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3,763,299

DRY SPINNING PROCESS FOR HOLLOW ACETATE FIBER

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5 Claims

ABSTRACT OF THE DISCLOSURE

A method of preparing hollow cellulose acetate filaments by dry spinning an acetone soluble cellulose acetate solution wherein the solvent comprises acetone having carbon disulfide dissolved therein. The solution is spun under conventional dry spinning conditions to produce hollow filaments.

Hollow cellulose acetate filaments have recently gained importance in their application to reverse osmosis procedures for purifying fluids or desalting water.

There are a number of known procedures for preparing such hollow filament bundles. In United States Patent No. 1,652,206, a conventional cellulose acetate solution is dry spun into a hot gaseous medium having a temperature which is substantially higher than the boiling point of the spinning solvent and the rate of flow of the hot gaseous medium in the spinning cell is regulated to produce an immediate superficial evaporation of the solvent at the filament surface.

United States Patent No. 1,904,209 discloses a process wherein a cellulose derivative solution is dry spun into an evaporative medium and solvent removal is caused to take place in two separate stages, the first stage causing surface coagulation only and the second stage causing complete coagulation.

United States Patent No. 2,222,797 discloses a dry spinning process for cellulose acetate wherein the dope is spun into a countercurrent evaporative medium and an additional high temperature current of gas is directed toward the filaments near the point of filament extrusion.

In A.P.C. Ser. No. 362,260, a method and apparatus are disclosed wherein the dry spun dope is first subjected to a strong current of warm gas to solidify the surface of the filament and then to a second hotter zone of gas to evaporate the solvent in the filament interior.

While the above-mentioned prior art disclosures are directed toward the manufacture of hollow acetate filaments, they employ special dry spinning techniques in the spinning cell to accomplish their objectives. Furthermore, in attempts to duplicate some of these procedures, a considerable number of the filaments obtained from a multiple hole jet are not completely hollow.

It is an object of this invention to provide a method for the manufacture of hollow cellulose acetate filaments.

It is a further object of this invention to provide a more efficient method for the manufacture of hollow cellulose acetate filaments by a conventional dry spinning procedure.

These and other objects are accomplished in accordance with this invention by a method of preparing hollow cellulose acetate filaments comprising extruding into a hot gaseous medium through filament-forming orifices, a heated spinning solution of from about 18 to 25% by weight of acetone-soluble cellulose acetate in a solvent mixture of from about 65 to 85% of acetone and from about 35 to 15% of carbon disulfide dissolved in said acetone, said spinning solution having a temperature lower than that which will cause blebbing at the extrusion orifices, and said hot gaseous medium being at a temperature sufficient to form a skin on the freshly extruded

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filaments and vaporize said carbon disulfide within the filament to form a hollow core therein and coagulate the inner surface to prevent collapse of the hollow filament. Small amounts of water are generally present in the spinning solution to improve the solubility of the acetate flake and amounts up to about 5% on the weight of the solution can be tolerated without adverse effects.

The preferred method of this invention involves conventional dry spinning apparatus for the production of cellulose acetate filaments utilizing a spinneret with round holes although other suitable apparatus and hole configurations may be adapted.

Secondary cellulose acetate or acetone soluble cellulose acetate is required for this process on the basis of its spinning properties. The method of preparing cellulose acetate of varying degrees of substitution is well known in the art and need not be particularly set forth herein.

The temperature at which the cellulose acetate spinning solution is maintained will depend on the viscosity of the particular solution and its change in viscosity on heating. Generally, a temperature of from about 50° to less than 80° C. will be employed. Heating of the spinning solution is necessary to reduce its viscosity for spinnability and to facilitate vaporization of the spinning solvent on extrusion. For solutions containing about 18–20% acetate, and a solvent of 3 parts by weight of acetone and 1 part carbon disulfide, the temperature of the solution at the jet or spinneret is advantageously maintained between about 65 and about 75° C. It is preferred that the spinning solution temperature is maintained at just below that at which blebbing occurs. "Blebbing" is that condition wherein the boiling of the solvent from the filament issuing from holes of the spinneret causes the spinning dope to spread over the face of the spinneret and bubble like foam.

The temperature maintained in the spinning cell or tube is such that evaporation of the spinning solvent occurs very shortly after extrusion of the filament whereby an outer skin is formed, the low boiling carbon disulfide in the core vaporizes to form a hollow space and the inner surface of the hollow filament is coagulated in order to prevent the formed inner space from collapsing intermittently along the filament. The temperature maintained in the tube near the spinneret is kept higher, preferably about 10° C. higher than the temperature of the extruded yarn.

The jet orifices from which the spinning solution is extruded preferably range in size from about 2.0 mils to about 5.0 mils in diameter for round hole jets. The yarn is preferably taken up at the bottom of the spinning tube at a speed ranging from about 200 to about 700 meters per minute.

In order to further demonstrate the method of this invention, the following examples are set forth.

EXAMPLE I

A cellulose acetate spinning solution consisting of 19% by weight of secondary cellulose acetate, 1.23% water and 79.77% of a solvent mixture of 3 parts acetone and 1 part carbon disulfide was spun into a heated spinning cell. The temperature of the spinning solution just prior to spinning was 70° C. and it was extruded through a spinneret having 20 round holes each having a diameter of 5.0 mils. The heated spinning cell consisted of an elongated tube having a steam heated jacket to which steam was supplied at 30 p.s.i.g. Hot air was supplied at the bottom of the spinning cell at an inlet temperature of 80° C., and air flow of 25 s.c.f.m. and relative humidity of 10% at 90° F. The spun yarn was taken up at the bottom of the cell at 200 meters per minute. A temperature profile of the spinning cell was made and showed that the air temperature at the spinneret was 80° C. The

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yarn produced had a denier of 500 and the individual filament denier was 25. Most of the filaments were continuously hollow throughout their lengths and the hollow spaces had almost perfectly round cross sections.

EXAMPLE II

A cellulose acetate spinning solution consisting of 19.44% by weight of secondary cellulose acetate, 1.64% water, and 78.92% of a solvent mixture of 3 parts acetone and 1 part carbon disulfide was spun into a heated spinning cell. The temperature of the spinning solution just prior to extrusion was 70° C. and the spinneret through which it was extruded contained 20 round holes each having a diameter of 2.0 mils. The heated spinning cell consisted of an elongated tube having a steam heated jacket to which steam was supplied at 30 p.s.i.g. Hot air was injected at the bottom of the spinning cell at an inlet temperature of 80° C. and an air flow rate of 25 s.c.f.m. and a relative humidity of 10% and 90° F. The spun yarn was taken up at the bottom of the spinning cell at 700 m./m. A temperature profile of the cell showed the air temperature at the spinneret to be 80° C. The yarn produced had a denier of 75 and the individual filament denier was 3.75. At least 75% of the filaments were continuously hollow.

Various changes and modifications may be made in practicing the invention without departing from the spirit and scope thereof and, therefore, the invention is not to be limited except as defined in the appended claims.

I claim:

1. The method of preparing hollow cellulose acetate filaments comprising extruding into a hot gaseous medium through filament-forming orifices a heated spinning solution of from about 18 to 25% by weight of acetone-soluble cellulose acetate in a solvent mixture of from about 65 to 85% of acetone and from about 35 to 15% of carbon disulfide, said spinning solution having a tem-

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perature lower than that which will cause blebbing at the extrusion orifices, and said hot gaseous medium being at a temperature sufficient to form a skin on the freshly extruded filaments and to vaporize said carbon disulfide within the filament to form a hollow core therein and to coagulate the inner surface of the filament.

2. The method of claim 1 wherein the spinning solution has a temperature of about 65 to about 75° C. at the extrusion orifices.

3. The method of claim 2 wherein the temperature of the hot gaseous medium is maintained at about 10° C. higher than the spinning solution at the extrusion orifices.

4. The method of claim 3 wherein the extrusion orifices are round holes having a diameter ranging from about 2 to about 5 mils.

5. The method of claim 4 wherein the yarn is taken up after complete coagulation at a speed of from 200 to 700 meters per minute.

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