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APPARATUS AND PROCESS FOR VAPOR COATING

Filed July 19, 1950

2 Sheets-Sheet 1

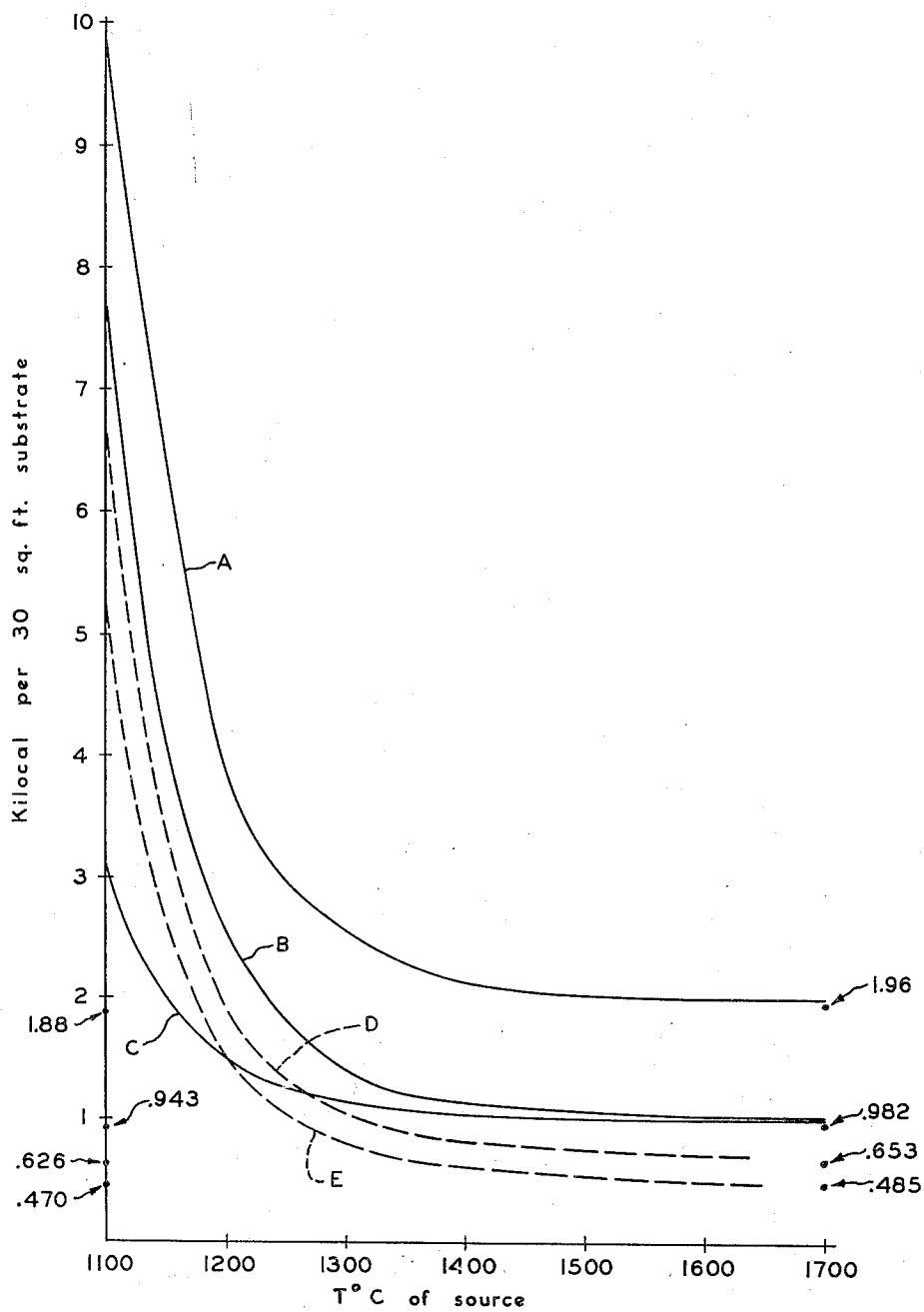


FIG. 1

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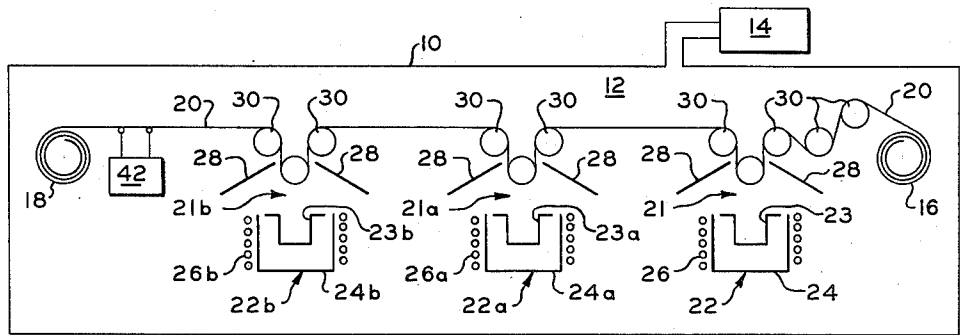


FIG. 2

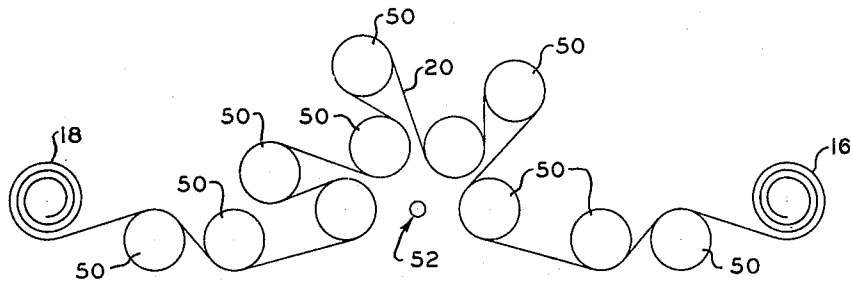


FIG. 3

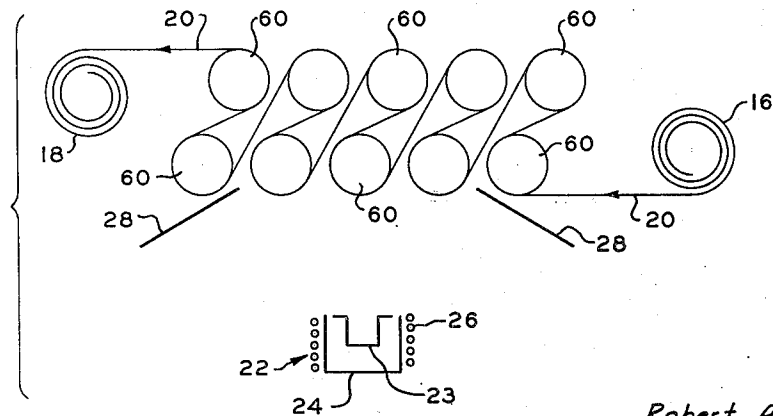


FIG. 4

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APPARATUS AND PROCESS FOR VAPOR COATING

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Application July 19, 1950, Serial No. 174,716

11 Claims. (Cl. 117-103)

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This invention relates to coating and more particularly to the coating of extremely heat-sensitive substrates with metals, such as aluminum, by vacuum deposition techniques. This invention is primarily directed to improvements in the coating techniques of the type described in the companion case of Clough et al., Serial No. 148,224, filed March 7, 1950, several aspects of the present invention being described, but not claimed, in the above companion case. This application is in part a continuation of the co-pending application of Stauffer, Serial No. 158,478, filed April 27, 1950.

In the vacuum deposition of metals, such as aluminum, on a heat-sensitive substrate, heat is transferred to the substrate from the source of the metal vapors. The effect of this heat is particularly apparent in dealing with aluminum, and the invention will be particularly described in connection with the vacuum deposition coating of aluminum on heat-sensitive substrates, such as cellophane and the like.

The heat reaching the substrate is very serious in the case of heat-sensitive materials. The heating of the surface of the substrate in severe cases can cause a chemical change. Probably the greatest difficulty, however, arises from local outgassing of the surface due to the high temperature to which the substrate is subjected as a result of the coating. This local outgassing has the effect of spoiling the aluminum coat by providing poor adhesion and nonuniformity of coating. The resultant film coated on the outgassing substrate is not shiny, and it is apt to have a bluish or black tint rather than the silver-white color desired.

In the prior art the local outgassing of such a heat-sensitive substrate has been prevented to a certain extent by attempting to outgas the substrate prior to the coating operation. This necessitates heating the substrate in a vacuum, a step which is itself apt to damage the substrate. Additionally, the apparatus necessary for carrying out such outgassing operations is relatively expensive and unduly increases the capital equipment costs necessary for coating such heat-sensitive substrates.

It is a principal object of the present invention to minimize, to a large extent, the effect of the heat reaching the substrate so that, even though the total heat reaching the substrate is of substantial proportions, the temperature of any particular portion of the substrate is not raised to a point where outgassing occurs.

Another object of the present invention is to

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provide an improved process of the above type which permits the vacuum deposition coating of extremely heat-sensitive materials without the necessity of previous outgassing thereof.

Still another object of the invention is to provide an improved coating apparatus for carrying out coating processes of the above type.

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

The invention accordingly comprises the process involving the several steps and the relation and the order of one or more of such steps with respect to each of the others, the apparatus possessing the construction, combination of 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 graph showing the degree of transfer of heat to the substrate as a function of temperature of the source and the quantity of metal coated on the substrate;

Fig. 2 is a schematic, diagrammatic view of one preferred form of apparatus embodying the present invention;

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

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

As explained in considerable detail in the above-mentioned companion application of Clough et al., the total heat transmitted to the substrate is a function of the temperature of the vapors leaving the source. This is due to the fact that the amount of radiant heat reaching the substrate decreases per gram of aluminum reaching the substrate as the temperature of the vapors is increased. This relationship is shown in Fig. 1 wherein the kilocalories reaching 30 square feet of substrate area are plotted against temperature of the vapors leaving the source. While greatly improved results are achieved by operating vacuum deposition processes at high vapor-source temperatures (i. e. in the neighborhood of 1200°-1600° C. and particularly 1300°-1500° C.) it has been found that in some cases even these improvements are not entirely adequate to prevent outgassing of extremely

heat-sensitive materials, such as cellophane and the like. This is particularly true when relatively dense coatings are desired on the heat-sensitive substrate.

In the present invention outgassing of these extremely heat-sensitive substrates is prevented by limiting the amount of heat carried to the substrate by the vapors to relatively small increments, and removing this heat at a rate sufficient to prevent the substrate from reaching that temperature at which outgassing becomes a problem. Since the heat carried to the substrate by the vapors is essentially a function of the weight of the aluminum deposited on the substrate, the reduction of the effect of the heat carried to the substrate is achieved by first coating the substrate with a relatively small proportion of the final coating thickness. This first coating is preferably about one-third, or less, of the final coat, this first coating operation being carried out with a sufficiently high vapor-source temperature so that the radiant heat is a small portion of the total heat reaching the substrate.

In a preferred embodiment of the present invention the substrate is chilled, prior to the first coating operation, to a temperature approaching, but above, that temperature at which the substrate becomes brittle. The substrate is thus able to absorb a considerable amount of the heat from the first coating operation without being heated to its outgassing temperature. This heat is then removed during and after the first coating operation and before the subsequent coating operations are accomplished. The heat of these subsequent coating operations is periodically removed so as to maintain the substrate at substantially all times below its outgassing temperature. The initial chilling and subsequent removal of heat is accomplished by contacting the substrate with cooled surfaces as it moves through the coating chamber. These cooled surfaces are preferably cooling rolls and are preferably cooled to a temperature below about 0° C. The area of the rolls contacted by the substrate is preferably sufficient to remove substantially all of the heat carried to the substrate by the vapors condensing thereon, the temperature of the rolls and the area and time of contact of the substrate with the rolls being insufficient to chill the substrate below that temperature at which it becomes brittle.

In the practice of the invention outlined above, the subsequent coating operations are preferably also carried out with a high vapor-source temperature within the preferred 1200°-1600° C. range. When more than two coating operations are employed, and the early coating operations are adequate to provide a highly reflective surface for radiant heat (i. e. above about 90% reflectance with smooth substrates), the later coating operations may be carried out at lower vapor-source temperatures since the great majority of the radiant heat is reflected from the previously coated substrate. However, best results are achieved with the high vapor-source temperatures, even when the substrate has received the highly reflective coating.

Referring now to Fig. 1 there is shown a number of curves which illustrate the various conditions under which the present invention is operated. In Fig. 1 curve A shows the total amount of heat absorbed by 30 square feet of the substrate as a function of temperature of the vapors leaving the source when .7 gram of aluminum is coated on the 30 square feet. This curve is based on the assumption that the substrate has

substantially no reflectance for radiant heat prior to coating. Curve B is similar to curve A but in this case only .35 gram of aluminum is deposited on the substrate. Curve C is similar to curve B but the substrate initially has been coated with .35 gram of aluminum to give an initial reflectance of approximately 68%. Curves D and E are similar to curves A and B but are based on coating, respectively, .233 gram and .195 gram of aluminum per 30 square feet of substrate. For convenience of illustration the calculation of the data for the curves in Fig. 1 has been based on the assumption that the source of the aluminum vapors is the sole source of radiant heat. In curve A the substrate during coating is assumed to have an over-all reflectance of approximately 61%. In curve B the substrate during coating is assumed to have an over-all reflectance of 39%, while in curve C the substrate during coating is assumed to have an over-all reflectance of 78.5%. The over-all reflectances of the substrate for curves D and E are assumed to be 26% and 20%, respectively. In the calculation of the data from which these curves are derived, the emissivity of aluminum has been assumed to be $0.2 (T^{\circ} C. - 1000) \times 10^{-4}$.

As can be seen from an examination of the various curves in Fig. 1, the total heat reaching the substrate per coating operation is a function of the vapor temperature and the amount of aluminum deposited on the substrate. When operating in accordance with curve A, the minimum heat is obtained when the temperature of the vapors is high (above 1400° C.). The same is true of curves B, C, D and E. However, the total heat reaching the substrate with curve A is about twice the heat reaching the substrate with curves B or C, is three times the heat reaching the substrate with curve D, and is four times the heat reaching the substrate with curve E. This is due to the fact that the heat carried to the substrate by the vapors only with curve A is between 1.88 and 1.96 kilocalories (depending upon the temperature), while the heat carried to the substrate by the vapors only with curves B or C is only between .943 and .982. The heat carried by the vapors only to the substrate with curves D and E is, respectively, one-third and one-quarter the heat carried by the vapors to the substrate with curve A.

Referring now to Fig. 2 there is shown one preferred apparatus for practicing the invention wherein 10 represents a vacuum-tight housing providing a vacuum chamber 12 which can be kept at a pressure in the micron range by means of a vacuum pumping system schematically indicated at 14. Within the chamber 12 there is provided a means for supporting a substrate to be coated, this means comprising, among other elements, a first spool 16 and a second spool 18 carrying therebetween the substrate 20. For supplying the metal vapors there is provided a plurality of metal-vapor sources 22, 22a, and 22b. These three sources of metal vapors comprise inner crucibles 23, 23a, and 23b, adapted to hold molten aluminum, outer crucibles 24, 24a, and 24b, and induction heating coils 26, 26a, and 26b. These sources define three coating stations, 21, 21a, and 21b respectively. The arrangement of these crucibles and their induction coils is preferably similar to that described in the above-mentioned copending application of Clough et al., the heat-radiating areas of the crucibles being preferably kept to a minimum. The metal vapors in the respective coating stations are con-

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fined by shields 23 which prevent unwanted migration of coating vapors and also prevent radiation to the substrate from the molten metal in the crucibles. For guiding the substrate during its passage through the vacuum chamber 12, there are provided a plurality of rolls 30, these rolls being preferably cooled to a temperature on the order of 0° C. or below.

In one preferred embodiment of the invention, the coated substrate is moved past a pair of rolls 40 connected to a resistance-measuring circuit 42 prior to being wound upon second spool 18. This resistance-measuring circuit is connected to a meter, speed-control device, or crucible-temperature control device. The design and operation of such a system is more fully described in the copending application of Godley, Serial No. 10,117, filed February 21, 1948. If desired, a plurality of these resistance-measuring devices may be employed, there being one for each coating station. In this case the measured resistance is preferably used to control the vapor-source temperature.

In the operation of the device of Fig. 2, the roll of substrate to be coated is positioned on the spool 16, fed around and between the various guide rolls 30, and connected to the wind-up spool 18. The vacuum chamber 12 is evacuated and the aluminum in the crucibles 23, 23a, and 23b is heated to an appropriately high temperature, as indicated by the preferred operating conditions shown in Fig. 1, this temperature being preferably about 1300° C. or above. The rolls are chilled, preferably below 0° C., by circulating a refrigerant therewithin, and the substrate is advanced through the apparatus at a speed adjusted, for example, to give a coating of .233 gram of aluminum per 30 square feet of substrate area. As shown by curve D, only a very small portion of the heat reaching the substrate (when a three-stage coating is employed and a high vapor-temperature is utilized) is attributable to radiant heat. The bulk of this heat is due to the heat carried over by the vapor. As the substrate moves from the supply roll 16 it passes, in the Fig. 2 embodiment, in contact with three chilled rolls 30 before it reaches the coating station 21. This permits the temperature of the substrate to be lowered to a temperature on the order of 0° C. or somewhat lower in those cases where the substrate does not become unduly brittle at temperatures below 0° C. As the substrate passes the coating station 21, the metal vapors in this coating station condense upon the substrate, thereby raising the temperature thereof appreciably. Since the initial temperature was quite low, the temperature of the substrate is not immediately raised above that point at which it will begin to outgas. This is also due to the fact that the back surface of the substrate is being cooled during this coating, and that the metal coating condensed on the substrate is almost immediately brought into contact with another chilling roll to remove the heat from this metal coating. This immediate chilling has the advantage that the metal coating, even though at a temperature high enough to cause outgassing, may be cooled sufficiently rapidly so that no substantial heating of the substrate takes place. The substrate is then further cooled before it enters the second coating station 21a, and is cooled during passage through this coating station. The metal coating added during this second coating station is also preferably brought immediately into contact with another cooling roll as it leaves this second

coating station 21a. The same procedure is followed in connection with the third coating station 21b.

As is apparent from Fig. 1, when the temperature of the vapors is approximately 1200° C., the total heat radiated to the substrate and carried to the substrate by the vapors at any coating station can be expressed approximately by the formula

$$\frac{4Y}{.7}$$

kilocalories for each 30 square feet of substrate, where Y is the amount of aluminum, in grams, coated on each 30 square feet of substrate at a particular coating station. When the temperature of the vapors is approximately 1300° C. or above, the approximate formula is

$$\frac{2.5Y}{.7}$$

kilocalories for each 30 square feet of the substrate. This is the amount of heat which must be removed for each coating station.

Referring now to Fig. 3 there is shown another embodiment of the invention where like numbers refer to like elements in Fig. 2. In this form of the invention a single source of metal vapors is employed, the substrate moving into and out of the vapor stream at a number of different points to give a plurality of coating stations. In Fig. 3 there are provided a plurality of cooling and guiding rolls 50 spaced around a vapor source 52. The rolls 50 are preferably arranged in two rows, the inner row serving to support the substrate during its passage through the various coating stations, and the outer row serving to chill the metal coating between coating stations. The metal-vapor source 52 is preferably a carbon rod wick of the type described more fully and claimed in the copending application of Godley et al., Serial No. 153,494, filed April 27, 1950.

The operation of the Fig. 3 device is similar to the operation of the Fig. 2 embodiment, the substrate being chilled before it reaches the first coating station, and being cooled during and between the first coating operation and the subsequent three coating operations. In the Fig. 3 device the conditions of curve E of Fig. 1 would be applicable.

In still another embodiment of the invention, illustrated in Fig. 4, a source of metal vapors of the type shown in Fig. 2 is used with a cooling and guiding roll arrangement of the general type shown in Fig. 3. In Fig. 4 the various guide rolls are indicated at 60, these being preferably arranged in two parallel rows above the vapor source 22. The vapor and radiation shields 23 prevent coating at the two end rolls on the bottom row so that the Fig. 4 device constitutes a three-stage coating device whose conditions of operation are indicated by curve D of Fig. 1.

In utilizing the present invention for coating cellophane and the like, the cooling and outgassing temperatures will vary somewhat depending upon the type of material being coated. For example, with moistureproof cellophane obtained from duPont, the grade MT-31 cellophane should not be chilled much below about 0° C. if embrittlement thereof is to be avoided. However, duPont grade MT-35 may be chilled to a temperature as low as -17° C. without embrittlement. These differences are believed to be due to the greater percentage of plasticizer in grade MT-35 (i. e. 15-16% in MT-35 vs. 14% in MT-31).

The minimum temperatures for other cellophanes and like materials may be readily determined by simple physical tests under controlled temperature conditions.

The maximum temperature to which the substrate may be subjected depends upon the substrate, the vacuum, and the length of time during which the substrate is held at the high temperature. With duPont moistureproof cellophane grade MT-31 outgassing becomes objectionable when this material reaches a temperature on the order of 40° C. Outgassing temperatures of other materials can be easily determined by coating the material in a vacuum under carefully controlled conditions. When the substrate is moved rapidly from the coating station to the coating chilling roll, the temperature of the metal coating may be allowed to rise as much as 70° C. above the temperature of the substrate. However, this high temperature must be substantially immediately removed if violent outgassing and heating of the whole substrate is to be avoided. Measured temperatures have shown that with a coating of about .233 gram per 30 square feet of substrate, and with a time lapse of ¼ second from the coating station to a cooling roll at -10° C., the substrate does not outgas appreciably even though the metal coating temperature, measured upon leaving the coating station, is about 100° C. above the temperature of the substrate entering the coating station. At the point where the metal coating temperature was measured, the temperature of the other surface of the substrate was still at the temperature of the substrate upon entering the coating station. Thus it can be seen that an appreciable length of time is necessary for the heat in the metal coating to diffuse into the substrate and cause outgassing. When the hot metal coating encounters the cooling roll, the heat in the metal coating is rapidly withdrawn and the heat does not substantially penetrate the substrate. When the cooling roll is at about -10° C. the temperature of the metal coating is brought to the temperature of the substrate after contact with the roll for only ¼ second.

When the operation of the devices of Figs. 2, 3, and 4 is commenced, it is desirable that the substrate be moved while the rolls are being chilled so as to prevent excessive local chilling of the substrate at those points in contact with the rolls. This is particularly important when the rolls are chilled to temperatures much below the embrittlement temperature of the substrate. Otherwise, breakage of the substrate may occur. To prevent undue chilling of the substrate a temperature-measuring means may be employed for measuring the temperature of the substrate as it passes the various rolls, and the speed of the substrate or degree of cooling of the rolls may be varied in accordance with the indicated temperature. This expedient is particularly desirable when the rolls are being chilled initially and no coating is being accomplished.

In the various forms of apparatus shown in the drawings, cooling rolls are indicated as the preferred cooling surfaces. Rolls have a number of advantages which make their use particularly desirable. They can be, and preferably are, driven so as to prevent undue strain on the substrate. Additionally, when the roll surface is traveling at the same speed as the substrate, there is no danger of scratching the freshly deposited metal coating.

It is preferable that the rolls be warm when the pressure in the vacuum chamber is above about 200 microns Hg abs. Otherwise moisture in the air may condense on the rolls and cause difficulty in pumping down the system to pressures below one micron, which low pressures are desired when coating with aluminum.

Since certain changes may be made in the above process 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. A process for coating a heat-sensitive plastic substrate by vacuum-evaporating aluminum and condensing aluminum vapors on the substrate, said process comprising the steps of providing a plurality of coating stations in a vacuum chamber, evacuating said chamber, evaporating aluminum in said chamber, moving said substrate past said coating stations, contacting said substrate by a stream of aluminum vapors during movement past each of said coating stations, substantially immediately after passing each coating station bringing said metal coating in contact with a surface cooled to a temperature below about 0° C. and maintaining said metal coating in contact with said cooled surface until substantially all that heat is removed from the substrate which was transmitted to said substrate by the metal vapors which condensed to form said metal coating, and maintaining at a temperature above about 1300° C. those vapors contacting said substrate prior to the time when it has a reflectance of about 70%, the metal coating applied at each coating station being brought into contact with said cooled surface in less than about one second after formation thereof.

2. A process for coating a heat-sensitive plastic substrate by vacuum-evaporating aluminum and condensing aluminum vapors on the substrate, said process comprising the steps of providing a plurality of coating stations in a vacuum chamber, evacuating said chamber, evaporating aluminum in said chamber, moving said substrate past said coating stations, contacting said substrate by a stream of aluminum vapors during movement past each of said coating stations to deposit an aluminum film on said substrate, immediately cooling said substrate after leaving each coating station by contacting the deposited aluminum film with at least 180 arcuate degrees of cooling surface in less than one second after leaving each coating station, maintaining a greater area of deposited aluminum film in contact with said cooling surfaces than the area of the substrate being coated at any instant of time to remove from the substrate substantially all that heat transmitted to the substrate by the metal vapors which condensed to form the aluminum coating, and moving said substrate and said cooling surfaces at the same speed.

3. Apparatus for coating a heat-sensitive plastic substrate by vacuum-evaporating aluminum and condensing aluminum vapors on the substrate, said apparatus comprising means providing a plurality of coating stations in a vacuum chamber, means for evacuating said chamber, means for evaporating aluminum in said chamber, means for moving said substrate past said coating stations so that said substrate is contacted by a stream of aluminum vapors during movement past each of said coating stations, cooling sur-

faces positioned within said chamber so as to contact said metal coating substantially immediately after the substrate passes each coating station, and means for maintaining said cooling surfaces at a low temperature, said cooling surfaces comprising a plurality of cooled rolls, said cooled rolls being arranged so that the freshly applied aluminum coating on the substrate engages at least 180° of roll surface between coating stations to remove from the substrate substantially all of the heat which was transmitted to said substrate by the metal vapors which condensed to form said metal coating, a plurality of said rolls being positioned adjacent a single source of aluminum vapors so that the substrate is guided into and out of spaced portions of the aluminum vapor stream emanating from the single source, the roll arrangement providing a plurality of coating stations adjacent the single source of aluminum vapors, with substantially complete removal, between coating stations, of the heat transmitted to the substrate at each coating station.

4. The process of claim 1 wherein said substrate is chilled, before it reaches said first coating station, to a temperature near, but above, that temperature at which it becomes brittle.

5. The process of claim 1 wherein said cooling surfaces are arcuate and said substrate contacts at least 180° of cooling surface between coating stations.

6. The process of claim 1 wherein said cooled surface comprises at least one roll cooled to a temperature between about 0° C. and -80° C., the metal coating being moved out of contact with said roll surface prior to the time when said substrate reaches a temperature at which it becomes brittle.

7. The process of claim 1 wherein said substrate comprises moistureproof cellophane and said substrate is contacted by a surface cooled below 0° C. prior to entering the first coating station.

8. The process of claim 1 wherein said cooling surface means removes, after each coating station, about 2.5 to 4 times

$$\frac{Y}{.7}$$

kilocalories for each 30 square feet of substrate, where Y is the amount of aluminum, in grams, coated on each 30 square feet of substrate at each coating station.

9. Apparatus for coating a heat-sensitive plastic substrate by vacuum-evaporating aluminum and condensing aluminum vapors on the substrate, said apparatus comprising means pro-

viding a plurality of coating stations in a vacuum chamber, means for evacuating said chamber, means for evaporating aluminum in said chamber, means for moving said substrate past said coating stations so that said substrate is contacted by a stream of aluminum vapors during movement past each of said coating stations, cooling surfaces positioned within said chamber so as to contact said metal coating substantially immediately after the substrate passes each coating station, means for maintaining said cooling means at a low temperature, said cooling surfaces comprising a plurality of cooled rolls, said cooled rolls being arranged so that the freshly applied aluminum coating on the substrate engages at least 180° of roll surface between coating stations to remove from the substrate substantially all of the heat which was transmitted to said substrate by the metal vapors which condensed to form said metal coating, and means for maintaining at a temperature above about 1300° C. those vapors contacting said substrate prior to the time when it has a reflectance of about 70%.

10. The apparatus of claim 9 wherein means are provided for driving said rolls at the same surface speed with which the substrate is advanced past said coating stations, said rolls being arranged so that the area of applied metal coating in contact with the rolls is greater than the area of substrate being coated at any instant of time.

11. The process of claim 2 wherein at least two of said coating stations comprise a single volume of high temperature aluminum vapor into and out of which said substrate is moved a plurality of times, the metal film on the substrate being advanced into contact with a substantial area of cooling surface immediately after leaving the volume of aluminum vapors.

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