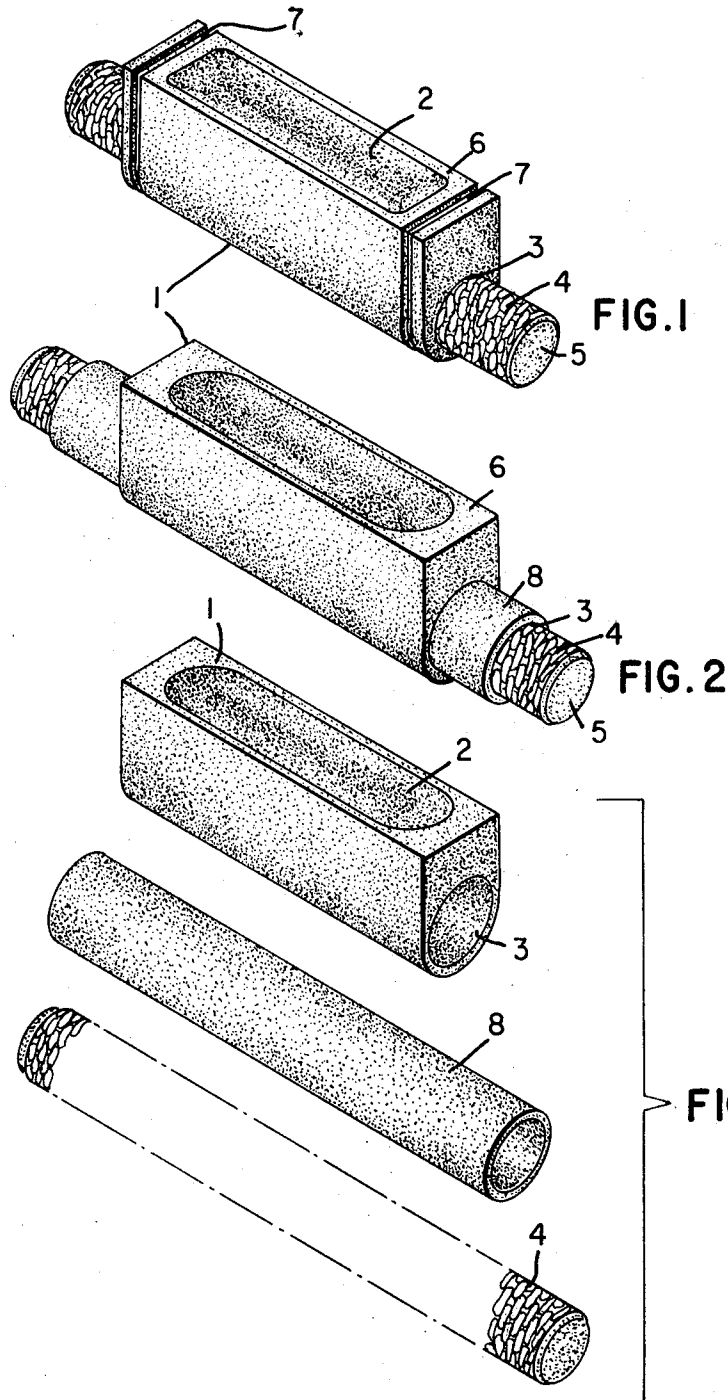


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METAL-EVAPORATING SOURCE
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METAL-EVAPORATING SOURCE

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ABSTRACT OF THE DISCLOSURE

An improved evaporation source for vacuum deposition of metals has a resistance heater element of intertwined helical coils of refractory metal wire. An elongated refractory vessel, having a cavity to contain the metal being evaporated, is supported and heated by the heater element. There is a barrier between the cavity and the heater to prevent liquid metal overflow from contacting the heater.

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to evaporation vessels for use in metal coating processes, where the metal is heated in vacuo to its evaporation temperature and then deposited on the article to be coated, and particularly when the evaporator vessel contains the charge of metal being evaporated, but does not constitute the heating element.

Description of the prior art

Electrically conductive, refractory heating elements have been used for evaporating metals, such as aluminum, silver, cadmium and the like, but they have commonly suffered from excessive corrosion by the liquid metals being evaporated or from thermal cracking due to the high temperature of operation. In addition, unless the ends of the heating elements were clamped in completely flexible binding posts, the heating element could fracture during operation because of thermal expansion or because of strains imparted to the rigid element when its ends were tightly clamped. Elements of this type have been made of graphite, titanium boride, titanium carbide and the like.

Evaporating heaters have also been made from refractory wire alone, such as tungsten, molybdenum and tantalum, but they are generally short-lived due to corrosion from the liquid metal, such as aluminum, being evaporated.

In an application filed on even date herewith, entitled "Metal-Evaporating Source," by Matheson et al., assigned to the instant assignee, an evaporation source comprising a refractory vessel and a mesh heater element is disclosed. The liquid metal, during operation, however, could overflow the cavity and contact the heater element, thereby corroding it and shortening its useful life.

SUMMARY OF THE INVENTION

We have invented a long-lived evaporating source which is substantially resistant to corrosion by the liquid metal being evaporated and is not subject to cracking because of strains imparted to the element when its ends are clamped in electric-supply binding posts. We fabricate an elongated evaporating vessel, having a cavity to contain the metal to be evaporated, from a refractory material which is resistant to corrosion by the metal at its evaporation temperature. The vessel is supported and heated by a novel resistance heater, comprising a mesh of intertwined refractory metal coils. The ends of the

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heater protrude beyond the ends of the vessel and are connected to, and supported by, the binding posts of a vacuum deposition apparatus, and the heater is resistively heated by an electric current flowing through it.

To prevent any liquid metal overflow from contacting the heater, a barrier is located between the cavity and the heater. The barrier can be a protective sleeve around the heater or a channel on the surface of the vessel to conduct the overflow away from the exposed ends of the heater.

The flexibility of the mesh construction permits the heater to absorb the strains resulting from thermal expansion or from minor misalignment of the binding posts to which its ends are clamped, without damage to the heater. In addition, these strains are not transferred to the evaporation vessel, since there is no rigid connection between the vessel and the heater. Thus, the life of the evaporation source is extended by substantially reducing failures due to vessel-metal corrosion, clamping strains or heater corrosion from metal overflow.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an elevational view of one embodiment of a metal-evaporating source in accordance with this invention.

FIG. 2 illustrates another embodiment of the invention, and FIG. 3 shows the separated components of FIG. 2.

DESCRIPTION OF THE PREFERRED EMBODIMENT

In FIG. 1, elongated refractory vessel 1 is shaped from a refractory material which is substantially resistant to corrosion by the liquid metal to be evaporated and is also substantially inert to heater 4 at operating temperatures. Cavity 2 is located on upper surface 6 of the vessel to serve as a container for the metal to be evaporated. It is desirable to limit the charge of metal in cavity 2 to an amount which is usable for semi-continuous operation without an excessive heater temperature; therefore the depth of cavity 2 is preferably less than about $\frac{1}{3}$ the depth of vessel 1. Ideally, cavity 2 is centrally located on surface 6 to minimize any thermal strains that may develop from uneven wall thicknesses around cavity 2. Heater compartment 3 is located in the lower portion of vessel 1, longitudinal therewith, and of suitable dimension to permit mesh heater 4 to be inserted therein. However, for optimum heat transfer from heater 4 to vessel 1, the cross sectional area of compartment 3 is at a minimum consistent with that which permits ready insertion of heater 4. We also prefer to locate compartment 3 within vessel 1 in such a position that the thickness of the lateral walls is substantially equal, in order to minimize thermal strains that may develop within vessel 1 from non-uniform wall thicknesses.

Between the respective ends of cavity 2 and vessel 1 are located channels 7, generally extending transverse to the long dimension of vessel 1 but at a distance from cavity 2 and the ends of vessel 1. Channels 7 extend at least across upper surface 6 but can also extend down the sides of vessel 1. The width and depth of channels 7 are usually small compared to the size of vessel 1 and their function is to conduct any liquid metal that overflows cavity 2 away from heater 4. However the cross-sectional area of channels 7 must not be so small that the surface tension of the liquid metal prevents it from flowing, under the force of gravity, in channels 7 and away from heater 4. When there is flooding of cavity 2 and the liquid metal overflows, channels 7 conduct it exteriorly down to the bottom of vessel 1, where it drops off harmlessly, generally onto a shield at the bottom of the deposition chamber.

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Mesh heater 4 is made of individual helical coils intertangled together. First, straight pieces of refractory wire are individually coiled about a mandrel with each turn spaced apart from adjacent turns to form individual helical coils. After removal from the mandrel, two coils are threaded into each other, a third is threaded into the second, a fourth is threaded into the third and the procedure is continued until a mat of intertangled coils is obtained. The mat is then longitudinally positioned around an elongated arbor so that the first and last coils are adjacent to each other, and another coil is threaded through both the first and last coils to join them. After removal from the arbor, ends 5 are welded to securely fasten the coil ends and form an integral flexible heating element in which the individual coils are free to expand and contract within each other. Preferably, a rod of the same metal is inserted and welded in the ends of the heater to lower the temperature of the ends below that of the body of the heater during operation.

Heater 4 is then inserted into compartment 3 of vessel 1 so that substantially equal lengths of heater 4 protrude from each end of the vessel 1. Although heater 4 does not fit tightly into compartment 3, there is enough surface contact to frictionally prevent vessel 1 from sliding or moving on heater 4 merely from its own weight.

In operation, the ends of heater 4, with vessel 1 supported thereon, are clamped to binding posts or other convenient means for supplying electric power to heater 4 within a vacuum chamber. Provision within the chamber is made for locating the article to be coated in a line of sight with cavity 2 so that the metallic vapor emanating therefrom will condense and deposit on the article. A charge of the metal to be evaporated is placed in cavity 2 and the pressure within the vacuum chamber is then reduced to less than about 0.001 torr. Sufficient electric power is then supplied to heater 4 to heat the metal charge at the chamber pressure to a temperature where the metal evaporates at a useful rate and deposits on the article within the chamber. In a semi-continuous coating process as, for example, where a continuous roll of plastic film is to be coated with aluminum, provision within the vacuum chamber is made for continuously unreeling the film from a roll, passing the film over cavity 2 and taking up the coated film on another reel, while the aluminum is continually being vaporized. The source of aluminum for cavity 2 is a spool of aluminum wire within the chamber with the free end of the wire directed into the cavity. The spool is de-reeled at the proper rate to maintain a substantially constant charge of molten aluminum in the cavity and to approximate the rate of evaporation of the aluminum. Sufficient electric current is maintained through heater 4 to obtain the desired evaporation rate. However, during the start-up period when the rate of supply of aluminum is being adjusted to the rate of evaporation, flooding of cavity 2 can occur if an excess of aluminum is supplied. When this happens, channels 7 prevent the aluminum overflow from contacting heater 4 by conducting it away to the bottom of vessel 1.

In another embodiment of the invention, shown in FIGS. 2 and 3, the barrier between cavity 2 and heater 4 is an elongated refractory sleeve 8. Sleeve 8 is disposed around heater 4 and prevents direct contact between heater 4 and the vessel 1. The ends of sleeve 8 extend beyond the ends of vessel 1 but not as far as the ends of heater 4, in order to permit the latter to be clamped in the binding posts. If liquid metal does overflow cavity 2 and upper surface 6, it can flow down the ends of vessel 1 onto the protruding sections of sleeve 8. However, in order to prevent a buildup of liquid metal on sleeve 8 which could then flow out over its ends and onto the exposed ends of heater 4, the upper surface of sleeve 8 is beveled or curved, preferably convexly. Thus the metal will flow down and off sleeve 8, and not out to the ends.

During operation, sleeve 8 will generally be at a higher

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temperature than vessel 1 since it is in closer proximity to heater 4 and also is not being directly cooled by evaporating metal. Therefore, sleeve 8 can sometimes be hot enough to evaporate liquid metal overflow, even when vessel 1 is not hot enough to prevent flooding.

Another possible cause of liquid metal contacting heater 4 is surface tension. When vessel 1 is readily wet by the liquid metal, the metal can spread out and creep on the surface of vessel 1. For example, when vessel 1 is made of graphite or boron nitride and the liquid metal is aluminum at a temperature above about 1400° C., the liquid aluminum wets the surface of vessel 1 and gradually spreads out away from cavity 2. In this case, the barrier of our invention impedes the flow of the liquid metal toward the exposed heater by substantially increasing the length of the path between the heater ends and cavity 2.

In FIG. 1, in a specific example of an evaporating source in accordance with this invention, designed for the semi-continuous evaporation of aluminum, vessel 1 was machined from a solid block of boron nitride, which is substantially resistant to tungsten and molten aluminum at the temperatures involved. The vessel had a length of 2½ inches, a width of ¼ inch and a depth of 1½ inches. Cavity 2 was centrally located in upper surface 6 and was approximately 1¾ inches long, ⅞ inch wide and ⅛ inch deep. Compartment 3 was formed by drilling a ⅞ inch diameter hole longitudinally through vessel 1 in the lower portion thereof, with a wall thickness at the bottom of the vessel of ⅞ inch. Channels 7 were machined ⅛ inch in from each end of vessel 1 on the upper and lower surfaces and on the two vertical surfaces parallel to the length of the cavity, which formed an effective barrier between cavity 2 and heater 4. The channels were ⅜ inch deep and ⅛ inch wide.

Heater 4 was made from eighteen tungsten coils intertangled together, as mentioned above. Each individual coil was made by winding 0.040 inch diameter tungsten wire on an 0.060 inch diameter mandrel at a pitch of 6½ turns per inch. After removal from the mandrel, each coil was trimmed to a length of 4½ inches. Seventeen coils were then intertangled to form a mat, which was then placed around a cylindrical arbor, ⅛ inch diameter, and the eighteenth coil was then intertangled through the first and seventeenth coils to form a cylindrical mesh element 4½ inches long with an outside diameter of about 1¾ inch. After removal from the arbor, a tungsten rod, about 1 inch long by ⅛ inch diameter, was inserted into each end of heater 4 and the ends of heater 4 were heliarc welded to secure the rods and coils.

Heater 4 was then inserted into compartment 3 of vessel 1 so that approximately 1¾ inches of the heater protruded from each end of vessel 1. The evaporating element was then mounted in a vacuum deposition chamber and approximately 1 inch of each end of heater 4 was clamped to water-cooled, copper, semi-rigid binding posts. Within the vacuum chamber were means for continuously supplying aluminum wire to cavity 2. The pressure within the chamber was reduced to less than 0.001 torr and an A.C. potential applied to the binding posts. At steady state conditions, there were 8 volts on the heater and about 500 amperes flowing through it. The temperature of the heater was about 1800° C., the temperature of the vessel was about 1400° C., and the aluminum was evaporated at the rate of 4 grams per minute.

In another embodiment of the invention, illustrated by FIGS. 2 and 3, the shape of vessel 1 was similar to that of FIG. 1 but without channels 7 between cavity 2 and the ends of vessel 1. In place thereof, the barrier was a sleeve 8 of boron nitride, which had an inside diameter of ⅞ inch and a wall thickness of ⅛ inch. Compartment 3 had a diameter slightly greater than ½ inch and the diameter of heater 4 was slightly under ⅞ inch. When heater 4 was inserted into sleeve 8 and both were inserted into compartment 3, about ½ inch of sleeve 8 protruded be-

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yond the ends of vessel 1 and about one inch of heater 4 protruded beyond the ends of sleeve 8. In another example of this embodiment (not shown) sleeve 8 consisted of two shorter sleeves of boron nitride protruding about the same distance from vessel 1, but not extending completely through compartment 3.

Although these particular vessels and sleeves were made of boron nitride, other aluminum and tungsten resistant materials such as aluminum nitride, titanium carbide, zirconium diboride and titanium boride, or any combination or mixture thereof, could also be used in the vapor deposition of aluminum, since they have adequate corrosion resistance for purposes of this invention.

Also, the vessels and sleeves can be prepared by hot pressing the refractory powder in a graphite die having the necessary configuration to produce the desired shape. Or they can be prepared by cold pressing the refractory powder, or casting a slurry thereof, in a suitably shaped mold, followed by high temperature sintering to yield the necessary physical stability and strength. In addition, our invention can be used for the vacuum deposition of other metals, such as gold, silver, chromium, nickel and the like, by proper selection of the refractory material to be used for corrosion resistance to the heater element and to the metal being evaporated. For example, the previously mentioned compositions are also suitable for the evaporation of gold, silver and copper.

Although an example of a cylindrical mesh heater has been described, other shapes having, for example, polygonal, elliptical or semi-flat cross sections, can be used. These configurations are fabricated by wrapping and fastening the mat of woven coils previously mentioned, on a correspondingly shaped pattern, and welding the ends of the heating element to maintain the desired configuration. It is also within the contemplation of this invention that the mesh heating element can be manufactured from other resistance-heating refractory metal wires having a melting point above about 2000° C., such as molybdenum, tantalum or niobium.

It is apparent that modifications and changes can be made within the spirit and scope of the instant invention, but it is our intention to be limited only by the appended claims.

We claim:

1. An evaporation source for the vapor deposition of metal comprising:
 - a resistance heating elongated element comprising intertwisted helical coils of refractory metal wire, the turns of said coils being held by the turns of adjacent coils, whereby the individual coils are free to expand and contract within each other;
 - an elongated refractory vessel supported on said element and adapted to receive heat from said element;
 - a cavity on the surface of said vessel, adapted to contain said metal, and
 - means to inhibit said metal from contacting said element.

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2. The evaporation source of claim 1 wherein said refractory metal wire is tungsten.

3. The evaporation source of claim 2 wherein said refractory vessel is made of boron nitride.

4. An evaporation source for the vapor deposition of metal comprising:

- a resistance heating elongated element comprising intertwisted helical coils of refractory metal wire the turns of said coils being held by the turns of adjacent coils, whereby the individual coils are free to expand and contract within each other;

- an elongated refractory vessel supported on said element and adapted to receive heat from said element; the ends of said element protruding beyond the ends of said vessel;

- a cavity disposed on the surface of said vessel to contain said metal, and
- means to inhibit said metal from contacting said element.

5. The evaporation source of claim 4 wherein said means comprising a barrier disposed between said cavity and said element.

6. The evaporation source of claim 4 wherein said means comprises an indentation in said vessel intermediate said cavity and the ends of said vessel, said indentation extending at least across the top surface of said vessel and disposed at a distance from said cavity.

7. The evaporation source of claim 4 wherein said means comprises an elongated refractory member disposed between said element and said vessel, the ends of said member intermediate the respective ends of said vessel and said element.

8. The evaporation source of claim 4 wherein said metal to be vapor deposited is aluminum.

9. The evaporation source of claim 7 wherein said heating element and said refractory member are tubular and coaxial.

10. The evaporation source of claim 9 wherein said refractory metal wire is tungsten.

11. The evaporation source of claim 10 wherein said refractory member and said elongated vessel are formed of boron nitride.

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