June 2, 1970 W. G. MATHESON ET AL 3,515,852

METAL-EVAPORATING SOURCE

Filed Aug. 10, 1967

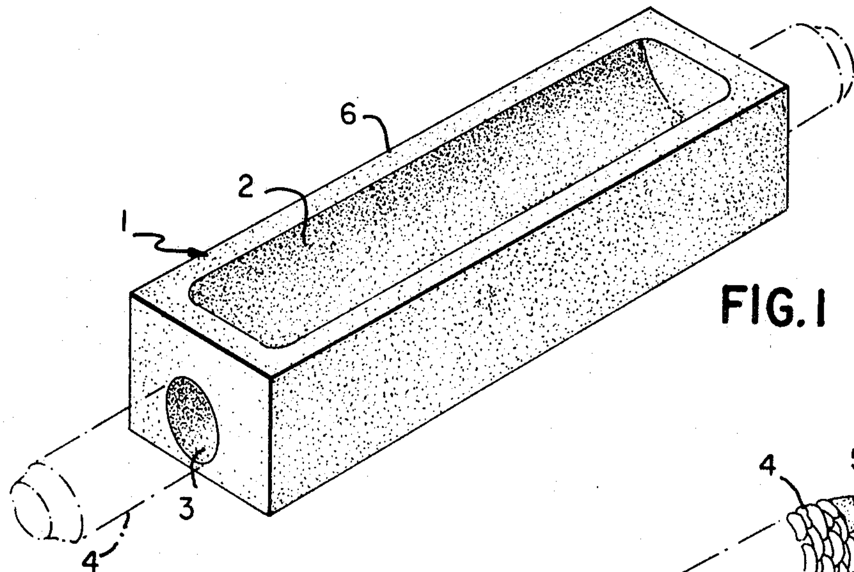

FIG. 1

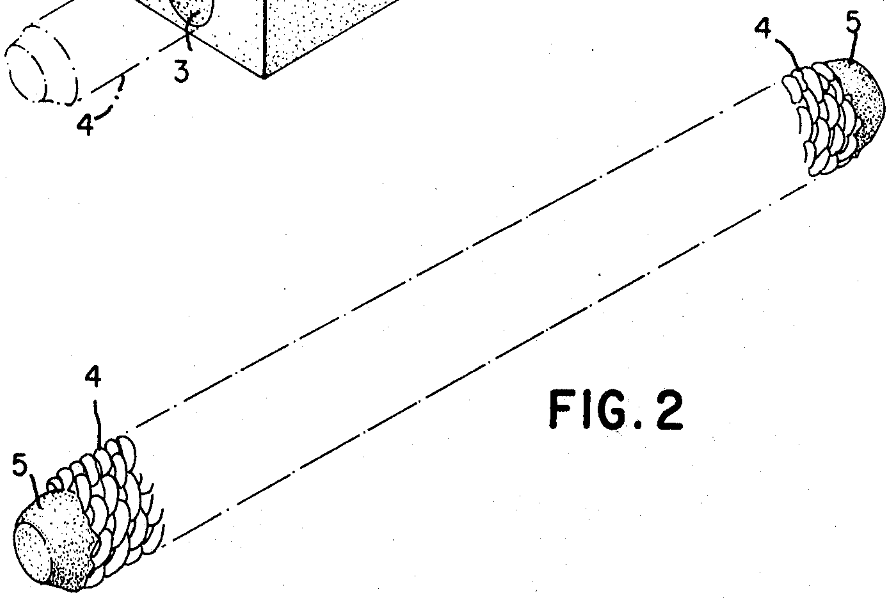

FIG. 2

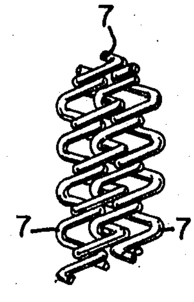

FIG. 3

WILFRID G. MATHESON
ROBERT STEINITZ
LESTER W. STROCK
INVENTORS
BY Laurence Burns
ATTORNEY

United States Patent Office

3,515,852

Patented June 2, 1970

3,515,852

METAL-EVAPORATING SOURCE

Wilfrid G. Matheson, Marblehead, Mass., Robert Steinitz, Harrison, N.Y., and Lester W. Strock, Salem, Mass., assignors to Sylvania Electric Products Inc., a corporation of Delaware

Filed Aug. 10, 1967, Ser. No. 659,747
Int. Cl. C23c *13/00*
U.S. Cl. 219—275 9 Claims

ABSTRACT OF THE DISCLOSURE

An improved evaporation source for vacuum deposition of metals has a resistance heater element of intertwisted 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.

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 where 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.

SUMMARY OF THE INVENTION

We have invented a long-lived evaporating element 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 intertwisted refractory metal coils. The ends of the 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. The close contact between the evaporation vessel and the body of the heater provides for efficient transfer of heat from the heater to the vessel.

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 or clamping strains.

Another advantage of the evaporation source of the instant invention is the uniformity in its electrical resistivity, which is dependent mainly on the construction of the mesh heater. This can be important in semi-automatically controlled production coating equipment, where it is desirable to maintain substantially constant heater currents and deposition rates when the evaporation sources are replaced. This is not possible with the electrically conductive refractory elements previously mentioned since their resistivity varies considerably from element to element.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a perspective view of a metal-evaporating source in accordance with this invention showing the heating element in phantom lines.

FIG. 2 is an enlarged perspective view of the heating element showing in detail the end portions with the main body portion in phantom lines.

FIG. 3 is an enlarged detail of the mesh construction of the heating element.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

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 the cavity to an amount which is suitable for semi-continuous operation without an excessive heater temperature; therefore the depth of the cavity is preferably less than about ⅓ the depth of the vessel. Ideally, cavity **2** is centrally located on the surface to minimize any thermal strains that may develop from uneven wall thicknesses around the cavity. Heater compartment **3** is located in the lower portion of the vessel, 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 the compartment within the vessel in such a position that the thickness of the lateral walls is substantially equal, in order to minimize thermal strains that may develop within the vessel from non-uniform wall thicknesses.

Mesh heater **4** is made of individual helical coils **7** intertwisted together, as shown in FIG. 3. 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 **7**. 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 intertwisted 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, as in FIG. 2, and form an integral, flexible heating element in which the individual coils are free to expand and contract within each other.

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

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, to a length of 1⅝ inches, a width of 9⁄16 inch and a depth of 1 1⁄16 inches. Cavity **2** was centrally located in upper surface **6** and was approximately 1⅜ inches long, 7⁄16 inch wide and ⅛ inch deep. Compartment **3** was formed by drilling a 7⁄16 inch diameter hole longitudinally through vessel **1** in the lower portion thereof, with a wall thickness at the bottom of the vessel of 1⁄16 inch. The bottom surface of the vessel was rounded so that the wall thickness surrounding the entire bottom half of the compartment was approximatelp 1⁄16 inch.

Heater **4** was made from eighteen tungsten coils intertwisted together, as mentioned above. Each individual coil **7** 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, coil **7** was trimmed to a length of 4 inches. Seventeen coils were then intertwisted to form a mat, which was then placed around a cylindrical arbor, ⅛ inch diameter, and the eighteenth coil was then intertwisted through the first and seventeenth coils to form a cylindrical mesh element, 4 inches long with an outside diameter of about 13⁄32 inch. After removal from the arbor, the ends of heater **4** were heli-arc welded to secure the ends of individual coils **7**.

Heater **4** was then inserted into compartment **3** of vessel **1** so that approximately 1 5⁄16 inches of the heater protruded from each end of the vessel. The heater was then mounted in a vacuum deposition chamber and approximately 1 inch of each end 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.

Although this particular vessel was 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 vessel can be prepared by hot pressing the refractory powder in a graphite die having the necessary configuration to produce the desired shape of the vessel. Or the vessel 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, the 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 vessel 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.

A rod of the same metal as the heater may be inserted in the ends of the heater to lower the temperature of the ends below that of the body of the heater. Also, if two or more refractory vessels are spaced apart on the same heater element, a similar rod may be inserted in that position of the heating element between the vessels to reduce the heat dissipated therein.

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 vacuum deposition of metals, comprising:

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

an elongated refractory vessel supported by said element and adapted to receive heat from said element and a cavity disposed on the surface of said vessel.

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

3. The evaporation source of claim **2**, wherein the depth of said cavity is less than about ⅓ of the depth of said vessel.

4. The evaporation source of claim **3**, wherein said vessel has an elongated compartment in which said heater is disposed.

5. The evaporation source of claim **3**, wherein said heater is cylindrical.

6. The evaporation source of claim **5**, wherein said vessel has a longitudinal cylindrical compartment therethrough in which said heater is disposed.

7. An evaporation source for the vapor deposition of aluminum comprising:

a cylindrical resistance heating elongated element comprising intertwisted helical coils of tungsten 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 having a cylindrical compartment longitudinally therethrough, said heater disposed in said compartment and a cavity disposed on the surface of said vessel.

8. The evaporation source of claim **7**, wherein the ends of said heating element protrude beyond the ends of said vessel.

9. The evaporation source of claim **8**, wherein said vessel is formed of boron nitride.

References Cited

UNITED STATES PATENTS

2,757,788	8/1956	Weber	219—271 X
2,903,544	9/1959	Reichelt et al.	
3,216,710	11/1965	Lenihan et al.	219—271 X
3,274,374	9/1966	Matheson et al.	338—316 X
3,405,251	10/1968	Spriggs et al.	219—275

JOSEPH V. TRUHE, Primary Examiner

M. C. FLIESLER, Assistant Examiner

U.S. Cl. X.R.

118—49.5; 219—424, 426