Sakaue

[45] Dec. 23, 1975

[54]	VACUUM EVAPORATION PLATING APPARATUS			
[75]	Inventor:	Fukumatsu Sakaue, Minami-ashigara, Japan		
[73]	Assignee:	Fugi Xerox Co., Ltd., Tokyo, Japan		
[22]	Filed:	Apr. 11, 1974		
[21]	Appl. No.: 459,955			
[30]	Foreign Application Priority Data  June 4, 1973 Japan 48-61799			
[51]	U.S. Cl Int. Cl. <sup>2</sup>			
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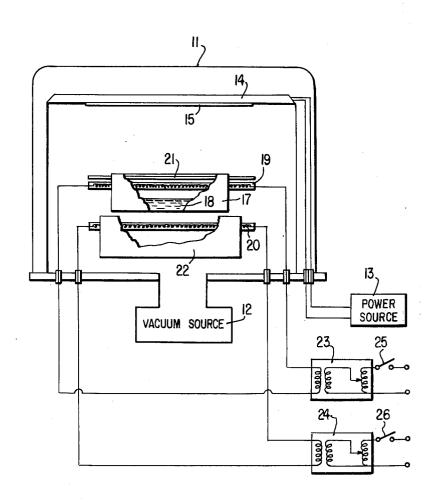
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Primary Examiner—Mervin Stein
Assistant Examiner—Steven Hawkins
Attorney, Agent, or Firm—Gerald J. Ferguson, Jr.;
Joseph J. Baker

# [57] ABSTRACT

A vacuum evaporation plating method and apparatus for suppressing fractional distillation of respective components of a substance to be evaporated comprising a heat source for heating with radiation the evaporating surface of the alloy, and an evaporator having a heat source for heating from either the interior or the bottom of the alloy.

### 8 Claims, 7 Drawing Figures



Te CONTENT

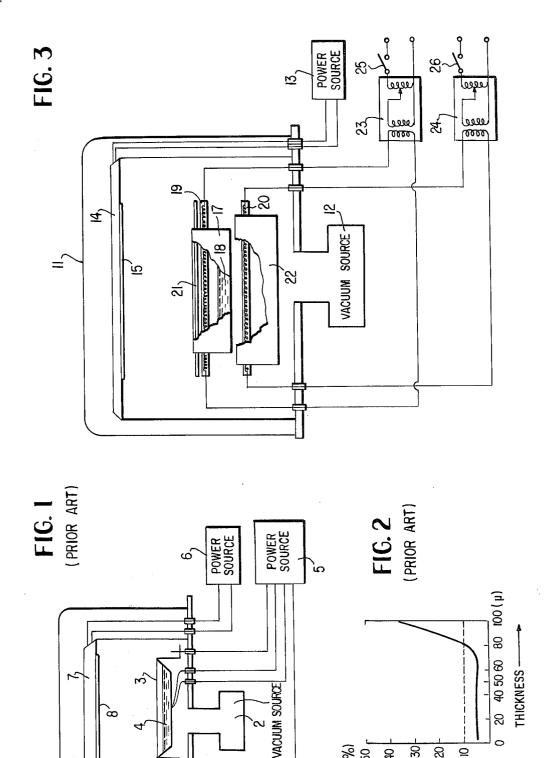


FIG. 4

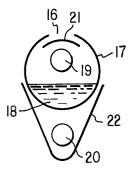


FIG. 5

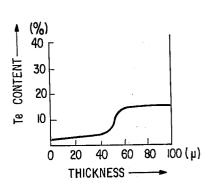
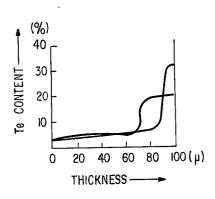
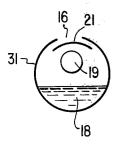


FIG. 6



**FIG. 7**,



# VACUUM EVAPORATION PLATING APPARATUS BACKGROUND OF THE INVENTION

# 1. Field of the Invention

The present invention relates to a vacuum evaporation plating method and apparatus capable of controlling and/or suppressing fractional distillation of respective components of a substance to be evaporated and

controlling the distribution of the deposited composi- 10 tion in the direction of the deposited film thickness, the substance being subject to fractional distillation when evaporated such as an alloy.

The vacuum evaporation process of this invention is represented by those processes where a certain sub- 15 stance is evaporated in a vacuum and the vapor thus generated is condensed on a base plate, this basic process being extensively used in many fields.

## 2. Discussion of the Prior Art

There is known apparatus for carrying out the afore- 20 orator which may be used in the apparatus of FIG. 3. mentioned vacuum evaporation technique. This apparatus typically includes an evaporator capable of being electrically heated and loaded with substance to be evaporated. The evaporator is heated, and the heat thus generated is transmitted to the substance, to thus 25 effectuate the desired evaporation. However, the conventional evaporation apparatus may be unsatisfactory for depositing semiconductor films or other layers which are adversely affected by the composition of the deposited film, since, when evaporating a substance 30 which is subject to fractional distillation when evaporated, like an alloy, the distribution of the composition of the deposited film thus obtained may lack desired

For instance, if a conventional evaporation plating 35 apparatus as shown in FIG. 1 is used, certain difficulties arise, which will now be discussed. The apparatus of FIG. 1 includes a tank 1, the interior of which is made vacuous to approximately 10-4 Torr by vacuum apparatus 2. An evaporator 3 is loaded with a sensitizer 4, which may be 10% Te - 90% Se (Te is tellurium, Se is selenium, and % is percent by weight as used here and hereafter). The evaporator 3 is electrically heated by a power source 5, the temperature of which is controlled to thus evaporate sensitizer 4. The vapor thus generated is deposited on a base 8 comprising an aluminum plate positioned on a base heating device 7, which is electrically heated from a power source 6. The sensitizer 4 is thus evaporated to obtain a sensitive plate for electrophotography, the deposited film having a distribution of Te in the direction of thickness as shown in

However, a satisfactory photosensitive plate for electrophotography should have a density of Te on the surface of 25% or less; therefore, a photosensitive plate 55 having Te distributed as shown in FIG. 2 has quite limited usefulness. In particular, the surface of the plate is subject to abrasion with attendant roughening thereof. This in turn, varies the characteristics of the photosensitive plate at the surface thereof and thus 60 lessens the usefulness of the plate.

#### SUMMARY OF THE INVENTION

A primary purpose of this invention is to provide an evaporation plating method and apparatus for option- 65 ally controlling, to a certain extent, the fractional distillation of a substance subject to fractional distillation when evaporated.

It is a further object of this invention to provide a method and apparatus of the above type for rendering uniform, to a certain extent, the distribution of the composition of a deposited film in the direction of the thickness thereof.

Other objects and advantages of the invention will become apparent from a reading of the specification and claims taken with the drawing.

#### BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic view of the general construction of a conventional vacuum evaporation plating appara-

FIG. 3 is a schematic view of the general construction of an illustrative vacuum evaporation plating apparatus in accordance with the present invention.

FIG. 4 is a side elevation of an illustrative evaporator which may be used in the apparatus of FIG. 3.

FIG. 7 is a side elevation of another illustrative evap-

FIGS. 2, 5 and 6 are graphs illustrating the distribution of Te in the direction of thickness of deposited film; FIG. 2 illustrating the film deposited by the conventional vacuum evaporation plating apparatus of FIG. 1, and FIGS. 5 and 6 respectively illustrating the films deposited by the embodiment of FIG. 4.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS OF THE INVENTION

Referring to FIG. 3, tank 11 has the interior thereof rendered vacuous to approximately 10-4 Torr, for example, by a vacuum apparatus 12. The tank 11 has a baseboard heating device 14 which is heated from an electric power source 13 and kept at a certain level of temperature. As can be seen device 14 is positioned in the upper section of tank 1, and is provided with a base plate 15.

Tank 11 has an evaporator 17 typically made of stainless steel plate 0.8mm thick. The evaporator has a 40 tubular shape as shown in the side elevation of FIG. 4 and is approximately positioned at the center of tank 11. The evaporator contains an evaporant or substance 18 to be deposited. An upper heater 19 heats the substance 18 by radiation and is positioned in the upper section of evaporator 17. A lower heater 20 heats the bottom section of the evaporator and is positioned in the lower section thereof. A shielding plate 21 typically made of a tantalum ribbon keeps substance 18 from spattering out of evaporator 17 and keeps heat radia-50 tion from upper heater 19 from adversely affecting the base plate 15. Plate 21 is positioned between the opening 16 of evaporator 17 and upper heater 19. A reflecting plate 22 with an open upper section is positioned around lower heater 20 to reflect heat generated by lower heater 20 over the bottom section of the evaporator. The ratings of upper heater 19 and lower heater 20 are typically 200V AC and 1KW, these heaters being respectively controlled by power source 23 and power source 24. Power sources 23 and 24 are placed in operation by switches 25 and 26 respectively.

A description of the results of two experiments conducted with deposition apparatus of the present invention as described above is given below.

#### EXPERIMENT 1

30 g of 10% Te - 90% Se was employed as substance 18 and the interior of tank 11 was made vacuous to 10<sup>-4</sup> Torr. The temperature of base plate 15 was set at

60°C and the space between base plate 15 and opening 16 of evaporator 17 was set to approximately 15cm. The lower heater 20 then had 150V applied thereto for two minutes. Switch 26 of the lower heater was turned off and switch 25 of upper heater 19 was turned on. 5 The upper heater 19 had 150V applied thereto for two minutes whereby all of substance 18 was evaporated and a deposited film of approximately  $100\mu$  was formed on base plate 15. The distribution of Te in the direction of thickness of the deposited film is illustrated 10 in FIG. 5. The distribution was determined by quantitative analysis with an XMA (X-ray micro analyser).

By comparing the distribution of Te illustrated in FIG. 5 with the distribution of Te illustrated in FIG. 2, it can be seen that with the present invention, fractional distillation can be controlled to a certain degree and the percentage of Te near the surface of the deposited film in the direction of thickness is such as to render the thereof when subjected to roughening abrasions.

#### **EXPERIMENT 2**

Lower heater 20 and upper heater 19 were energized where only the energization time of Experiment 1 was modified.

- a. The lower heater 20 was first energized for three minutes and then turned off. Upper heater 19 was then energized for one and a half minutes and then turned off.
- b. The lower heater 20 was first energized for four minutes and then turned off. Upper heater 19 was then energized for one and a half minutes and then turned off.

Quantitative analyses by an XMA revealed the distribution of Te in case (a) above to correspond to curve (a) of FIG. 6 and the distribution of Te in case (b) above to correspond to curve (b) of FIG. 6.

From the results thus obtained, it is evident that a certain degree of control can be obtained with the 40 present invention over the distribution of the constituents in the direction of thickness of the deposited film when the evaporant is subject to fractional distillation. This is effected by control of upper and lower heaters 19 and 20.

In the foregoing embodiment of the invention, the evaporator 17 was heated from the bottom section by lower heater 20; however, an evaporator 31 (see FIG. 7) may comprise a resistant substance so that evaporator 31 itself has an electric current passed there- 50 through, whereby substance 18 is heated from within. Thus, in this embodiment, lower heater 20 is not necessary.

The vacuum evaporation apparatus of the present invention includes an evaporator having a heat source 55 for heating by radiation the evaporation surface of an evaporant together with a heat source for heating the evaporant either from within or from the bottom portion thereof so that fractional distillation of the evaporant can be controlled to thereby control the distribu- 60 tion of the film constituents in the direction of thickness of the deposited film. Further, the composition of the deposited film in the direction of the thickness thereof can be kept virtually constant in a certain range of thickness by the proper selection and use of both of 65 the aforesaid heat sources. It should also be appreciated that both the heat sources may be operated at the same time or heat source 19 prior to heat source 20 in

order to effect the advantageous results of this inven-

Thus, even though the deposited film is subjected to abrasion, the surface composition thereof may be kept virtually free of undesired variations so that the deposited film has a long life with the initial characteristics thereof maintained intact over its useful life. These advantageous results are especially apparent in the field of those semiconductors where the characteristics of a deposited film is considerably influenced by the distribution of the constituents of the deposited film, for instance, photosensitive materials used in electrophotography.

Furthermore, the vacuum evaporation plating apparatus of the present invention can be manufactured at low cost, due to the simple construction thereof, and evaporation can be controlled in accordance with an established program. Further, due to the low level of film virtually free from variations in the characteristics 20 calorific capacity of the apparatus as a whole, the operational performance is also considerably improved.

What is claimed is:

1. A vacuum evaporation plating apparatus for controlling fractional distillation of respective components 25 of a photosensitive material, said apparatus comprising a single container for said photosensitive material;

a first heating means for heating with radiation the evaporating surface of the photosensitive material, said first heating means being disposed above said material such that said electromagnetic radiations impinge directly on said evaporating surface;

a second heating means for heating the photosensitive material from either the interior or bottom thereof;

means for receiving the vapors of the heated photosensitive material for forming a photosensitive film;

- said first and second heating means controlling the deposition of said components to control the relative percentages of said components in the photosensitive film deposit through the thickness
- 2. Apparatus as in claim 1 including means for activating said second heating means and then said first heating means.
  - 3. Apparatus as in claim 1 where said photosensitive material is 10% by weight tellurium and 90% by weight selenium, the thickness of said deposit on the receiving means being at least  $100\mu$  and the percentage of tellurium near the surface of said deposit being less than 25%.
  - 4. Apparatus for vacuum-evaporating a multi-element photosensitive material having at least first and second elements where said first element evaporates less readily than said second element, said apparatus

a single container for said multi-element material;

first heating means including a heating element for radiating continuous wave electromagnetic radiations onto the evaporating surface of said material to evaporate the material, said first heating means being disposed above said material such that said electromagnetic radiations impinge directly on said evaporating surface;

second heating means for heating said multi-element material from beneath the upper surface thereof; and

means for receiving the vapors of said material to form photosensitive film deposit of said material on said receiving means.

- 5. Apparatus as in claim 4 including reflecting means for preventing the radiation of said heating element 5 from directly reaching the receiving means upon which the film deposit forms, said first and second heating means controlling the deposition of said first and second elements to control the relative percentages of said thickness thereof.
- 6. Apparatus as in claim 5 where said containing means for the multi-element material is tubular with a longitudinally extending opening in the top thereof,

said heating element extending longitudinally in said containing means over said multi-element material and said reflecting means extending longitudinally in said containing means between said opening and said heating element.

- 7. Apparatus as in claim 4 where said first element is tellurium and said second element is selenium.
- 8. Apparatus as in claim 7 where said tellurium is elements in the photosensitive film deposit through the 10 10% by weight and said selenium is 90% by weight, the thickness of said deposit being approximately at least  $100\mu$  and the percentage of tellurium near the surface of said deposit being less than 25%.

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