Sept. 13, 1960 C. A. BAER ET AL 2,952,569

METHOD AND APPARATUS FORMING AN ICE SEAL IN VAPOR DEPOSITION

Filed Jan. 28, 1958 2 Sheets-Sheet 1

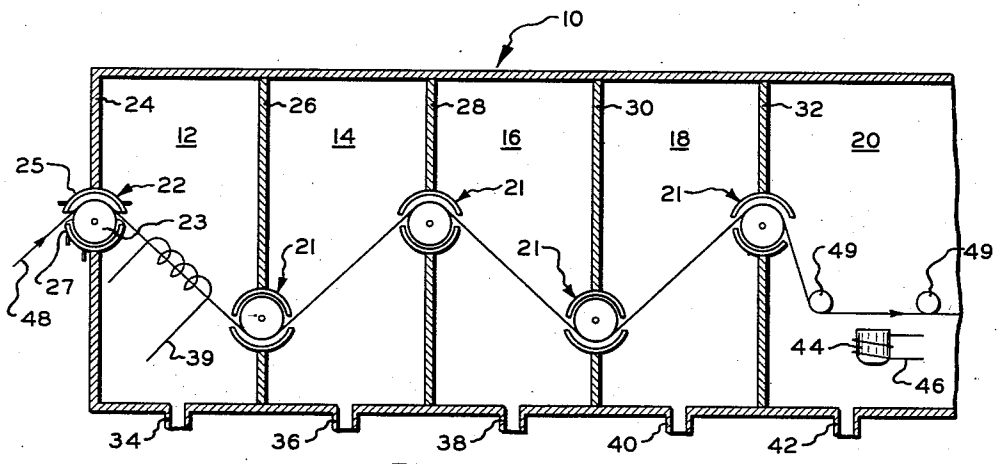


FIG. 1

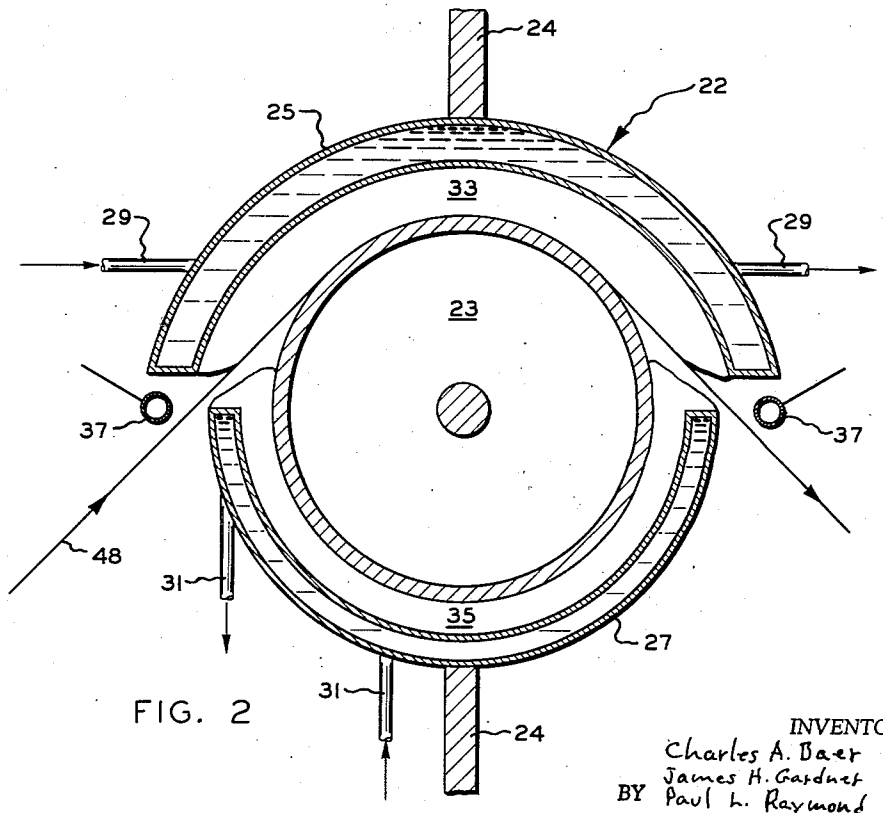


FIG. 2

INVENTORS
Charles A. Baer
James H. Gardner
BY Paul L. Raymond
Oliver W. Hayes
Wilfred J. Garbrick
ATTORNEY

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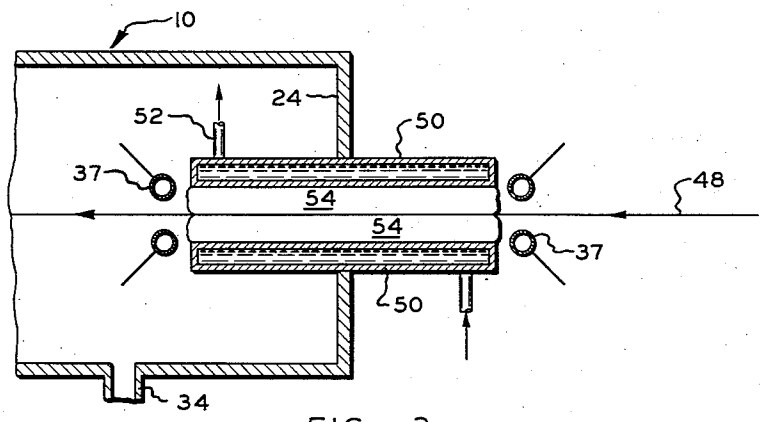


FIG. 3

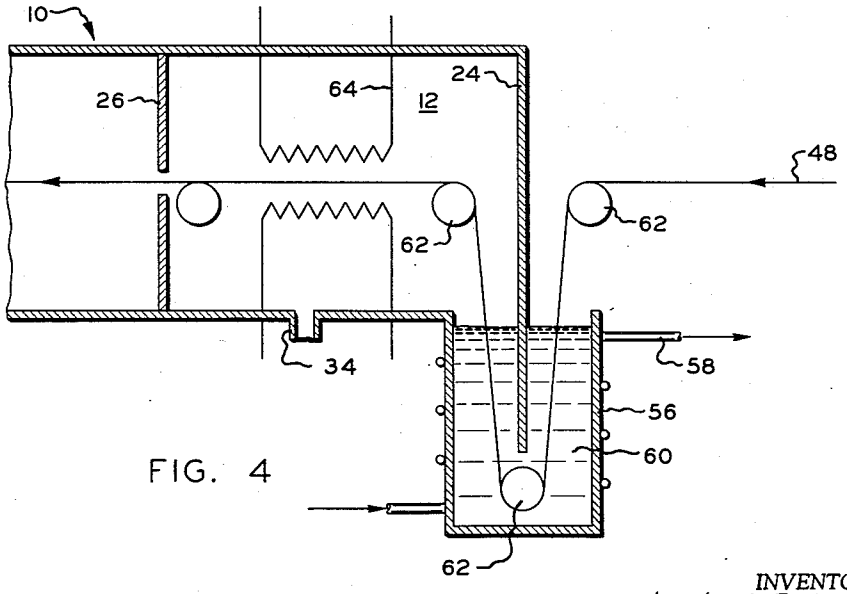


FIG. 4

INVENTORS
Charles A. Baer
James H. Gardner
BY Paul L. Raymond
Oliver W. Hoyes
Wilfred J. Baranick
ATTORNEY

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METHOD AND APPARATUS FORMING AN ICE SEAL IN VAPOR DEPOSITION

Charles A. Baer, Needham, James H. Gardner, Wayland, and Paul L. Raymond, Natick, Mass., assignors, by mesne assignments, to National Steel Corporation, a corporation of Delaware

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13 Claims. (Cl. 117—119)

This invention relates primarily to a vacuum coating and to vacuum seals suitable for continuously passing a metallic material or substrate directly from the atmosphere into a vacuum chamber forming part of a vacuum coating device.

A principal object of the present invention is to provide an improved apparatus for the continuous vacuum coating of metallic substrates.

Another object of the invention is to provide a novel type of vacuum seal for apparatus of the above type.

Still another object of the invention is to provide a vacuum seal between the atmosphere and a vacuum chamber efficient enough to effectively reduce a leakage into the chamber.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the method involving the several steps and the relation and the order of one or more of such steps with respect to each of the others and the apparatus possessing the construction, combination or elements and arrangement of parts which are exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawings wherein:

Fig. 1 is a diagrammatic, schematic view of one embodiment of the invention;

Fig. 2 is a diagrammatic, expanded view of the vacuum seal illustrated in Fig. 1;

Fig. 3 is a diagrammatic, schematic view of another embodiment of the invention, and

Fig. 4 is a diagrammatic, schematic view of still another embodiment of the invention.

The vacuum coating of a moving substrate such as sheet steel or iron with a metal such as aluminum is usually carried out in a chamber or compartment maintained under a pressure of less than about 1 micron Hg abs. The desired degree of vacuum being determined, in any particular case, by the particular metal being deposited, and the nature of the substrate being coated. Current commercial vacuum coating operations are either batch or semi-continuous types. More recently attempts have been made at truly continuous operations, that is, where the metallic strip or sheet is continuously introduced from the atmosphere into the vacuum system and removed therefrom after coating. One method proposed for accomplishing this is the introduction and removal of the metallic substrate through a series of chambers or compartments connected by low throughput apertures. By means of a suitable vacuum pumping system, a pressure gradient is maintained between each chamber.

Among the many problems associated with continuous coating operations is the extreme difficulty in preventing the entry of air from the atmosphere into the vacuum

chamber at the point of entry of the substrate and the large pumping requirements needed to maintain the vacuum chamber in communication with the atmosphere at a reduced pressure. In the present invention, there is provided a vacuum sealing means which substantially reduces the leakage of air into the vacuum chamber and the pumping requirements therefor.

The present invention is directed to an apparatus for forming a vacuum seal for continuously passing a metallic substrate through openings in vacuum chambers forming part of a vacuum coating device, said apparatus comprising elongated passages extending between vacuum chambers, means for cooling said passages to a temperature below the freezing point of water to form therein an ice seal, said ice being sufficiently extended or thick so as to contact the substrate over an extended area when said substrate is within said passages, and a means for continuously moving said substrate through said passages. In one preferred embodiment of the invention one passage extends between the atmosphere and a vacuum chamber. In another embodiment there is provided means for heating said substrate within the vacuum chambers to remove moisture from the substrate. In still another embodiment of the invention, the seal can be formed from a liquid having a freezing point less than about 20° C. and leaving a vapor pressure at 20° C. of more than 0.1 mm. Hg abs.

Referring now to Fig. 1 wherein **10** represents a vacuum coating device comprising four compartments or chambers **12**, **14**, **16** and **18**, and a coating chamber **20**, all of which are connected by means of vacuum seals **21**. Chamber **12** is in communication with the atmosphere by means of vacuum seal **22**. The chambers are defined by partitions or walls **24**, **26**, **28**, **30** and **32**. Chambers **12**, **14**, **16**, **18** and **20** are evacuated through conduits **34**, **36**, **38**, **40** and **42**, respectively, by means of suitable pumping systems, not shown. The compartments are maintained at a steep pressure gradient, the pressure decreasing from atmosphere pressure outside compartment **12** to below 0.1 millimeter of pressure inside coating chamber **20**. Within coating chamber **20** is a source **44** here illustrated as crucible means for holding the metal such as aluminum to be melted and evaporated. The source **44** is suitably heated by means **46** here illustrated as induction heating means.

A metallic substrate **48** such as sheet steel or iron is guided into chamber **12** by means of seal **22** and through the other chambers by means of a series of vacuum seals **21**. The substrate is guided over the source **44** of coating vapors by means of rollers or idlers **49**. The substrate **48** after coating is guided through another series of compartments (not shown) which are also maintained at reduced pressures and withdrawn to atmospheric pressure.

Vacuum seals **21** between the vacuum chambers can be of any suitable type, such as for example, those illustrated in the copending application, Serial No. 697,414, filed November 19, 1957, or they can be particularly between chambers **12** and **14**, like vacuum seal **22**, as long as the seal (e.g. ice) does not appreciably sublime at the pressure in the chambers. Suitable means for introducing water to form these seals are easily provided. Vacuum seal **22** can be of the types detailed in Figures 2, 3 and 4.

Referring now to Fig. 2 wherein like numbers refer to like parts of Fig. 1, the vacuum seal **22** comprises a rotatable, cylindrical roller **23** and close-fitting stationary housings **25** and **27**. Both housings are provided with means **29** and **31** for cooling the housings to a temperature the below the freezing point of water. The housings are here illustrated as hollow members provided with

means for passing a suitable coolant therethrough. The housings can also be solid members with cooling means, e.g., coils attached to surfaces most remote from the roller.

The seal is designed so as to cause the substrate **48** to follow an arcuate path of between 90 and 180 degrees and preferably of at least 120 degrees. The rollers **23** or other rollers **49** can be free moving or provided with suitable driving means.

In the operation of Figures 1 and 2 a suitable metallic substrate **48** such as sheet steel is taken directly from a production unit or line therefor or suitable reel or roll thereof and wound through the chambers in the manner indicated in Fig. 1. Receiving reels not shown are provided to take up the coated substrate after it emerges from the vacuum system. The crucible **44** is charged with metal to be evaporated, e.g. aluminum. The housings **25** and **27** are cooled to a temperature below the freezing point of water by passing a suitable coolant therethrough. When a sufficiently low temperature, e.g., minus 500° C. has been attained on the surfaces of the housings facing roller **23**, water in finely divided form such as a spray is introduced into the space or openings between the housings and the roller. The water on contacting the chilled housing surfaces solidifies. The water spraying is continued until solid ice seals **33** and **35** are built up over substantially the entire opening. The ice seals **33** and **35** are hereafter maintained by the condensation on the housings moisture contained in the atmosphere, or solidification of any water film remaining on the strip after cleaning. Intermittent spraying of water into the openings or saturation of the atmosphere with moisture in close proximity to the openings can also be used. The seal need not be completely solid, that is, it can be maintained in a state where it is composed of solids and semi-solids, e.g., slush. Thus, not being quite as hard as solid ice, it permits unrestricted substrate movement since it is easily shaven away by irregularities in the substrate, should any be present.

After the formation of the illustrated seals **33** and **35**, the chambers are evacuated by means of suitable pumps so that the pressure decreases from chamber **12** to a minimum pressure in chamber **20**. The source is heated to melt and evaporate the metal charged therein. When the desired evaporation temperature and pressures within each chamber has been attained then a thin layer of ice adjacent the substrate **48** and roller **23** is melted so as to free the roller and substrate for movement. The substrate is then continuously advanced into chamber **12**.

Only as much of the ice is melted as is necessary to permit the free movement of the substrate and roller. The melting of a thin layer of ice can be accomplished in several ways. For instance, the roller **23** can be provided internally with suitable heating means or the substrate can be connected to a suitable electrical circuit such that it will become heated by resistance. Once the substrate and roller have been freed from the ice seal, further heating is not necessary since the rapid movement of the substrate will keep it free. However, the application of some heat to the substrate is advantageous in maintaining the free movement of the substrate.

The ice seal has many advantages. For example, after the ice seal is formed the chambers are substantially completely isolated and thus the evacuation of the chambers to the desired pressures is accomplished much more readily. The ice seal is deformable and thus does not hinder the movement of the substrate. When the ice adjacent the substrate and roller is melted, to permit movement thereof, a very thin film of water exists between the ice **33** and substrate **48** and between the ice **35** and roller **23**, thus maintaining the seal effective. Additionally, the thin film of water acts as a lubricant and facilitates the movement of the substrate through the seal.

Since the substrate will contain moisture on its surfaces as it enters the vacuum system, it is heated by suitable heating means **39**, such as shown in Fig. 1. The moisture can be removed or evaporated from the substrate by heating it by induction means such as illustrated, by resistance heating means, by electron bombardment, glow discharge and the like. The presence of moisture on a substrate such as, for example, sheet steel or iron is not harmful since the moisture is in contact therewith only for a very short time and in atmospheres which are substantially oxygen-free. In order to prevent excessive build up of ice at the ends of this ice seal, suitable heating means such as radiant heaters **37** can be used.

Referring now to Fig. 3 wherein like members refer to like parts in the previous figures. In Fig. 3 the vacuum seal comprises a pair of elongated plates **50** in face-to-face relationship so as to receive the substrate, therebetween the plates **50** which extend through the wall **24** are slightly greater in width than the substrate **48** to be coated. Both plates are provided with means **52** for cooling the plates to a temperature below the freezing point of water. The plates **50** are here illustrated as hollow members provided with means for passing a suitable coolant therethrough. The plates, however, can also be solid members provided with cooling means, e.g., coils attached to the outer surfaces. The forming of ice seals **54** is as described in connection with Figs. 1 and 2.

Referring now to Fig. 4 there is shown another embodiment of the invention. In Fig. 4 the vacuum seal comprises a container **56** which is provided with suitable cooling means **58** here illustrated as coils through which is passed a coolant which will cool the water **60** in the container below its freezing point so as to form ice also designated as **60**. The wall **24** of chamber **12** extends below the surface of the water (and ice) in the container **56**. Rollers **62** suitably positioned are provided to move the substrate **48** through the container and its contents. Within chamber **12** there is provided means such as resistance heating means **64** for heating the substrate to remove the moisture therefrom. In this embodiment, when the substrate is to be moved, thin films of ice adjacent thereto are melted by causing the substrate within the seal to be heated by resistance.

The embodiment shown in Fig. 4 permits the use of liquids other than water for forming a seal. The liquid employed must be one which is (*a*) normally liquid; (*b*) easily solidifies or freezes; (*c*) easily removed from the substrate such as by heating; (*d*) substantially inert to the substrate under the conditions employed, and (*e*) non-toxic liquids having a freezing point less than about 20° C. and having vapor pressures at 20° C. of more than 0.1 mm. Hg abs. are suitable in the present invention. Liquids other than water which can be employed in the seal of Fig. 4 are chloroform, bromoform or other polyhalogenated hydrocarbons.

Since certain changes can be made in the above method and apparatus without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description, or shown in the accompanying drawings, shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum wherein the substrate is continuously passed through the coating chamber from the atmosphere, the improvement which comprises an elongated passage extending between the atmosphere and a vacuum chamber, means for cooling said passage to below the freezing point of water to form therein an ice seal, said ice being sufficiently extensive so as to contact the substrate over an extended area when said substrate is within said passage, and means for continuously moving said substrate through said passage.

2. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum, wherein the substrate is continuously passed through the

coating chamber from the atmosphere, the improvement which comprises an elongated passage extending between the atmosphere and a vacuum chamber, means for cooling said passage to below the freezing point of water to form therein an ice seal, said ice being sufficiently extensive so as to contact the substrate over an extended area, means for continuously moving said substrate through said passage, and means for heating said substrate within said vacuum chamber to remove the moisture from said substrate.

3. Apparatus of claim 2 wherein the metallic substrate is sheet steel.

4. Apparatus of claim 2 wherein said heating means comprises induction heating means.

5. Apparatus of claim 2 wherein said heating means comprises resistance heating means.

6. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum wherein the substrate is continuously passed through the coating chamber from the atmosphere, the improvement which comprises a roller and a close-fitting housing for said roller, extending between the atmosphere and the vacuum chamber means for cooling said housing to a temperature below the freezing point of water to form between said housing and said roller an ice seal, said ice being sufficiently extensive so as to contact the substrate as it follows an arcuate path around said roller, said path describing an area of between about 90 and 180 degrees.

7. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum wherein the substrate is continuously passed through the coating chamber from the atmosphere, the improvement which comprises a pair of elongated plates in face-to-face relationship extending between the atmosphere and the vacuum chamber, means for cooling said plates to a temperature below the freezing point of water to form therebetween an ice seal, said ice being sufficiently extensive so as to contact over an extended area opposite sides of the substrate as it passes between said plates and means for continuously moving said substrate between said plates.

8. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum wherein the substrate is continuously passed through the coating chamber from the atmosphere, the improvement which comprises a container of a liquid having a freezing point less than about 20° C. and having a vapor pressure at 20° C. of more than 0.1 mm. Hg abs.; said container extending between the atmosphere and a vacuum chamber, said liquid being easily removed from the substrate by heating and being substantially inert to the substrate at the temperature of the substrate while contacted by the liquid, the wall of said vacuum chamber in contact with the atmosphere extending downwardly into, and below, the surface of the liquid in said container, means for cooling said container to a temperature below the freezing point of the liquid therein to form a solid, said solid contacting opposite sides of the substrate as it follows an elongated path through said container, and means for continuously moving said substrate from the atmosphere through said container into said vacuum chamber.

9. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum wherein the substrate is continuously passed through the coating chamber from the atmosphere, the improvement which comprises a container of water extending between the atmosphere and a vacuum chamber, the wall of said vacuum chamber in contact with the atmosphere extending downwardly into, and below, the surface of the water in said container, means for cooling said container to a temperature below the freezing point of water to form ice, said ice contacting opposite sides of the substrate as it follows an elongated path through said container, and means for continuously moving said substrate from the atmosphere through said container into said vacuum chamber.

10. In an apparatus for coating a metallic substrate by vapor deposition of a coating material under vacuum wherein the substrate is continuously passed through the coating chamber from the atmosphere, the improvement which comprises an elongated passage, means for cooling said passage to below the freezing point of water to form therein an ice seal, said ice seal being sufficiently extensive so as to contact the substrate over an extended area when said substrate is within the passage, and means for continuously moving the substrate through the passage.

11. Method for forming a vacuum seal for continuously passing a metallic substrate such as sheet steel or iron from the atmosphere through an opening into a vacuum chamber forming part of a vacuum coating device which comprises continuously passing a substrate through an elongated passage extending between the atmosphere and a vacuum chamber, and cooling said passages to a temperature below the freezing point of water to form therein an ice seal.

12. Method of claim 11 which comprises heating said substrate in said vacuum chamber.

13. Method for forming a vacuum seal for continuously passing a metallic substrate such as sheet steel or iron from the atmosphere through an opening into a vacuum chamber forming part of a vacuum coating device which comprises the steps of cooling an elongated passage extending between the atmosphere and a vacuum chamber and containing the substrate therein to a temperature below the freezing point of water to form therein an ice seal, providing heat to the substrate within said passage for a short period of time to just melt the ice adjacent thereto so that said substrate is movable, thereafter, continuously moving said substrate through said passage while maintaining said ice seal therein, and heating said substrate in said vacuum chamber to remove moisture therefrom.

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