the evacuated chamber and deposits upon the article 14 to give a coating of aluminum. The aluminum present in the intermetallic compounds is also apparently distilled away from such compounds and the molybdenum set free remains upon the filament. Thus, while an alloy of aluminum is provided for evaporation purposes the deposits formed are of pure aluminum and show all the high reflectivity of pure aluminum. By way of contrast, the aluminum alloys, due to the intermetallic compounds, are grey and dull and will not take a polish such as is true of pure aluminum. By the use of the special aluminum alloys herein provided, aluminum mirrors of equal high reflectivity to those secured when evaporating pure aluminum are obtained.

In the thermal evaporation art, it has been suggested that the cause of spitting has been small amounts of air or other gases trapped in the aluminum, during the casting of the original billets, becoming heated and freed explosively at the time of the melting of the aluminum pieces in the vacuum. This, however, does not seem to be the case, but rather is caused by the above described temperature differential effects of the aluminum pieces and electric filament.

In general, it has been found that suitable aluminum alloys accomplishing the objects of this invention in producing coatings of aluminum by thermal evaporation are those containing molybdenum in an amount of less than 10% and the balance substantially aluminum. Further, it has been discovered that the amount of molybdenum in the aluminum alloy may be in the amount of 0.5% to 10.0%, with good results being obtained with an alloy of aluminum containing 2.0% molybdenum. Forming of the alloys either into a wire or a pellet is more readily carried out with the smaller amounts of the alloying molybdenum as the presence of the intermetallic compounds in the aluminum alloys cause these alloys to be much harder than pure aluminum.

Other specific aluminum alloy compositions which may be used in following the invention are by way of example:

1. Molybdenum 0.85%, balance aluminum.
2. Molybdenum 2.3%, balance aluminum.

In stating that the balance of the alloy, besides the molybdenum content, is substantially aluminum, it is the intent herein to signify that such balance is pure aluminum or commercially pure aluminum, the alloy being such as would result from the addition of the molybdenum to such forms of aluminum. The other elements such as iron, silicon, copper, etc. present in small trace amounts in commercially pure aluminum and thereby introduced in the alloys are of obviously no importance in the special alloys herein provided or in their action, since commercially pure aluminum and pure aluminum are equally bad in causing spitting.

I claim:

1. The method of providing a smooth, uniform coating of aluminum on a surface, which consists in providing a filament selected from the group consisting of tungsten, molybdenum, tantalum and columbium, placing on said filament an aluminum alloy containing between 0.5% and 10.0% molybdenum and the balance substantially aluminum, said molybdenum coating with said filament and said aluminum to retard the speed at which the aluminum spreads out on the filament when heated preliminarily

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to being evaporated, and heating said aluminum alloy to evaporate the aluminum component thereof from said filament onto a surface to be coated.

2. A method according to claim 1, wherein the aluminum is alloyed with about 2.0% of molybdenum.

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