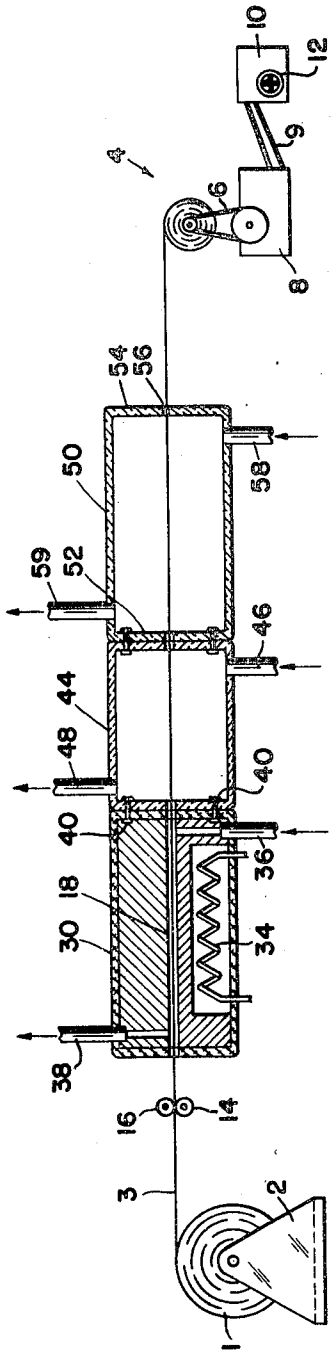
July 28, 1959 H. J. HOMER ET AL 2,897,091

METHOD OF HIGH SPEED GAS PLATING OF SYNTHETIC RESINS

Filed Oct. 27, 1954

FIG-1



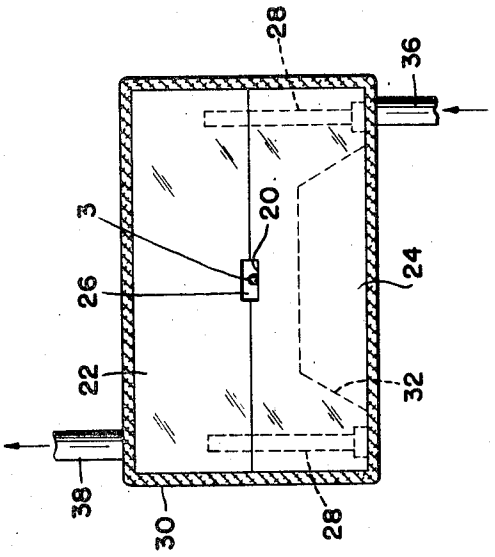




FIG-2

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# METHOD OF HIGH SPEED GAS PLATING OF SYNTHETIC RESINS

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3 Claims. (Cl. 117—47)

This invention relates to the metallizing of fibers, fabrics, and the like, and is particularly directed to the metallizing of fibers and fabrics formed synthetically and which fibers normally tend to soften and to be altered deleteriously physically when subjected to temperatures required for metallizing.

The invention particularly contemplates a method for the production of metallized fibers, fabrics or filaments in the form of continuous lengths in such manner that the material is not subjected to deleterious distortion even though the plating temperature utilized is above the temperature at which the material would normally soften. This is accomplished in the practice of the invention by directing the material in the form of continuous lengths across a hot plate at such a speed that the skin temperature of the material is raised but the body temperature is substantially unaffected.

In the materials of the type described the heat conductivity is relatively poor and accordingly the transmission of heat from the surface skin to the interior is slow. Accordingly the surface skin may be raised to a temperature above that at which the material would normally be softened, and by metallizing the material while the skin only is heated—transmission of sufficient heat to the interior of the body of the material to distort the same is inhibited.

A particular object of the present invention is to provide apparatus which is capable of effecting the above noted mode of operation. Such apparatus comprises in combination means to draw the material for example in filamentary form at a speed of at least about 150 feet per minute across a heated surface such as a hot plate which itself would normally have a length of only about 3 to 5 inches. Such is sufficient when the hot plate is at an elevated temperature to quickly raise the skin temperature of the material.

For the purpose of metallizing there is associated with the hot plate a chamber which is adapted to receive therethrough a heavy concentration of a heat decomposable metal bearing gas. The material leaving the hot plate enters this chamber with the skin temperature above both the normal distortion point of the material and above the decomposition temperature of the heat decomposable metal bearing compound, and accordingly the gases coming into contact with the hot skin decompose and deposit an adherent sheeting of metal over the rapidly moving filament.

Means are provided in the apparatus of invention for winding the filament which itself cools in the gas plating chamber, due both to the contact of the filament with the metal bearing gases and the gases of decomposition evolved upon the breakdown of the metal bearing gases. Thus there is little residual heat in the filament as it is wound and substantially no opportunity is presented for distortion of the material.

In the practice of the process of the invention it is desirable to provide the hot plate within a substantially enclosed chamber and to provide over the hot plate an



atmosphere of a non-oxidizing inert gas, such as carbon dioxide or nitrogen. Most suitably this inert gas flows counter-current to the movement of the material and serves to inhibit not only oxidation of the material but also provides somewhat of a lubricating action to inhibit any tendency of the material to adhere to the hot plate.

Preferably the pressure of this gas is above atmospheric and the pressure may be used to control the noted lubricant quality when the material is operated upon in such manner that the skin temperature reaches or may reach a critical point. Thus the flowing gas serves to increase the scope of utility of the process.

Different materials will respond in a different manner to the action of heat both at the skin and in the body thereof and the necessity for control will also depend to some extent upon the nature of the plating gas.

Materials which are useful in the practice of the invention include nylon, which softens at about 320° F. and completely melts at 482° F., Orlon, most varieties of which soften at a temperature of about 450° F., saran (a vinylidene chloride) which softens at 240 to 280° F., polyethylene, which softens at from 220 to 230° F., polystyrene, 190 to 230° F., and Vinyon at 167 to 176° F.

It is possible with the practice of the invention to raise the skin temperature of the fibers well above the normal softening point without detriment to the product, if the same is quickly cooled as by immersion in the atmosphere of plating gas hereinbefore described. The heating surface may suitably be in the form of a flat plate, or for example, a tubular member over the interior surface of which the fibers are passed, but in general it is preferred that the surface be highly polished for optimum heat transfer and a minimum of abrasion.

Accordingly it is now feasible, following the principles of the invention, to plate a relatively heavy film of adherent metal on the quickly moving material since sufficient heat will be retained throughout the passage of the plating chamber for deposition on the material over substantially the full length of the chamber.

Plating gases which are useful in the practice of the invention include particularly nickel carbonyl which decomposes substantially completely at a temperature in the range of 375 to 400° F. and which decomposes more slowly at temperatures down to as low as 175° F.; iron pentacarbonyl decomposes well at a temperature in the range of 400–600° F.; chromium carbonyl decomposes at a temperature of between 375 to 450° F., molybdenum at temperatures between 450 to 650° F., tungsten carbonyl at temperatures between 525 and 775° F.

It will thus be noted that particularly for the deposition of molybdenum and tungsten it is essential that the skin temperatures of materials referred to hereinbefore be raised in some way above their normal softening points in order to effect the attainment of a suitable metallic deposit. Such is afforded by the practice of the present invention.

The invention will be more fully understood by reference to the following detailed description and accompanying drawings wherein:

Figure 1 is a schematic representation in elevation, of apparatus, including the heating chamber, useful in the practice of the invention; and

Figure 2 is a left hand end view of the heating chamber of Figure 1.

Referring to the drawings there is shown at **1** in Figure 1 a reel of nylon yarn which is to be metallized with molybdenum. The reel **1** is supported on a stand **2** for rotation and the yarn indicated at **3** is drawn rightwardly in the figure through the apparatus and wound up on a

reel at 4, which reel is powered through power transmission means, including belting 6, gear box 8 and motor 10 which is connected to the gear box through a variable speed drive belt 9. A handle 12 permits manual adjusting of the position of the belt in a known manner and provides for varying speeds of drawing of the yarn 3.

Referring again to the leftward portion of Figure 1 there is shown at 14, 16 a pair of guide rolls through which the yarn 3 is passed into the heating chamber 18 over the heating plate indicated at 20 in Figure 2.

As more clearly shown in Figure 2 the heater 18 comprises a pair of heavy copper or other suitable metal bars 22, 24, each of which is slotted longitudinally to provide an opening 26 through which the yarn 3 passes in its traverse. The copper bars are suitably retained together by screws 28 and the combined body is provided with heat insulation 30. The lower copper bar 24 is cut out to provide a central removable portion 32 onto which there is inserted an electrical resistance element 34, which is of such a nature as to permit the attainment of a temperature of approximately 1200 to 1300° F. The copper plate in which the resistor is substantially embedded is raised to this temperature and at equilibrium the surface 20 will be at the same temperature.

Rightwardly in the figure the heater is provided with an inlet 36 which extends through the copper bar and communicates with the slot 26. Through this conduit or inlet there is passed in the practice of the invention a heavy flow of carbon dioxide and an outlet 38 is provided in order to attain continuous flow of $CO_2$ leftwardly in Figure 1 through the slot. Most suitably this outlet is connected to a vacuum source operating at a pressure of about 13–14 pounds per square inch absolute in order to provide a continuous flow through the slot.

The entire heater assembly, that is the metal portion thereof, shuold not be longer than about 5 inches generally, and may suitably be as low as 3 inches. In the latter case it may be preferable to use other higher melting point metals than copper for the heater surface in order to safely attain the required temperature without softening of the heater metal at the slot and in order to adequately heat quickly the materials described hereinbefore.

Referring again to Figure 1, lag screws 40 are secured to the right hand end of the heater 18 and a short chamber 44 of insulating material such as mica, and through this chamber there is passed a continuous flow of carbon dioxide. This chamber itself need not be more than about 2 inches in length, and in fact is substantially a short pipe.

The carbon dioxide which enters the chamber is preferably warmed prior to introducing it into the chamber in order that there will be substantially no, or at least a minimum cooling, of the yarn which passes therethrough. In given instances this chamber may be eliminated from the apparatus; but it is effective to isolate the heater from the gas plating chamber itself which is required to be maintained cool.

The inlet 46 and the outlet 48 of the chamber 44 provide for the passage of inert gas such as carbon dioxide. The gas plating chamber 50 is most suitably of glass to permit visual inspection of the process and is itself inwardly flanged at 52 to provide means for securing the same to the mica chamber 44. Similarly, at the outlet the chamber 50 is provided with inward flanges 54 which define a narrow slit opening 56 for the exit of the rapidly moving yarn.

Most suitably the mica chamber is connected to a source of vacuum operating at a pressure of 12–14 pounds per square inch absolute, and accordingly there is some tendency for the carbon dioxide, as well as the plating gas described hereinafter, to be drawn toward the mica chamber and to be expelled through the vacuum system. This eliminates the necessity for providing close fitting seals at the right hand end of the gas plating chamber, as substantially no, or very little, plating gas, even though it be higher than atmospheric pressure, tends to course to the opening 56 and that gas which does so course will not deposit metal at the opening due to the lower temperature prevailing there. Due to the lower pressure in the mica chamber, gas on the contrary tends to flow almost completely in the direction of the mica chamber, and most suitably both the mica chamber 44 and plating chamber are connected to a recovery system for the metal bearing compound.

Plating gas, molybdenum carbonyl, is introduced into the chamber 50 through inlet port 58, travels leftwardly through the chamber about the yarn and through the outlet 59 to a recovery process. The outlet most suitably is under a relatively low vacuum pressure, for example, about 2 pounds per square inch absolute, and accordingly the flow of plating gases is substantially completely counter-current to the movement of the yarn.

In given instances, if desired, the carbonyl may be warmed prior to entry into the plating chamber, but such is not necessary as the heat of the skin of the yarn itself is ordinarily sufficient to effect adequate deposition of metal on the rapidly moving yarn strand. It is necessary, however, in order to effect a thorough coating of a thickness of, for example, up to a mil, that the carbonyl be flowed through the chamber 50 in a voluminuos quantity. Accordingly much of the carbonyl will not be decomposed, but that which does contact the heated yarn will, when the yarn is at a temperature above about 450° F., deposit substantial amounts of molybdenum below about 450° F., the deposit is less rapid and is effective substantially only to increase the total deposition.

In the specific arrangement described in connection with Figure 1 the yarn may be pulled through the equipment at a rate of about 200 feet per minute, the length of the heating element being about 4 inches, and the temperature attained by the yarn skin about 800–850° F. The plating chamber 50 itself is suitably about as long as the heating surface; due to the flow of gases over the yarn, will be much slower than the heating as the latter is accomplished by contact with hot metal, and accordingly a plating chamber which is greater in length than the heating element itself may be effectively employed.

The yarn may attain a metallic coating quite readily, having a thickness of between about 1 to2½ microns, and with the yarn itself on the order of about 20 microns, it will be appreciated that a substantial thickness of metal is deposited.

Further it is to be noted that the metallic coating will be substantially pure molybdenum since there is little opportunity for the formation of carbide as the gases of decomposition, including carbon monoxide, are well diluted by the voluminous quantity of plating gas, and are further extracted rapidly from the plating area.

In a further embodiment tungsten carbonyl may be utilized as the plating medium and in this instance Orlon, which as noted hereinbefore, normally softens at about 450° F., may be employed as the fibrous material. To secure adequate decomposition of the tungsten it is necessary to raise the Orlon to a temperature of about 600° F., and for this purpose the heating plate may be about 5 inches long, the temperature thereof about 1000° F. and the speed of the traverse of the Orlon about 150 feet per minute.

The plating in this instance is also effected by the flowing of voluminous quantities of the carbonyl passed the skin heated yarn and a deposition of metal of about 1 to 1½ microns attained before the yarn is cooled below the minimum decomposition point of the tungsted carbonyl.

The plating of the lower softening point materials, such as Vinyon, may be suitably effected with nickel carbonyl, which has its most effective decomposition rate at about 350–450° F., but which as hereinbefore noted, decomposes to some extent at lower temperatures. The Vinyon which softens at the notably low temperature of 176° F.

must be moved very rapidly over the hot plate, and hence a speed of about 300 feet minute is preferred with a copious counter-curent flow of carbon dioxide over the heating plate.

The skin temperature may be adequately raised into the decomposition zone of the carbonyl without deformation of the material and a coating thickness of 0.5 to 1 micron attained in a one-pass operation.

The metallized filaments described are not affected deleteriously by the heat, and in fact in the case of nylon particularly, the breaking strength thereof is increased by approximately 10 percent.

The yarn may be readily formed into fabrics as the metallization does not impede the flexibility, and in the case of colored yarns, for instance, colored acetates with very thin metallic deposits, the coloring is not destroyed but is in fact enhanced.

It is further to be noted that by simply making the equipment of suitable width fabrics may be metallized in the same manner, and tapes are particularly suitable for the practice of the invention, such as for example, tapes of resinous materials of the nature of the chemical constituents already described. When so metallized the fabrics become substantially impervious to the passage of moisture, and accordingly are useful as backings in vapor barrier structures, as well as for the formation of draperies and curtains. This is particularly so when the material metallized is inorganic in nature, such as glass, the low melting glasses being particularly important in this respect.

It is to be noted that tapes and fabrics may likewise be metallized with the apparatus of invention simply by providing a suitable width thereof. Such metallized fabrics and tapes when completely surface coated are resistant to the passage of moisture or liquid seepage and may be suitably used, for example, in wallpaper. Further the metallization eliminates the tendency of tapes and fabrics to develop static charges and accordingly the



material is useful in belting. Further the metallization tends to render material such as rope flame proof.

It will be understood that this invention is susceptible to modification in order to adopt it to different usages and conditions and accordingly, it is desired to comprehend such modifications within this invention as may fall within the scope of the appended claims.

We claim:

1. In the method of metallizing heat-sensitive organic filamentary materials which comprises the steps of moving the material in continuous lengths through a heated chamber to raise the temperature of the material and then moving the material while heated into a gas plating chamber and in contact with a metal bearing thermally decomposable gaseous metal compound which is heat-decomposed at the temperature of said material whereby metal is deposited on the material, the improvement which consists in raising the skin temperature of the material at the surface only higher than the temperature of said thermally-decomposable compound and sufficient to cause the skin portion of the material to soften without raising the temperature of the body portion to a point where it softens, and contacting the material while thus heated with said thermally decomposable compound to effect metal deposition.

2. The method in accordance with claim 1, and wherein said thermally decomposable gaseous metal compound is a metal carbonyl.

3. A product made in accordance with the method as set forth in claim 1.

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