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3,079,640

HEAT TREATING POLYVINYL ALCOHOL FIBERS
IN A MOLTEN METAL BATH

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No Drawing. Filed May 29, 1958, Ser. No. 738,616
4 Claims. (Cl. 18-48)

The present invention relates to the treatment of textile fibers. More specifically, this invention relates to an improved method for treating textile fibers including the step of treating the fibers in a bath of molten metal.

Molten metal baths find considerable use in the treatment of textile fibers. For example, such baths are useful in the heat treatment of synthetic fibers such as polyvinyl alcohol, nylon, and polyvinyl chloride fibers. Also, molten metal baths have utility in the hot rolling or thermal contraction of thermoplastic textile fibers. Additionally, synthetic and natural fibers such as linen, cotton, wool, viscose rayon, and the like are dyed by techniques involving the use of molten metal baths.

Processes for the treatment of textiles involving passing the textile fibers through a bath of molten metal, especially processes involving heat treatment or hot rolling, have the serious disadvantage that mechanical or chemical damage to the fiber often occurs as a result of contact of the fibers with the molten metal or oxidation products of the metal. The molten metal tends to adhere to the surface of the fiber. Also, the fiber is often stained by contact with the molten metal or with its oxidation products. The adhering metal and stains are extremely difficult to remove from the textile fibers by chemical treatment with acids or other agents or by mechanical cleaning methods without impairing the quality of the textile.

It is an object of the present invention to provide an improved method for treating textile fibers including the step of treating the fibers in a bath of molten metal.

Other objects will be apparent from the following description of the invention.

In accordance with the present invention, textile fibers which are to be treated in a bath of molten metal are first coated with a protective coating agent which is effective to prevent adhesion of the molten metal or oxides thereof to the fibers and which is effective in preventing staining and other damage to the fibers. Subsequently, the fibers containing the protective coating are treated in the bath of molten metal without substantial damage to the fibers.

Protective coating agents which are employed in this invention are compounds which are readily applied to be treated in the metal bath and which are substantially non-reactive with the fibers to be coated. Additionally, the protective coating agents should be substantially non-volatile at the temperature at which the molten metal is maintained. We have found that polyhydric compounds such as ethylene glycol, propylene glycol, and glycerine are useful protective coating agents for practice of the invention. Derivatives of the polyhydric compounds, for example, mono- or polyether derivatives such as ethylene glycol monoethyl ether, or mono- or polyester derivatives such as ethylene glycol monostearate or ethylene glycol dilaurate can be used. Ether esters of the polyhydric compounds can also be used.

In addition to the above, polyglycols have been found to be especially suitable for use in the invention. Derivatives of the polyglycols can be employed. Polyethylene glycols ranging from about 200 to 7500 in molecular weight are commercially available and are especially useful in the invention. Polyethylene glycol derivatives in-

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cluding ester and ether derivatives such as polyethylene glycol 400 monostearate are also useful in the invention. Polypropylene glycols ranging from 150 to 2100 in molecular weight are commercially available and are very useful as protective coating agents in the invention. Derivatives of the polypropylene glycols including ether and ester derivatives can be used. Mixed polyethylene-polypropylene glycols having molecular weights up to 15,000 are available and are useful in the practice of the invention. Polyglycols and derivatives having molecular weights outside the above cited ranges can be used in the invention.

Compounds other than those described above can be used as protective coating agents according to this invention. Saturated aliphatic hydrocarbons such as tetradecane, hexadecane, paraffin wax, light lubricating oil, polyethylenes, polypropylenes, polyisobutylenes, and the like can be used. Aromatic hydrocarbons such as biphenyl can be used.

The protective coating agents are advantageously applied to the textile fibers by applicator rolls prior to treatment of the fibers in the molten metal bath. Alternatively, the fibers may be passed through an aqueous or organic solvent solution of the coating material. Other methods for applying the coating agent such as spraying will be apparent to those skilled in the art and can be used in carrying out this invention.

The amount of protective coating agent employed in the invention can vary. Amounts of coating agent in the range of about 0.2 to 30% based on the dry weight of the fiber are generally suitable with amounts of about 0.5 to 10% being preferred.

After the fiber has been coated with the protective coating agent, the fiber is passed to a molten metal bath wherein it is treated. Any of the commonly known low melting alloys including Wood's metal, Rose's metal, and various solders, which are liquid at the temperature desired for treating the fibers can be employed as the molten metal bath. Illustrative alloy compositions which can be used together with the melting point of each composition are given in the following table.

TABLE 1

Percentage by weight				Melting point, °C.
Bi	Cd	Pb	Sn	
50.0	12.5	25.0	12.5	68
50.1	10.0	26.6	13.3	68
50.0	-----	25.0	25.0	93
40.0	-----	20.0	40.0	100
57.2	-----	17.8	25.0	100

The temperature and duration of the fiber treatment in the metal bath will vary depending upon the fiber and the desired treatment. Generally, lower temperatures such as 95° C. are employed for dyeing treatment while higher temperatures, for example, 230° C. are employed in heat treating fibers such as polyvinyl alcohol fibers. A general range of molten metal bath temperatures is about 80 to 250° C. although temperatures outside this range are sometimes used.

Usually the fiber is treated in the molten metal bath for fairly short times of the order of about 1 to 30 seconds although considerably longer treatment times are sometimes employed.

After treatment of the textile fiber in the molten metal bath, the protective coating can be removed from the fiber by washing with water or by treating the fiber with organic solvents or cleansing solutions. The use of protective coating agents which are soluble in water and

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which therefore can readily be removed from the fiber by washing with water are conveniently and preferably employed in carrying out the present invention. If desired, the coating agents may be recovered and reused by procedures which will be apparent to those skilled in the art.

The following examples illustrate the invention.

Example I

Polyvinyl alcohol fiber, 200 denier/30 filaments, was coated with polyethylene glycol having a molecular weight of 200 through the use of applicator rolls. The fiber was coated with the polyethylene glycol in amount of about 1% by weight of the fiber.

The coated polyvinyl alcohol was then passed through a 1 meter long bath of molten Wood's metal which was maintained at a temperature of 210° C. The fiber travelled at a speed of about 15 meters per minute entering the bath. In the bath the fiber was stretched such that the speed of the fiber leaving the bath was about 75 meters per minute.

After treatment in the molten bath, the polyethylene glycol was readily removed from the fiber by washing with water. The fiber thus obtained was free from adhering metal or metal oxides and evidenced no stains or other damage. The polyethylene glycol had no discernible adverse effect on the fiber.

Example II

Polyvinyl alcohol fiber, 200 denier/30 filaments, was coated with a 10% solution of polyethylene glycol having a molecular weight of about 1000 in benzene through the use of applicator rolls. The fiber was dried and contained a coating of the polyethylene glycol in amount of about 1% by weight of the fiber.

The coated polyvinyl alcohol was then passed through a 2 meter long molten bath of Wood's metal which was maintained at a temperature of about 220° C. The fiber travelled at a speed of about 100 meters per minute entering the bath. In the molten metal bath the fiber underwent shrinkage such that the speed of the fiber leaving the bath was about 85 meters per minute.

After treatment in the molten bath, the polyethylene glycol was readily removed by washing with water. The fiber thus obtained was free from adhering metal or metal oxides and was not stained or otherwise damaged. The polyethylene glycol had no discernible adverse effect on the fiber.

Example III

Acetate rayon, 100 denier/20 filaments was immersed in an aqueous solution containing about 5% of polyethylene glycol monolauryl ether. The rayon was dried and passed through a 1 meter long bath of molten Wood's metal maintained at a temperature of about 130° C. wherein the rayon was heat drawn. Residence time in the molten bath was about 1.5 to 6 seconds.

Upon completion of the heat treatment, the polyethylene glycol was removed from the fiber. No metal or metal oxide adhered to the surface of the rayon.

Example IV

Polyvinyl chloride, 150 denier monofilament, was coated with polypropylene glycol having a molecular weight of about 1000.

The coated polyvinyl chloride was then heat treated for about 1.5 to 6 seconds in a molten bath of Wood's metal which was maintained at a temperature of about 120° C.

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After completion of the heat treatment, the polypropylene glycol was removed from the monofilament. No staining or other damage to the monofilament was observed as a result of the treatment in the molten metal bath.

Example V

Nylon, 100 denier monofilament, was coated with ethylene glycol by immersion in a bath of ethylene glycol.

The coated nylon monofilament was heat treated in a molten bath of Wood's metal which was maintained at a temperature of about 150° C. for about 3 to 8 seconds.

After completion of the heat treatment, the ethylene glycol was removed from the nylon monofilament by washing with water. No adhesion of metal or metal oxides or staining or other damage had resulted from the treatment in the molten metal bath.

Wood's metal which was employed in the above examples has of composition by weight of 50% Bi, 12.5% Cd, 25% Pb, and 12.5% Sn.

We claim:

1. The method of treating polyvinyl alcohol fibers which comprises coating the fibers with a protective coating agent which is effective to prevent adhesion of metal and metal oxide products to the fibers and to prevent staining and other damage to the fibers as a result of heat treatment of said fibers in molten metal maintained at a temperature effective for said heat treatment, said coating agent being non-volatile at the temperature at which the molten metal is maintained and being removable from said polyvinyl alcohol fibers after passage through said bath, and said coating agent consisting essentially of a member selected from the group consisting of polyethylene glycol, polypropylene glycol, and mono alkyl ethers and fatty acid esters thereof, heat treating the coated fibers in a heated bath of molten metal, removing said coating agent from said fibers after said heat treating and subsequently recovering the treated fibers substantially undamaged and free of stains and adhering metal and metal oxidation products.

2. The method according to claim 1 wherein said molten metal bath is maintained at a temperature of about 210° C. to about 250° C.

3. The method according to claim 1 wherein said protective coating agent comprises a polyethylene glycol.

4. The method according to claim 1 wherein said protective coating agent comprises a polypropylene glycol.

References Cited in the file of this patent

UNITED STATES PATENTS

1,856,475	Frost	May 3, 1932
2,125,827	Turkington	Aug. 2, 1938
2,139,640	Mall et al.	Dec. 6, 1938
2,282,568	Finzel	May 12, 1942
2,395,396	Conaway	Feb. 26, 1946
2,400,304	Hamel	May 14, 1946
2,420,720	Pechukas et al.	May 20, 1947
2,474,502	Suchy	June 28, 1949
2,511,472	Kmecik	June 13, 1950
2,518,193	Signaige	Aug. 8, 1950
2,636,803	Cline et al.	Apr. 28, 1953
2,693,995	Hannay	Nov. 9, 1954
2,788,291	Stertz	Apr. 9, 1957
2,886,475	McKay	May 12, 1959

FOREIGN PATENTS

900,758	France	July 9, 1945
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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,079,640

March 5, 1963

Kigen Kawai et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 52, after "to" insert -- the fibers to --.

Signed and sealed this 8th day of October 1963.

(SEAL)

Attest:

ERNEST W. SWIDER
Attesting Officer

EDWIN L. REYNOLDS

Acting Commissioner of Patents

