May 29, 1973 J. T. CARLETON 3,736,175

VACUUM COATING METHOD

Original Filed Dec. 16, 1970

FIG. 1

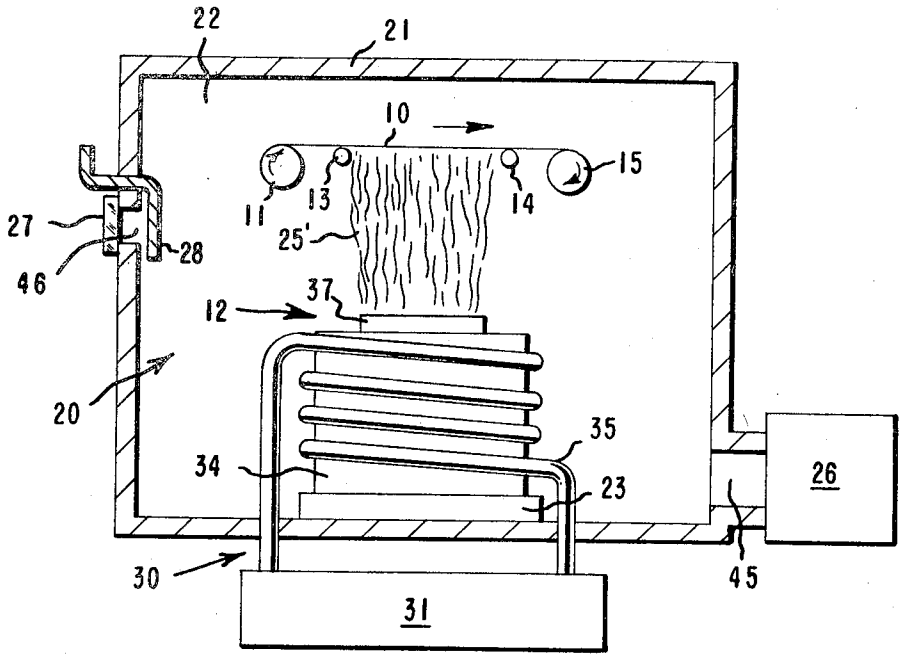
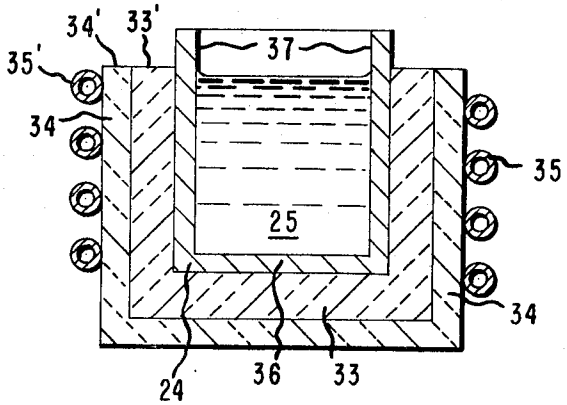


FIG. 2



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VACUUM COATING METHOD

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Continuation of abandoned application Ser. No. 98,824, Dec. 16, 1970. This application June 2, 1972, Ser. No. 259,116

Int. Cl. C23c 17/02

U.S. Cl. 117—107.1 1 Claim

ABSTRACT OF THE DISCLOSURE

In vacuum metallizing, creep within a crucible is inhibited by heating the body portion of the crucible to a temperature above the vaporization temperature of the metal coating stuff while heating a vertically extending lip portion of the crucible to a temperature which is below said vaporization temperature but above the melting point temperature of said metal coating stuff.

This is a continuation, of application Ser. No. 98,824, filed Dec. 16, 1970 and now abandoned.

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to vacuum metallizing apparatus and, more particularly, to the melting and vaporization of materials in a crucible in a vacuum environment and by induction heating.

Description of the prior art

The use of induction heating for vaporizing metal contained in a crucible in the vacuum metallizing apparatus arts is old.

The material to be vaporized is placed in a crucible which, in turn, is placed within a heating coil of an induction heating means located in the vacuum chamber. The chamber is pumped to a suitable vacuum, and normally a high frequency alternating current is applied to the coil by which the crucible and metal charge are heated inductively to vaporizing temperatures. With a number of materials the heated molten metal or melt tends to creep up the sides of the crucible, onto the lip portion, and in some instances may wick over the edges into the insulation between the heating coils and the crucible. With reactive materials, such as aluminum, the coils may be attacked and the useful life shortened. Further, the crucible and insulating attack may lead to undesirable contaminants in the vacuum chamber.

One approach to solving this problem is shown in U.S. Patent 2,665,320 to Chadsey, Jr. et al., in which the crucible lip is heated to a substantially highter temperature than the molten metal in the crucible so that the lip tends to vaporize the molten metal reaching the lips. This approach has certain disadvantages; for example, the lip radiates added heat in the chamber which must be removed by additional cooling of the structure of the chamber and the substrate or object to be coated and, more importantly, the material vaporized from the lip may travel in all directions above the lip where it may deposit in undesired areas rather than on the object to be coated. This is not only wasteful of material but increases the downtime for cleaning the chamber.

SUMMARY OF THE INVENTION

The vacuum metallizing apparatus illustrating the method of the present invention provides a means for heating a crucible in a novel manner thereby to improve the efficiency of the apparatus.

Briefly described, a crucible cointaining metal to be vaporized is positioned within a vacuum chamber of the vacuum metallizing apparatus and the lip or lip portion of such crucible is heated so it does not vaporize the metal which is vaporized by the heat supplied to the main body portion of the crucible.

This invention solves a problem existent in the prior art by providing an improved method for controlling melt (molten metal) creeping during melting and vaporization.

Such invention essentially encompasses the discovery of the improvement of heating the main body portion of the crucible hotter than the lip portion to control creeping of the molten material up the wall of the crucible toward the lip portion thereof.

It is known that certain metals when vaporized have a tendency to wet and creep up the sides of the crucible which cointains them and in which they are melted (aluminum vaporized in a graphite crucible, for example). Such a tendency is more pronounced when the crucible is heated by induction coils and the rim of the crucible is insulated to protect the coil and to increase thermal efficiency.

One of the prime disadvantages of "creeping" is that the molten metal on the sides of the crucible tends to be deposited onto the object to be metallized in a direction other than the desired vertical direction, a step of crucial and major significance in the deposition of any objection.

By locating the crucible lip portion way from the coil, the heat generated in the lip is substantially reduced and, by further removing the insulation from the lip, the heat differential between the lip portion and main body portion of the crucible is further enhanced. This heat differential tends to decrease the tendency of the liquid (molten) metal being vaporized to creep along and over the edges of the crucible and, thus, be vaporized other than in the proper direction, thus bringing about a substantial gain in increased efficiency of the deposition step.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a diagrammatic sectional view of the vacuum metallizing apparatus of this invention, with parts omitted for clarity, showing a web of sheet material being coated in a vacuum chamber by vaporized metal melted and vaporized in a crucible by means of an induction heating means surrounding the main body portion of the crucible but positioned below the lip portion thereof.

FIG. 2 is a sectional view of the crucible, the insulation therefor and the induction heating means for heating such crucible in a novel manner and in the apparatus of FIG. 1.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to the drawing and particularly FIG. 1, a web **10** of sheet material (such as plastic film) to be coated by the web coating or vacuum metallizing apparatus of this invention is supplied from a supply source, such as supply roll **11**, and moved into operative association with means **12** for coating the web **10** in a manner to be described. From the supply source **11**, the web or substrate **10** passes over transfer rolls **13** and **14** and onto a windup roll **15**, all of which parts may be appropriately mounted in a vacuum chamber of the metallizing apparatus of this invention, as is known.

In the drawing, there are shown various parts of a typical vacuum metallizing apparatus **20** which includes a gas-impervious body member or wall **21** defining a vacuum chamber **22**. Within the vacuum chamber **22** and mounted on appropriate support means **23** is a container or crucible **24** in which is contained material **25** (as shown in FIG. 2) to be vaporized so as to produce metal vapor **25'** for coating the moving web **10**.

The container or crucible **24**, wherein the material or metal **25** to be vaporized is located, has a mouth or opening positioned between the transfer rolls **13** and **14** which positions the moving web **10** adjacent the crucible opening during the metallizing operation. The crucible **24** supports therein a pool of the material **25**, such as molten aluminum, in the appropriate position to supply the stream of aluminum vapors **25'** upwardly to condense on the moving web **10** upon the melting and vaporization of the metal **25**.

Means are provided to melt and vaporize the metal **25** in the crucible **24** in the form of an induction heating means **30** mounted in the body member **21** of the metallizing apparatus. The metal **25**, upon vaporization, deposits and condenses on the web **10** near the outlet of the crucible **24** where it condenses to form a coating.

The vacuum chamber **22** is coupled through a port **46** to a source of high vacuum **26** for evacuating the vacuum chamber **22** as is known in the art.

Crucible **24** is of an electrically conductive material, such as graphite, and it rests on the base or support **23** which is insulated.

Insulating base **23** is preferably of a heat-resistant ceramic material so that occasional minor spillover of the metal in startup will not cause damage.

The chamber **22** has means defining a viewing portion including a glass **27** positioned over a port **46** and a rotatable shield **28** operable from outside the chamber **22** which is used to protect the glass **27** from being coated when not used for operation.

A power supply **31** provides the energy for induction heated means inside the vacuum chamber **22**. The frequency of the power supply may vary from 1,000 to 500,000 hertz or more depending upon the arrangement and materials of construction.

The crucible **24** containing the material **25** to be vaporized is surrounded by insulation **33**, **34** which is preferably essentially not inductively heated by the induction field provided by the heating means **30**.

The web substrate **10** may be a film together with roll mechanisms to unwind and wind up the film. There may further be cooling plates or rolls behind the substrate or film. Other well-known items such as masks to control deposition on the substrate, baffles, shutters, and cooling coils may be present. These are not shown as they are not basic to the invention.

FIG. 2 is a detailed sectional view of the crucible, insulation and heating means. The crucible **24** is constructed of suitable material such as carbon, or metal carbides and nitrides which may be heated by eddy currents. A suitable material is graphite or a combination of graphite and carbon. The crucible **24** may be made from a hollowed-out cylinder, half cylinder, open box, or other suitable shape. The walls may be coated to prolong the life and prevent attack of the molten charge **25** upon the crucible.

The basic inventive concept is applicable to a number of materials, but it is particularly directed to metals and molten aluminum.

The insulation and support materials **33** and **34** separate the crucible **24** from coils **35** of the heating means **30**. A satisfactory combination is a fibrous or powdery material which may be graphite fibers, aluminum oxide, or zirconium oxide. The outer layer may be built of ceramic paper such as "Fiberfrax" (The Corborundum Company). The induction coils **35** are made of a conductive material such as copper and may be covered with a tape such as fiberglass and cemented in place around the outer insulation **34**, as best seen in FIG. 1. The coils **35** are preferably hollow to permit the circulation of coolant.

The crucible consists of a main body portion **36** and a lip portion **37**.

The lip portion **37** of the crucible **24** is raised above the upper edges **33'** and **34'** of the insulation **33** and **34** and above the top of the uppermost induction heating coil element **35'**. Raising the lip **37**, thus causes it to be and remain cooler.

At temperatures of 1,000° C. or more, sufficient cooling may occur if the crucible lip **37** is raised less than one inch. It is to be remembered that the loss of heat by radiation varies with the fourth power of the absolute temperature, and the edge may be 200° C. or more cooler than the melt. With lower melting materials, it may be desirable to have a heat sink nearby or contacting the edge to provide the necessary temperature gradient without having the crucible edge unduly raised. It is within the concept of this invention to provide such additional cooling over the natural emission or to vary the crucible height and shape to accentuate the temperature difference.

The desired temperature difference between the molten material **25** (melt) and the lip or lip portion **37** can be determined by simple experimentation to suit the material and particular configuration of crucible and heater. The lip **37** should be above the melting point of the material **25** being vaporized, otherwise, condensation of the vapor will occur and plugging or choking of the crucible mouth may occur. Once the crucible height or heat sink condition has been established, further adjustment is not required. A few trial runs where the lip **37** is extended different distances are usually sufficient to establish the conditions. If the melt creeps to the lip **37** of crucible **24**, vaporization spreads over the chamber **22** coating the walls and the window glass **27** if the cover **28** is opened this glass **27** may be used for observation of the crucible edge with optical or infrared pyrometers.

Induction heating is heating material by means of an electric current that is caused to flow through the material or its container by electromagnetic induction. It is known that those crucibles made of materials such as graphite and combinations of graphite and carbon, while relatively inert to molten aluminum, also are readily wetted by such molten aluminum. In a crucible of this type, it has been found that the aluminum has a tendency, due to its ability to web the material of crucible, to climb up the side of the crucible and over the edge or lip thereof. This results in wasting the material, a reduction of metallizing efficiency and other problems. The solving or minimizing of this creeping problem is the touchstone of this invention.

The heating means in essence comprises an induction coil which is capable of inducing eddy currents in the metal **25** and in crucible **24** to heat the metal to the desired vaporizing temperature. Extending from the main body portion **36** of the crucible **24** is the lip portion **37**. Essentially, the induction coil stops at the upper edge of the main body portion **36**, leaving the lip portion **37** freely extending thereabove.

Since the lip portion **37** is at a lower (nonvaporizing) temperature, the material in contact with such lip portion will not be vaporized thereby and will be prevented from creeping over the upper edge or lip of the crucible.

The lower temperature of the lip portion **37**, with its direct effect on the viscosity of the molten (liquid) material **25**, slows the movement of such liquid material **25** proportionately and, thus, decreases or stops creeping of such liquid up the walls of the crucible **24**. Further, the material **25** that does creep up the walls does not vaporize due to the non-vaporizing temperature of such walls and, hence, the vapors originate only from the proper areas of the crucible **24**.

In the operation of the apparatus schematically illustrated, the web **10** which may be paper, plastic or the like, is positioned between the transfer rolls **13** and **14** in the coating chamber. The web **10** can be introduced from the outside through appropriate seals (not illustrated) or it can be mounted in the vacuum chamber **22**. The vacuum chamber **22**, where the vapor coating of the web **10** occurs is preferably pumped down by pump to a pressure of less than 1 micron Hg Abs.

The surface of the metal **25**, such as aluminum, is raised by the heating means **30** to a very high temperature on the order of 1200 to 1300° C., so copious quantities of aluminum vapors **25'** are released. Due to the high vacuum in the coating chamber **22**, the aluminum vapors **25'** travel in substantially straight lines from the surface of the aluminum pool **25**. Most of these vapors **25'** condense on the web **10** which is moved adjacent the top opening of the crucible **24** containing the metal **25**.

The following examples will aid to further point out the invention.

EXAMPLE 1

A graphite crucible 15.5 inches long by 5 inches wide with a wall thickness of ¾ inch was arranged with the top edge of crucible at the same level as the insulation and top coil of the induction heater in a vacuum chamber. The crucible surface had previously been coated with boron nitride to prevent attack of aluminum on the graphite surface. The crucible was charged with aluminum to within one-half inch of the rim. The vacuum chamber was pumped down to 10^{-4} torr. The induction heater was operated at 9600 hertz. The crucible and contents were raised to evaporating temperature (about 1400° C.). The aluminum crept up the sides of the crucible and evaporated from the edge. When the window was used to observe the evaporation, it readily fogged with aluminum deposit indicating wide-spread distribution of the aluminum vapor.

EXAMPLE 2

The procedure was repeated as for the comparative example except the crucible edge was raised one-half inch above the insulation and coil top. When evaporation was started, the process was observed through the window. The window fogging required about 10 times as long as in the comparative example. The aluminum did not creep over the edge. The edge was estimated to be about 200° C. cooler than the melt. By raising the crucible edge, the power input was slightly increased but this was more than compensated for by the decrease in chamber contamination and life of the induction coil.

By the practice of this invention, the coating efficiency of a vacuum metallizing apparatus may be dramatically improved due to the solving or minimizing of the problem of creeping of the melt during the vaporizing operation.

I claim:

1. A method of vacuum metallizing wherein an object to be metallized is placed within a vacuum chamber adjacent a crucible containing the metal to be vaporized including the step of heating, by induction heating, the main body portion of the crucible to a temperature above the vaporizing temperature of the metal thereby to form vapors to metallize the exposed surface of the object as the object is advanced past the crucible while heating a vertically extending lip portion of the crucible to a temperature lower than the vaporizing temperature of the metal and above the melting temperature of such metal.

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MORRIS KAPLAN, Primary Examiner

U.S. Cl. X.R.

118—49.5