Published Apr. 27, 1943

Serial No. 360,776

ALIEN PROPERTY CUSTODIAN

MANUFACTURE OF ARTIFICIAL FILAMENTS AND FIBERS FROM VISCOSE

Heinrich Fink, Rottweil am Neckar, and Gaston Plepp, Wolfen (Kreis Bitterfeld), Germany; vested in the Alien Property Custodian

No Drawing. Application filed October 11, 1940

This invention relates to the manufacture of artificial filaments and fibers of high tensile strength from viscose.

One of its objects is to provide a process for obtaining such threads.

Another object is to manufacture these threads by using novel spinning baths.

Still another object is the new threads obtainable by the new process.

These and other objects will be seen from the following specification.

Processes have recently become known which permit the production of artificial filaments or fibers of high tensile strength from viscose without employing strongly acid baths. Filaments, for example, having a tenacity above two grams per denier have been manufactured by spinning in a spinning bath which contains, if necessary, besides ammonium sulfate and sodium sulfate less than 7% sulfuric acid, whereby the filaments are drawn at least 25% and decomposed to cellulose hydrate. It has also been proposed to swell the filament in an intermediate bath after having left the spinning bath which contains less than 7% sulfuric acid, and then to take up or finish up the drawing operation. As intermediate baths there were used either water or solutions of alkaline reacting substances to which were added neutral substances, inorganic or organic compounds, alcohol, aldehydes, amines, even acids. During or after the swelling operation the filament is hereby drawn 25–100% or more of its original length and is finally decomposed to cellulose hydrate.

There are also described other processes in which a filament from viscose spun in an acid bath is strongly drawn in hot water. In order to increase the effect of the spinning baths and to augment the strength of the filaments, according to another process there were added bivalent salts, for example zinc sulfate, to the baths and the filaments spun in these baths were drawn in hot diluted acids.

Finally one had proposed to manufacture high tenacity artificial fibers and filaments by spinning the viscose into filaments in a spinning bath containing sulfuric acid and salts. After precipitation the filaments leave the precipitating bath and are swollen in an alkaline bath containing zinc or aluminium. The swollen filaments are then drawn in the swelling bath or after the swelling bath in the open air or in hot water or in hot steam.

Our invention is based on the observation that it is possible to obtain high tenacity filaments in a simple way by using ordinary "miller-baths" of more than 7% sulfuric acid, which, however, contain neither zinc nor any other polyvalent metal, also without applying such swelling baths which contain polyvalent metal compounds. These filaments show high tenacities in the wet state their elongation being above 10%.

The new process consists in spinning viscose in a spinning bath containing sodium sulfate and sulfuric acid with a sulfuric acid content of, for example, 10–14%. The freshly spun filaments are then swollen in a hot alkaline swelling bath not containing any polyvalent metal compounds and the swollen filaments are strongly drawn either in the swelling bath or in separate hot salt solutions or in hot steam. The new invention compared with former processes has the advantage that no polyvalent metal compounds are necessary neither in the spinning bath nor in the swelling bath. Furthermore it has the advantage that spinning baths containing sodium sulfate and 10–14% sulfuric acid are not so easily disturbed during operation as baths with only 7% sulfuric acid. Compared with the known processes which contain no polyvalent metal salts in the spinning baths or which employ these swelling baths at ordinary temperature only, the new process yields filaments of better physical constants. The present invention is especially suitable for mass production in the textile industry, since there it is necessary to attain the best effects in a most simple and inexpensive way.

In the manufacturing process an intermediate bath may be placed between the acid spinning bath and the alkaline swelling bath which liberates the filament from the adhering acid and which prevents the alkali from being used up too quickly in the swelling bath. As intermediate bath there may be advantageously employed at temperatures of 40–60° C a water bath containing salts and preferably wetting agents being stable in an alkaline medium. The wetting agents being stable against alkalies are known from literature, for example from the "Textilhilfsmitteltabellen" by Dr. I. Hetzer, 2. edition, Berlin 1938. The swelling bath itself on the other hand is used at temperatures above 70° C, preferably at temperatures above 90° C. It consists of a 10–30% solution of sodium sulfate which contains 0,2–5% caustic soda. In such a swelling bath the freshly spun filament still containing cellulose xanthate groups may be drawn 50–100% of its original length.

During the manufacturing process according to the present invention preferably high grade cellulose having a high α-cellulose content is used. It has proved to be advantageous to employ a viscose which contains a certain amount, slight as it may be, of a most high polymeric cellulose. In this way a fiber is obtained which shows high tenacity and good elongation.

Example I

A viscose of 8% cellulose and 6.5% alkali is spun at a γ-value 45 into a spinning bath containing sulfuric acid from a nozzle of 600 openings each of 0.07 mm. diameter. The spinning bath contains 10% sulfuric acid and 30% sodium sulfate and is kept at a temperature of 45–50° C. The filament is wound up from the nozzle with a speed of 25 meter and is conducted through the spinning bath a distance of 15 cm. After leaving the precipitating bath the filament is conducted a space of 60–80 cm. through a 20% sodium sulfate solution heated up at 50° C and then a space of 80–100 cm. through a swelling bath which contains 1–2% caustic soda and 20% sodium sulfate and is kept at a temperature of 95–100° C. During the passage through the swelling bath the filament is drawn 100% of its original length and finally neutralized under tension in using sulfuric acid of a 5% concentration. The space in the neutralizing bath is again 80–100 cm. The filaments spun in this way have a dry tenacity of 3.3 grams per denier and a wet tenacity of 2.2 grams per denier at an elongation of 12–15%.

Example II

A high grade cellulose containing 96% α-cellulose is worked up into a viscose of 6% cellulose and 8.5% alkali which is spun at a γ-value of 45. The precipitating bath represents an aqueous solution of 12% sulfuric acid and 28% sodium sulfate and is kept at a temperature of 45–50° C. The space in the precipitating bath is 15 cm. long. The artificial filament extruded through a nozzle, 600 openings, 0.07 mm. diameter, is wound up at a speed of 25 meters and is drawn 100% of its original length in a swelling bath heated at 95° C either directly after leaving the precipitating bath or after passing an intermediate bath. The hot swelling bath contains 2% caustic soda and 25% sodium sulfate. The intermediate bath which is put in after the precipitating bath and before the swelling bath consists of a 2% solution of sodium sulfate and is kept at 50° C. Sulfuric acid carried over by the filament into the intermediate bath is neutralized by caustic soda, so that the bath remains neutral all the time. The space of the filament in the washing- and swelling bath is around 80–100 cm. After leaving the swelling bath the filament is neutralized with diluted sulfuric acid. The filaments thus obtained possess a dry tenacity of 3.7 grams per denier and a wet tenacity of 2.6 grams per denier at an alongation of 12–15%.

Example III

Manufacturing process as in Example I with the difference that between the swelling bath and the precipitating bath at 50° C a 20% solution of sodium sulfate is used which contains 2% "Igepal." The filaments thus obtained show a dry tenacity of 3.6 grams per denier and a wet tenacity of 2.4 grams per denier at an elongation of 12–14%.

Example IV

6 parts of a highly-viscous linters-viscose being won from unripened alkali cellulose by sulfidizing with 50% carbon disulfide in a nitrogen atmosphere and being dissolved into a viscose of 5% cellulose and 6.5% alkali (γ-value 60; time of fall of a steel ball 700 sec.) are mixed after ripening for 24 hours at 10° C with 94 parts of an ordinary low viscous viscose from sulfite cellulose with 8% cellulose, 6.5% alkali (γ-value 45; time of fall of a steel ball 25 sec.). The viscosity of the viscose after mixing amounts to 44 sec. of the falling ball. The viscose is spun into a filament at 45° C from a bath consisting of 10% sulfuric acid and 30% sodium sulfate which after passing at 40° C a feebly alkaline sodium sulfate washing bath is drawn 100% at 95–100° C in a bath containing 10–20% sodium sulfate and 1–2% caustic soda and is then neutralized. The filaments show a titre of the single filament of 1.8–2 and a dry tenacity of 3.7–4 grams per denier, a wet tenacity of 2.4–2.6 grams per denier and an elongation of 16–20%.

HEINRICH FINK.
GASTON PLEPP.