April 27, 1937. H. PFANNENSTIEL ET AL 2,078,339

MANUFACTURE OF ARTIFICIAL SILK

Filed May 2, 1932

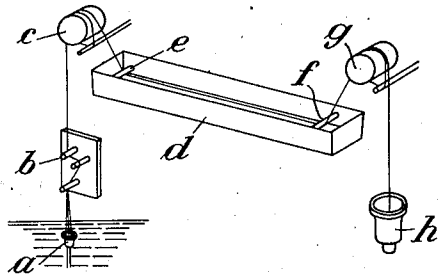

Fig. 1

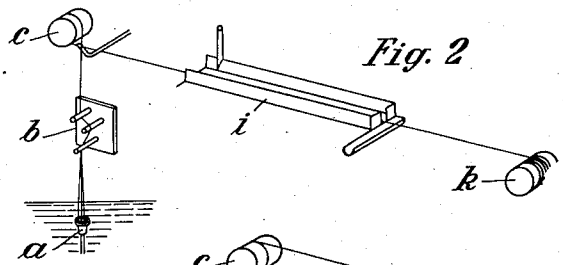

Fig. 2

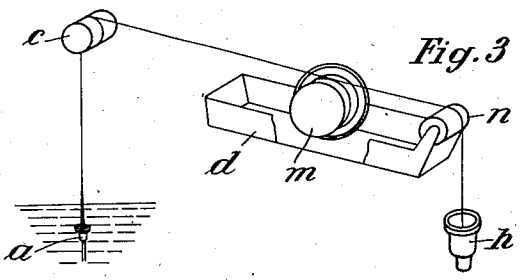

Fig. 3

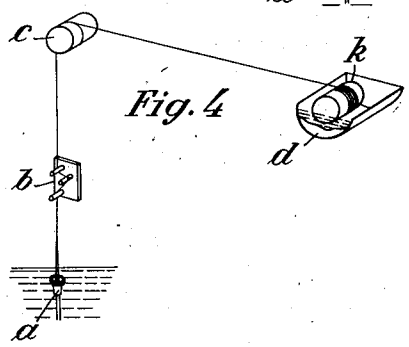

Fig. 4

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Patented Apr. 27, 1937

2,078,339

UNITED STATES PATENT OFFICE

2,078,339

MANUFACTURE OF ARTIFICIAL SILK

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Application May 2, 1932, Serial No. 608,802
In Germany May 2, 1931

2 Claims. (Cl. 18—54)

Our present invention relates to the manufacture of artificial silk and more particularly to the manufacture of artificial silk having a high tensile strength.

One of its objects is to provide a process of producing artificial silk having a high tensile strength and an improved extensibility. Further objects will be seen from the detailed specification following hereafter.

Artificial silk made from viscose by spinning in a bath having a high content of titratable acid, accompanied by stretching, has a low extensibility, in consequence of which the material has a low capacity for being dyed, and other difficulties are experienced in working up the silk. Attempts have therefore been made to impart an increased extensibility to the silk by an after-treatment of the finished material with a shrinking or a swelling agent.

The present invention is based on the observation that the operation of washing threads spun in a strongly acid bath must not be applied to the threads while they are under high tension but must be applied in any suitable manner to the threads while they are being led to the collecting device and while they are capable of contracting to a determined extent. This procedure imparts to the threads a substantially increased extensibility which is conditioned by the shrinkage of the threads. The extent to which the threads can shrink as a result of the washing operation is determined by the reduced tension to which they are subjected in the second zone, and thus remains constant for a given adjustment of the apparatus.

In order to increase the extensibility of the threads it is necessary that they shall be capable of undergoing a longitudinal contraction whilst they are subjected for the first time to the treatment with a washing agent. For example, the threads cannot undergo longitudinal contraction when they are treated with a shrinking agent whilst they are on a roller which serves for stretching them; consequently, such a procedure does not lead to an increase in their extensibility. On the other hand, it is possible to apply the shrinking treatment which is to cause longitudinal contraction, to threads while they lie loosely on a roller; this condition is satisfied if the roller on which the threads undergo treatment with the liquid rotates with a lower peripheral velocity than the preceding roller which delivers the threads to the first-named roller. It is important that in no case must the treatment of the threads with a washing agent while they are capable of contracting be preceded by a like treatment applied to the threads while under high tension.

The problem of increasing the extensibility exists only in the case of viscose silks of high tensile strength that is to say in the case of silks having a tensile strength exceeding 2 grams per denier and produced with the aid of the known sulfuric acid baths containing a high proportion of free sulfuric acid or of sulfuric acid which is semi-bound. The present invention is therefore limited to the use of such spinning baths. If there is used a spinning bath having a smaller concentration of acid, such as is used for the manufacture of threads of normal commercial tensile strength and normal extensibility, the extensibility is not increased, or is increased only to a very limited extent, when the freshly spun threads are allowed to shrink under the conditions used in the present invention.

Water, especially hot water, is a suitable agent for reducing the concentration of acid on the threads in carrying out the invention, for a thread which has been spun in a strongly acid bath shrinks strongly and thus acquires an increased extensibility even when it is brought into water, provided that the thread is not under such a tension as would oppose the shrinkage. The contraction experienced by a thread spun in a strongly acid bath when it is brought, for instance, into hot water, may amount to 15 to 30 per cent. or even up to 50 per cent. of the original length. However, shrinking agents having a stronger action, such as a dilute solution of an alkali or of a hydratizing salt, are also useful. Instead of applying the shrinking agent in the form of a bath through which the thread is conducted, the thread may be sprayed with the shrinking liquid.

The threads may be stretched before being subjected to the action of the shrinking liquid in any known manner. The stretching may be applied by means of rollers or rods, either driven or not, or it may be due to the use of a long bath or a long passage through the air, or by the selection of a high speed of draught, compared with the speed of delivery of the spinning liquid.

In applying the invention it has been found that not every viscose is suitable in like degree for the shrinking process. While, as is known, an unripened viscose is to be preferred for producing threads which can be subjected to a high degree of stretching, a somewhat advanced spinning ripeness of the viscose is advantageous for attaining good shrinkage. Therefore, viscoses having common salt ripeness between 3 and 6 are

preferred for this invention, that is to say viscoses of which one drop into a solution of sodium chloride of 3 to 6 per cent. strength shows coagulation phenomena by the beginning of thread formation.

The accompanying drawing illustrates diagrammatically apparatus for carrying out the invention:

In Fig. 1, *a* is the spinning nozzle submerged in the bath and *b* is the stretching device consisting of glass rods, each out of line with each other. The thread is drawn by a roller *c*, around which the thread passes twice to avoid slipping. The stretched thread is then exposed to the action of a swelling agent in a bath *d*, in which it passes under two guides, such as rods or rollers *e* and *f* arranged in the bath. After shrinkage has occurred, the thread is drawn from the bath by means of the roller *g* at a speed essentially smaller than that at which it is delivered by the roller *c*, it being of importance that the thread lies only lightly against the glass rods or rollers in the shrinking bath. *h* is the spinning box, in which the thread is finally collected.

In Fig. 2 the parts *a*, *b* and *c* are essentially the same as in Fig. 1. From the roller *c* the thread passes through a trough *i* into which the shrinking or swelling agent is caused to act on the thread. The thread leaving the trough is wound on a spool *k* which is driven at constant peripheral speed by means of conoidal gear, so that the thread is drawn in determined degree more gradually from the bath than it is delivered into the bath from the roller *c*.

In Fig. 3 the thread leaving the spinning nozzle *a* is extended by means of the roller *c* and then passes over the roller *m* which has a lower peripheral speed than that of the roller *c*. This roller *m* has its lower part immersed in the shrinking liquid in the bath *d*. After leaving the roller *m* the thread runs over an idle guide roller *n* and then through the spinning funnel into the spinning box *h*.

In Fig. 4 the thread leaving the nozzle *a* is extended by passage over a number of glass rods arranged in staggered relationship and is drawn by the roller *c* around which it is led in one or more turns. During its passage from the roller *c* to a roller *k* the thread is in a condition in which it is capable of shrinking, owing to the fact that the peripheral velocity of the roller *k* on which the thread is wound is lower than that of the roller *c*. The roller *k* rotates in a trough *d* containing the shrinking liquid.

The following examples illustrate the conditions to be observed:

Example 1.—A non-matured alkali cellulose is sulfided with 32 per cent. of CS_2 (calculated on the weight of the cellulose) and dissolved to form a viscose containing 6 per cent. of cellulose and 6.5 per cent. of alkali. The viscose is ripened at 18° C. until it shows a common salt ripeness of 5, and is then spun in a precipitating bath which contains 36.5 per cent. of sulfuric acid monohydrate, 30.5 per cent. of ammonium bisulfate and 12 per cent. of sodium bisulfate. The temperature of the precipitating bath is 43° C. The spinning arrangement is that illustrated in Fig. 2. The thread leaving the precipitating bath is stretched over three glass rods and led one and a half times round a glass roller *c* of 20 cm. diameter; on its way to the spool on which it is to be wound the thread is treated in a water-bath having a temperature of 45° C., the length of immersion being 45 centimeters. It will be seen that the thread is subjected to shrinking while still in an acid moist condition. The peripheral speed of the collecting spool is 25 meters per minute. The viscose pump is adjusted to a total titer of 120 deniers and delivers correspondingly with the collecting speed of 25 meters per minute 5.85 cc. of viscose per minute. The spinning nozzle has 120 perforations, each of 0.07 mm. diameter. The speed of the draught roller is 32 meters per minute, that is to say it is so adjusted that the thread on its way to the collecting spool can shrink by about 22 per cent. The thread being wound on to the spool may advantageously suffer a further sprinkling with water to wash away the acid of the spinning bath. Artificial silk having a tensile strength of 330 to 375 grams per 100 deniers with an extension of 12.5 to 14.5 per cent. may be obtained, whereas without the interposition of the draught roller an extension of only 7 to 8 per cent. with the same tenacity can be obtained.

Example 2.—A viscose containing 5 per cent. of cellulose and 5.5 per cent. of caustic soda is spun at a common salt ripeness of 6 in a bath consisting of 20 parts of methanol and 80 parts of concentrated sulfuric acid at a temperature of 12° C., the other conditions being as indicated in Example 1. There are obtained threads having a tensile strength of 350 grams per 100 deniers and an extensibility of 12.5 per cent.

Example 3.—A viscose prepared as described in Example 1 is spun in a precipitating bath consisting of sulfuric acid of 72 per cent. strength at 12° C., the spinning arrangement being that shown in Fig. 1. The thread is drawn by the draught roller *c* at a speed of 33 meters per minute and is drawn from the shrinking bath by the roller *g* at a speed of 25 meters per minute; it is then collected in the spinning box *h*. The shrinking bath consists of water of 45° C. The thread obtained may have a tenacity of 300 to 400 grams per 100 deniers and an extension of 12 to 18 per cent.

With the rate of supply of the viscose and the nozzles used in the foregoing examples and when the draught is 32 to 33 meters per minute, the threads are first stretched to a finer titer of 94 to 93 deniers; when they are wound at a speed of 25 meters per minute they are allowed to shrink to a final titer of 120 deniers.

What we claim is:

1. A process of spinning artificial silk from viscose which comprises subdividing the spinning operation into two distinct zones; in the first zone squirting viscose through a spinning nozzle into a spinning bath adapted for the production of artificial silk threads having a tensile strength exceeding 2 grams per denier, stretching the freshly precipitated threads until they have a titer finer than that desired for the final thread while avoiding any treatment with a washing agent for reducing the concentration of acid in the thread, and in the second zone subjecting the threads coming from the stretching device to a positive tension lower than that prevailing in the first zone, thus allowing the stretched threads to shrink to the desired final titer and simultaneously promoting the shrinkage of the threads by a treatment with a shrinking agent.

2. A process of spinning artificial silk from viscose which comprises subdividing the spinning operation into two distinct zones; in the first zone squirting viscose having a common salt ripeness of 3 to 6 through a spinning nozzle into a spinning bath adapted for the production of

artificial silk threads having a tensile strength exceeding 2 grams per denier, stretching the freshly precipitated threads until they have a titer finer than that desired for the final thread while avoiding any treatment with a washing agent for reducing the concentration of acid in the thread, and in the second zone subjecting the threads coming from the stretching device to a positive tension lower than that prevailing in the first zone, thus allowing the stretched threads to shrink to the desired final titer and promoting the shrinkage of the threads by washing with water.

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