Nov. 4, 1958 H. A. TOULMIN, JR 2,859,130

METHOD FOR GAS PLATING SYNTHETIC FIBERS

Filed June 16, 1954

Fig. 1.

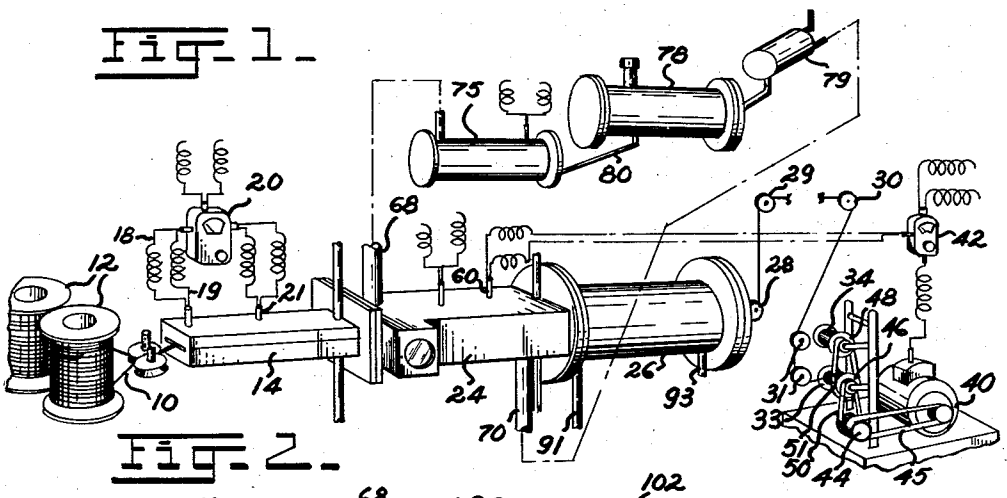


Fig. 2.

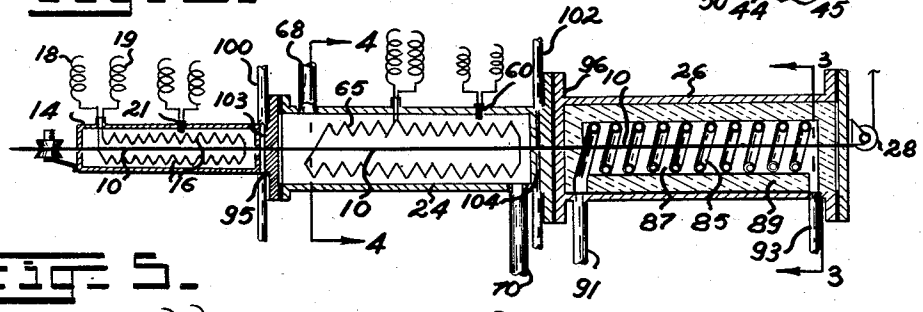


Fig. 5.

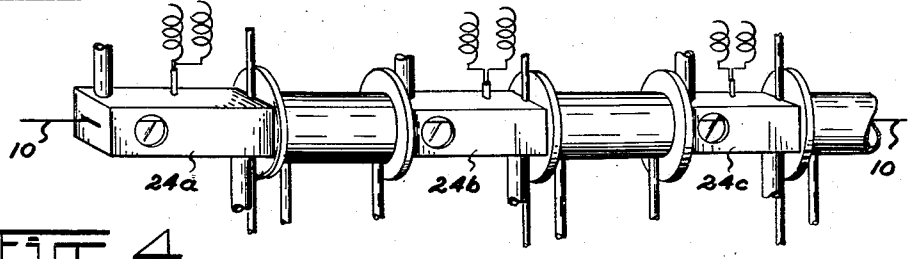
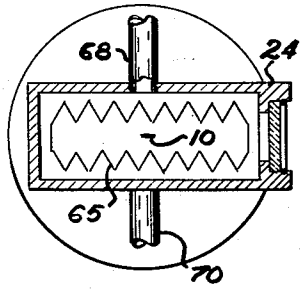


Fig. 4.



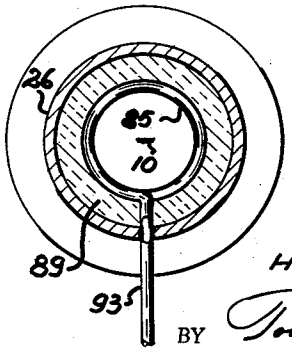


Fig. 3.

INVENTOR

HARRY A. TOULMIN JR.

BY Toulmin & Toulmin.

ATTORNEYS

United States Patent Office 2,859,130
Patented Nov. 4, 1958

2,859,130

METHOD FOR GAS PLATING SYNTHETIC FIBERS

Harry A. Toulmin, Jr., Dayton, Ohio, assignor to The Commonwealth Engineering Company of Ohio, Dayton, Ohio, a corporation of Ohio

Application June 16, 1954, Serial No. 437,085

1 Claim. (Cl. 117—47)

This invention relates to the metal plating of fibers, fabrics and the like, and particularly to fibers and fabrics which are made of natural or synthetic resins and which fibers tend to soften or change physically when subjected to temperatures ordinarily required for effecting the metal plating operation.

This invention is especially adapted for metal plating of natural and/or synthetic resin fibers or mixtures thereof, or fabrics woven or fabricated therefrom, and which fibers or fabric tend to deteriorate, discolor or otherwise change physically when subjected to relatively high temperatures, and such as are generally employed in gaseous metal plating.

The invention is of particular utility for gaseous metal plating of synthetic fibers or fibrous masses such as are made principally of synthetic fibers or filaments, for example, nylon, Dacron, Orlon, Dynel, Acrilan, Saran, Vinyon and the like.

The invention will be described with more particularity as fibers or filaments of continuous length, but it will be understood as aforementioned that such fibers or filaments may be in the form of fabricated products such as fabrics, mats, either woven, matted or otherwise formed into articles which can be subjected to gaseous metal plating.

It is an object of the invention to provide a method and apparatus which can be employed to effect metal plating of fibers, filaments and the like by bringing the same in contact with a suitable heat decomposable gaseous metal compound or mixtures of such compounds and employing temperatures sufficiently high to cause decomposition of the gaseous metal compound and deposition of the metal constituents thereof onto the surface of the fibers without causing softening or injury to the fibers thus treated.

Another object of the invention is to provide a method of treating fibers composed of synthetic resins and the like and which have a relatively low softening or melting point whereby the same can be gaseous metal plated even at temperatures above their softening point.

Another object of the invention is to provide an improved method of treating fibers, especially synthetic resinous fibers, so as to make it possible to gaseous metal plate them by limiting the period of time that the synthetic resinous fibers are in contact with the heat decomposable gaseous metal plating compound, and whereby the fibers are not subjected to a temperature for long enough time to cause softening or melting of the fibers.

Another object of the invention is to provide an apparatus wherein fibers of the character described can be gaseous metal plated, such apparatus comprising a chamber for carrying out gaseous metal deposition in combination with means for drawing the fibers through the chamber at a predetermined rate which may be varied depending upon the fiber being plated.

A still further object of the invention is to provide an apparatus through which fibers may be drawn as a continuous length fiber and subjected to gaseous metal plating, provision being made to move one or more fibers, filaments, or fabric formed of the fibers, therealong through said chamber at a predetermined speed, the duration of exposure of the fiber to the temperature in the gas plating chamber, and which temperature may be above the established softening point of the particular fiber, being controlled and limited so as to prevent deterioration of the fiber during gaseous metal plating.

A still further object of the invention is to provide an improved apparatus and method for treating the fibers, both synthetic and natural, to gaseous metal plating and wherein the heating of the heat decomposable gaseous metal compound in contact with the fibers, and time of exposure thereto is controlled so as to prevent softening or deterioration of the fiber or fibers during gaseous metal plating of the same.

This and other objects and advantages will become apparent from the following description and reference to the drawing, wherein one embodiment of the apparatus and method of gas plating fibers is illustrated.

In the drawing:

Figure 1 illustrates, in perspective and diagrammatically, a suitable apparatus for gaseous metal plating continuous fiber lengths in accordance with this invention;

Figure 2 is a vertical sectional view taken through the apparatus illustrated in Figure 1, being taken substantially on the line 2—2 of Figure 1;

Figure 3 is a cross-sectional view taken substantially on the line 3—3 of Figure 2;

Figure 4 is a cross-sectional view taken substantially on the line 4—4 of Figure 2; and

Figure 5 is a view in perspective, similar to Figure 1, with the omission of the fiber drawing mechanism and illustrating a modification thereof wherein multiple gaseous metal plating chambers and cooling means are provided for carrying out successive and continuous gas plating treatments.

Attempts heretofore made to metal plate fibers, both natural and synthetic, have been unsuccessful, particularly where the fiber is composed of material such as synthetic resin or organic or animal substances which tend to soften or deteriorate at the temperature required for effecting the gaseous metal plating. The plating operation utilizing heat decomposable metal compounds requires that the temperature be raised high enough to cause the gaseous metal compound to decompose and deposit the metal constituent on the fibers. Each heat decomposable metal compound has a temperature at which decomposition takes place. However, decomposition may take place slowly at a somewhat lower temperature or while the vapors are being heated up through a particular range. For example, nickel carbonyl almost completely decomposes at a temperature in the range of about 375° F. to 400° F. This carbonyl, however, starts to decompose slowly at about 175° F. and decomposition continues during the time of heating from about 200° F. to 380° F. A large number of metal carbonyls and hydrides also become effectively and efficiently decomposed at a temperature in the range of 350° F.–400° F. When working with most metal carbonyls it is preferred to operate in a temperature range of about 375° F. to 450° F.

As will be seen, in order to deposit metal coatings on the fiber or fabric materials exposed thereto, it is necessary that the same be subjected to a temperature in the general decomposition range of the gaseous metal compounds used to effect the metal plating.

This has presented a very difficult problem which, insofar as is known, has not been solved heretofore. The present invention provides an apparatus and method for achieving this.

In accordance with the preferred method, the fiber, either in the form of continuous length filaments or fabric, is subjected to a pre-warming treatment and then drawn through a gaseous metal plating chamber wherein the same is subjected to gaseous metal plating and immediately thereafter the fiber is cooled by moving the same through a refrigerating chamber. The fibers or fabric which may be made of synthetic resin having a softening or melting point below the temperature of gaseous metal plating, is prevented from deteriorating during the gaseous metal plating by limiting the period of time in which the fiber is subjected to gaseous metal plating. Thus, by subjecting the fiber to a controlled time of treatment a flash gaseous metal deposition may be effected without bringing about injury or physical change of the fibers or fabric material being treated.

In accordance with the process, a stream of gaseous material is brought in contact with the fiber, preferably pre-warmed and the gaseous metal plating carried out before the fiber becomes heated to a temperature high enough to cause deterioration. The gaseous plating atmosphere may be formed by mixing an inert gas with the vapors of a volatile metal compound which is heat decomposable or by atomizing a liquid metal compound into a blast of hot inert gas or other equivalent method which provides a substantially gaseous metal for deposition onto the fibers.

As the inert gas there may be used carbon dioxide, helium, nitrogen or the like which is inert to the gaseous metal compound and to the fibers being treated.

Metal to be deposited may be introduced as gaseous metal carbonyls or vaporized solutions of the metal carbonyls, in readily vaporizable solvents, for example, petroleum ether or the like, also nitroxyl compounds or hydrides may be used such as nitrosyl carbonyls, metal hydrides, metal alkyls, metal halides, and the like.

Illustrative compounds of the carbonyl type are nickel, iron, chromium, molybdenum, cobalt and mixed carbonyls.

Illustrative compounds of other groups are the nitroxyls, such as copper nitroxyl; nitrosyl carbonyls, for example, cobalt nitrosyl carbonyl; hydrides, such as antimony hydride, tin hydride; metal alkyls, such as chromyl chloride; and carbonyl halogens for example, osmium carbonyl bromide, ruthenium carbonyl chloride, and the like.

An important factor which results in the successful operation of the method and apparatus for gaseous metal plating the fibers and fabrics of the character aforementioned, is that of controlling the speed of the passage of the fiber material through the gaseous plating chamber. For example, nylon softens at about 320° F., whereas Orlon softens at a higher temperature, for instance about 450° F. Saran becomes soft at about 260° F. Accordingly, the speed of travel of the fibers through the apparatus, especially through the gaseous metal plating chamber, is controlled so that at no time do the fibers themselves reach a temperature such as to cause them to soften or deteriorate.

The speed or duration of time in which the fibers are subjected to gaseous metal plating depends upon the fiber being treated. Further, where the time allowed for gaseous metal plating of the fiber is insufficient to deposit the required thickness of metal coating on the fiber, then the fiber is returned for successive plating treatments and passed through multiple gaseous metal plating chambers, as illustrated in the drawings.

Referring to the drawing, and particularly to Figures 1 and 2, there is disclosed an embodiment of the invention wherein the fibers **10** to be plated are drawn from one or more spools **12** and moved first through a pre-heating chamber **14**. This chamber is suitably heated, as by the electrical heating elements **16**, arranged around the walls of the chamber and so as to encompass the fiber drawn therethrough. Heating elements **16** are connected to a source of electric current by leads **18** and **19**. The temperature of the pre-heating is preferably controlled by a rheostat **20** actuated by a thermocouple **21** disposed in the chamber **14** and electrically connected thereto. After the fiber has been drawn through the preliminary heating chamber **14** it is passed into the gaseous decomposition metal plating chamber **24** wherein the same is subjected to gaseous metal deposition. Thereafter the fiber is moved along through the cooling chamber **26** and thence over the guide pulleys **28**, **29**, **31**, to the storage spools **33** and **34**.

To control the speed at which the fibers are drawn through the gaseous metal chamber and associated heating and cooling chambers, a variable speed motor **40** is utilized. This motor is arranged for variable speed operation which may be accomplished by the use of a temperature-actuated rheostat **42**. The speed or R. P. M. of the motor **40**, as thus controlled, is arranged to drive the pulley **44** through the V-belt **45**. Different speeds may also be provided through the use of different diameter driven pulleys operated by the motor **40** if desired. Pulley **44** is, in turn, drivingly connected to the pulleys **46** and **48** by means of the V-belts **50** and **51** respectively.

The temperature and speed control means **42** are connected to the thermocouple **60** which is arranged in the gaseous metal plating chamber, as illustrated in Figures 1 and 2, whereby the speed of the motor may be increased or decreased depending upon the temperature in the gaseous plating chamber. Thus, by setting the temperature control mechanism **42** in accordance with the temperature desired in the gaseous metal plating chamber, the apparatus may be operated so a to maintain the temperature and speed of movement of the fiber through the gaseous chamber substantially constant for any particular setting.

The gaeous metal deposition chamber **24** is suitably heated as by the use of resistance coil **65**, the same being arranged around the chamber so as to evenly heat the same and bring about decomposition of the gaseous metal compound introduced therein. The gaseous metal compound admixed with inert carrier gas, e. g. carbon dioxide, nitrogen, helium or the like, are introduced to the plating chamber **24** through a conduit **68** and the spent gases, including unused metal compound, are withdrawn through a conduit **70** and to a condenser **79**, the gaseous metal compound being restored as a liquid in a tank or container **78** which is connected through conduit **80** to the vaporizer **75** from which it is returned through the conduit **68** to the gaseous metal plating chamber, as described.

The cooling chamber **26** comprises a coil **85** which is arranged in the chamber **87** through which the fiber is drawn after its passage through the gaseous plating chamber. Arranged around the cooling coil **85** is insulating material **89** which may consist of mineral glass wool or the like insulating material. Cooling fluid is admitted to the coil **85** through the conduit **91** and withdrawn therefrom through the conduit **93**.

To prevent seepage of gaseous metal compound from the plating chamber **24** through apertures at the opposite ends, as at **95** and **96**, which permits the entrance and egress of the fibers, inert gas such as carbon dioxide is admitted through the conduits **100** and **102** to the closure chambers **103** and **104** which are arranged about these apertures. This inert gas is maintained at a slightly higher pressure than that inside the gaseous plating chamber, thus preventing the gaseous metal compound from passing out through these apertures.

In the modification illustrated in Figure 5, the apparatus is of similar construction as illustrated and described in Figures 1 and 2, and the same reference characters are applied to designate the same. In this particular modification three gaseous plating chambers **24*a***, **24*b***, and **24*c*** are utilized to effect a triple flash metal plating of the fibers. Where additional gaseous metal treatment

is desired, a plurality of gaseous metal plating chambers may be employed with interposed cooling chambers whereby the process may be carried out to provide multiple-gaseous plating treatment. In this manner, the fibers may be subjected to any desired number of gaseous metal plating treatments to produce metal coating of a predetermined thickness.

The following examples illustrate the process of gaseous metal plating different fibers with various heat decomposable gaseous metal compounds as described.

Example I

Fibers of nylon (polyamide synthetic) were drawn through the apparatus as described, and subjected to a preliminary heat treatment of 250° F. Thereafter the synthetic fibers are drawn through the gaseous metal plating chamber and subjected to a temperature of 375° F. The gaseous metal plating material constitutes nickel carbonyl diluted with carbon dioxide gas, the rate of gas flow being maintained at approximately 4 liters per minute at a temperature of 78° F. and 125 mm. Hg. The fiber in the gaseous metal chamber was drawn through the chamber at a rate of 1 linear foot per second, and such as to expose the fiber to gaseous metal treatment for approximately 3 seconds. Where the plating chamber is of a length of approximately 3 feet, this provides a gaseous plating time of 3 seconds, being insufficient to heat up the fiber to a temperature such as to cause it to soften. Thereafter the gaseous metal treated fiber is drawn through the cooling chamber so as to quickly cool the fiber and prevent it from retaining the residual heat and causing distortion or deterioration thereof. The cooling chamber was maintained at a temperature of approximately 32° F. by circulating brine through the cooling coil.

The process provides nylon fibers having a coating of nickel metal, the thickness of the coating being between about 0.00002–0.00004-inch. Where a greater thickness of the metal coating is desired, the fiber is subjected to repeated gaseous metal plating treatments. For carrying out a plurality of successive gaseous metal treatments, the apparatus illustrated in Figure 5 may be used.

Example II

In this instance Orlon (acrylonitrile synthetic) fiber is coated with iron utilizing iron carbonyl as the heat decomposable gaseous metal compound. The fiber is drawn through the apparatus similarly as described in Example I, being subjected to a preliminary heat treatment of 350° F. The gaseous metal deposition in the plating chamber is conducted in this instance at 475° F. The atmosphere in the plating chamber consists of nitrogen containing about 2% by volume of iron carbonyl. The duration exposure of the fiber in the gaseous metal plating chamber to this temperature is held to approximately 10 seconds and the speed of the fiber was accordingly adjusted so as to provide for this plating time in the gaseous metal plating chamber.

Inasmuch or Orlon has a higher softening temperature, namely about 450° F. as compared with about 320° F. for nylon. Accordingly Orlon fibers may be exposed for a somewhat longer length of time in the plating chamber without injury.

After passing the fibers through the plating chamber and cooling chamber as in Example I, the resultant Orlon fibers are provided with a metal coating of iron having a film thickness approximately 0.00004 to 0.00006-inch.

Example III

In this instance a fabric made of Saran (vinylidine chloride polymer) is nickel coated utilizing nickel carbonyl similarly as in Example I, by passing the same through the heating and gaseous metal plating chamber at a speed such as to complete the exposure of the fiber in the gaseous metal chamber in 5 seconds. The temperature for carrying out the gaseous metal deposition of the nickel being held at approximately 375° F.

In this instance the thickness of the nickel metal coating on the fiber approximates 0.0004–0.00006-inch.

As will be understood, other gaseous metal compounds may be employed, or mixtures thereof, to produce the desired metal coating on the fiber or fabric being treated.

It will be understood that this invention is not to be restricted to the specific fibers mentioned and examples given above, but that it is susceptible to various modifications and changes which come within the spirit and scope of this disclosure and as more particularly set forth in the appended claim.

What is claimed is:

A method of gas plating to metallize organic fibers or filaments selected from the group consisting of polyamide synthetic fiber, acrylonitrile synthetic fiber and vinylidene chloride polymer synthetic fiber, and which tend to soften under prolonged heating at 350 to 475° F., said method comprising the steps of subjecting said fiber to a pre-warming treatment to 250° F. whereby the fiber is heated to a temperature of near its heat-sensitive temperature, then moving the preheated fiber into a metal plating chamber and bringing a blast of inert carrier gas containing a heat-decomposable metal compound into contact with the pre-warmed fiber, subjecting the fiber to further heating to a temperature between 350 and 475° F. and above the temperature at which said fiber tends to soften with prolonged heating and sufficient to cause thermal decomposition of said heat-decomposable metal compound and deposition of the metal constituent thereof onto said fiber, said fiber being subjected to said heated atmosphere during gaseous metal plating for a predetermined short period of time and which time is insufficient to cause softening or deterioration of said fiber but such as to gas plate the fiber with metal, and thereafter quickly cooling the resultant gas plated fiber by moving the same into a chamber maintained at a temperature of about 32° F.

References Cited in the file of this patent

UNITED STATES PATENTS

2,344,138	Drummond	Mar. 14, 1944
2,382,432	McManus et al.	Aug. 14, 1945
2,402,269	Alexander et al.	June 18, 1946
2,516,058	Lander	July 18, 1950
2,602,033	Lander	July 1, 1952
2,616,165	Brennan	Nov. 4, 1952
2,656,283	Fink et al.	Oct. 20, 1953