Wire Fed Evaporation of Copper from Refractory Metal Boats

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ABSTRACT

In the 1980's a vacuum metallic foil making process was developed at Sidrabe, Inc. Machines were built to evaporate 10 microns of copper onto a naked coating drum and then immediately follow that with hundreds of microns of magnesium-mercury alloy to make sea water activated battery anode plates. This dual layer foil was then peeled from the drum and rewound in a continuous process. These machines used transverse mounted refractory metal boats to evaporate the copper. Copper wire was fed into both ends of the boat. Vapor escape ports on the boats were sized to provide uniform cross web deposition. The boats could evaporate Cu vapor in any direction. In 1994 the boat design was changed to allow retrofitting an array of smaller boats into conventional aluminizers, with some necessary power supply changes. This allows coating to any web width and at varying web speeds. This type of boat has been used for 12 years to make flexible printed circuit material. The boats may be used for Cu, Au, and Sn with little change needed. Some material changes are needed to evaporate Ag. Examples of their performance will be given.

INTRODUCTION

Modern vacuum equipment for coating deposition on polymeric films requires the use of high-performance sources of vapor for evaporation of such metals as aluminum and copper. For aluminum deposition ceramic boats, permitting high speed of evaporation, are used. The resistive evaporators are widely used for copper evaporation. Its design is quite simple, as well as the manufacture; its application does not need expensive power supplies, or control equipment either.

Boats or crucibles, made from molybdenum, tantalum or tungsten, are usually used for batch copper evaporation. Due to the copper melt specific for short-circuiting effect, the boats can be only used as disposable parts, allowing small amount of copper load for evaporation. Using a crucible permits multiplying the load to a few tens or more grams, but with increasing evaporation load the evaporator has more thermal inertia, as a considerable period of time is required for a warming-up and melting of a complete load of copper. In addition, due to high outgassing and boiling of copper at its melting, only copper, which has been preliminary melted in vacuum can be used for loading in a crucible. For keeping a crucible workable, any copper remaining in the crucible is to be entirely evaporated until heating of the crucible is switched off. If this is not done, at cooling down of a crucible with remaining copper the crucible can be destroyed due to a big difference between linear expansion coefficients of the two materials. The productivity of such an evaporator is low and lies in limits from fraction of grams to some grams of copper per minute. Arising from the aforesaid need for improvements, the development of productive and time proof copper evaporators was successfully implemented.

EXPERIMENTAL

We have much previous experience in the design and production of different types of vapor sources. More then 30 years ago we took part in development of material for the high-current batteries. The material-development process included also producing of free copper condensate 10 microns thick on the coating drum polished surface, the immediate sequential deposition of a magnesium and mercury layer with a thickness of 250 microns, peeling of the combined material off the drum, and its take off with rewinding on a shaft in the condition of free foil. Within this work we devised our first, durable, quite effective resistive evaporator for copper. The device description was given in details in [1]. The resistive evaporator is widely known in general. Its design is quite simple, as well as the manufacture; its application does not need expensive power supplies, or control equipment either. The copper evaporator is composed of 2 tubular evaporation elements, wire feeding mechanism, shields and power feed terminals. The tubular evaporation elements are manufactured of a refractory metal. For the period after the first evaporator was manufactured and up to the present time we devised several alternative thermal evaporators, intended for different application and evaporation of different metals (Figure 1). From all of them the most applicable became the "boat" version (position 1).

The "boat" is mostly used for the evaporation of copper, silver and gold. For these metals evaporation is performed by feeding with wire of a diameter 1.2-1.6 mm. After some inconsiderable design modification the boat was applied for the evaporation of tin and indium. Since tin's melting point is low, while evaporation temperature reaches and exceeds 1400 °C, tin wire starts melting long before it reaches the evaporation zone. This disadvantage was solved by putting



Figure 1: Resistive evaporators with long-life time.

the tin wire inside a guide tube, made additionally (Figure 1, position 2). Indium was fed in the liquid state to the evaporator. For the provision that all load of liquid indium reaches the evaporation zone, a receiving funnel was made and installed (Figure 1, position 3).

Our developed boat is a linear tubular device with a Mo thin-wall tube as holding part and vapor director. The tube is fixed on the power feed terminals. Along the tube axis, symmetrically to internal wall there is a core of porous material. The tube has a number of apertures for the vapor directional outflow. The tube can rotate on the axis and this allows having vapor outlet not only in vertical direction but in any wanted direction. There are end contacts for fixation the boat on evaporator. The end contacts are made with a small taper angle for fixing through graphite foil inserts in the water-cooled copper terminals. On the end contacts there are two drilled holes which make a certain space (chamber) for fed metal receiving and melting (Figure 2) and some channels for feeding the metal to the porous core. To provide required difference of temperature thermal bridges are made between end contacts and melting chamber. During the coating cycle the wire is fed and melted in the melting chamber at the ends of the boat. Melted copper further enters into the porous core, moistens and soaks the core over its entire length, heats up and evaporates from the core surface in all directions. Copper vapor, is multiply reflected from the Molybdenum tube internal wall, goes out of the tube via the apertures, and reaches the substrate. During the evaporation, the temperature of the porous core is 1600 - 1650 °C.



Figure 2: End contact of the boat with melting chamber and thermal bridge.

Note in Figure 2 that the outer shield is positioned for sideways direction of the vapor. Vapor distribution from a single boat in transversal direction is wide enough, that it allows arranging the boats in a line, in combination with other, and using them in vacuum machines for web coating in a manner similar to Aluminizers. Figure 3 shows coating uniformity relative coating thickness for deposition on plane substrate distanced from the boat by 200 mm.



Figure 3: Uniformity of coating relative thickness on a plane substrate.

Provision of perfect tube-wetting conditions and comprehensive surface cleanliness of the porous core guarantee evaporation without macro-particles surge. For the stability of evaporation conditions in time the boat should operate in the conditions of stabilized voltage on end contacts. In this case, for example, small increase of copper amount in a porous core will be compensated by the proportional increase of current and consequently power and evaporation speed. And vise versa, if copper content on the evaporator drops down, current goes down considerably, decreasing power and evaporation speed. Thus, initial content of copper on boat becomes restored. Evaporation process becomes somewhat self-stabilizing; the boat operates in stable working conditions in conformity with a preset voltage range.

Evaporation speed or productivity of a boat is between 4 and 10 g/min for all above mentioned metals. Depending on required evaporation speed voltage on copper terminals should be in a range of 2.6 to 3.0 V. During a cycle voltage setting should be sustained constant with accuracy $\pm 0.5\%$. For longer operability of the boat its heating up should be smooth

within time no less than 5 minutes. Boat life-time depends on type and purity of evaporated metal and number of boat heating and cooling cycles. It is about 40 hrs for copper, and 60-80 hrs for In. The boats are mostly applied for copper and other metal coatings on polymer film. Depending on substrate width, some our roll-to-roll coaters allow installation up to 18 boat arrays. When closed loop controls were not provided, uniformity of coating thickness was provided with uniformity $\pm 10\%$ on film width 1600 mm.

Evaporator basic parameters:

Power, kW, no more	6
Voltage on terminal connectors, V, range 2.6	3.0
Current, A, range 1300	1800
Current frequency, Hz	. 50(60)
Evaporation speed, g/min, (on Cu, Ag), range	4 – 10
Boat life-time, hrs (for Cu).	40
Dimensions:	
- Length, mm	202
- Tube diameter, mm	20

CONCLUSIONS

A new design of high-performance thermal evaporator of copper, silvers, gold, indium and tin was developed and tested. Time-life of the developed evaporator arrives up to 40 hours and from each evaporator it is possible to evaporate up to 25 kg of foregoing metals. The developed constructions of vaporizers exhibited reliable and stable performance on industrial roll-to-roll vacuum machines.

REFERENCE

 Zhunda A.N., "Long-action resistive copper evaporator. in physical vapor deposition." Riga: *Zinātne*, 1986, p. 34-37, (In Russian).