Aug. 21, 1962 HIIZU AIZAWA ET AL 3,049,755

PROCESS AND APPARATUS FOR STRETCH SPINNING CUPRAMMONIUM RAYON

Filed July 25, 1960 2 Sheets-Sheet 1

FIG. 1

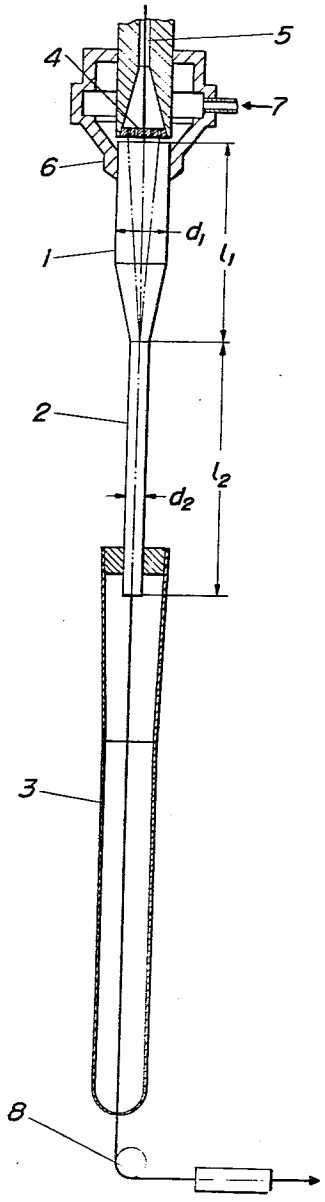
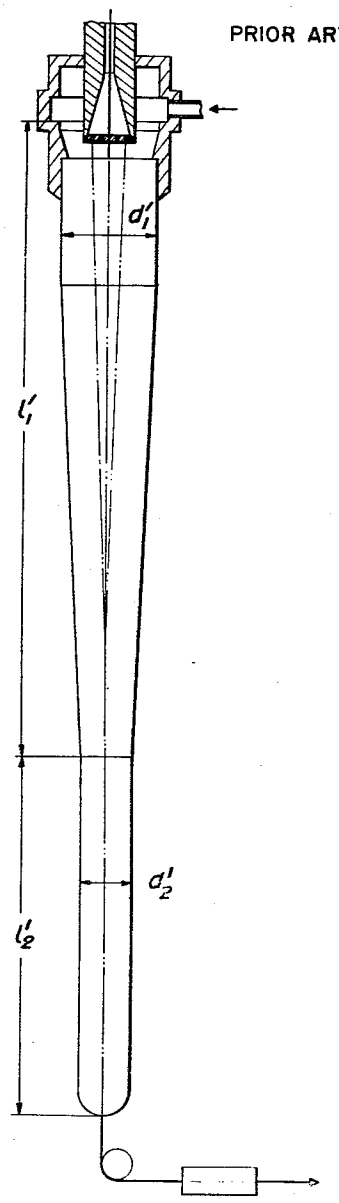


FIG. 3

PRIOR ART



INVENTOR.
HIIZU AIZAWA
FUKUSA ISHIDA
BY
Wenderoth, Lind & Ponack
Attys.

Aug. 21, 1962 HIIZU AIZAWA ET AL 3,049,755
PROCESS AND APPARATUS FOR STRETCH SPINNING
CUPRAMMONIUM RAYON
Filed July 25, 1960 2 Sheets-Sheet 2

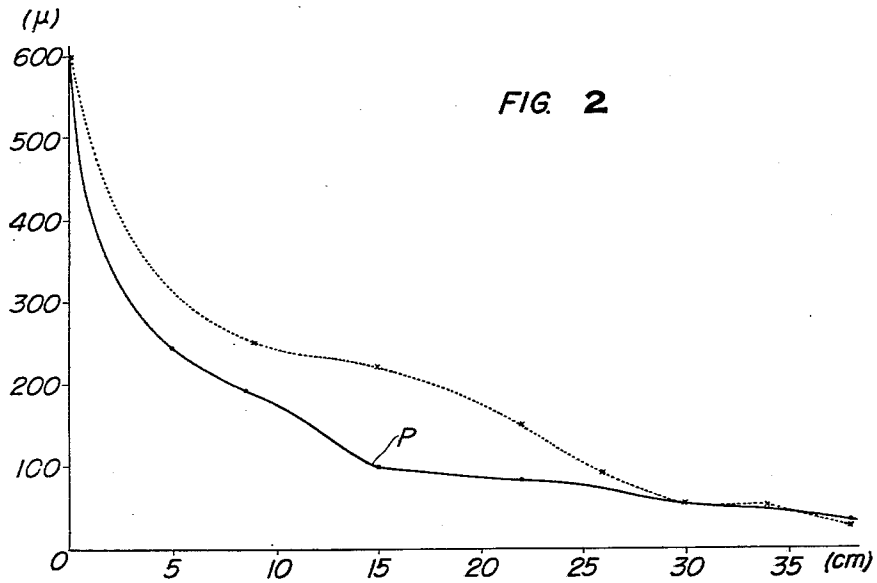


FIG. 2

INVENTOR.
HIIZU AIZAWA
FUKUSA ISHIDA
BY
Wenderoth, Lind + Ponack
Attys.

United States Patent Office 3,049,755 Patented Aug. 21, 1962

3,049,755

PROCESS AND APPARATUS FOR STRETCH SPINNING CUPRAMMONIUM RAYON

Hiizu Aizawa, Nobeoka-shi, and Fukusa Ishida, Hyuga-shi, Japan, assignors to Asahi Kasei Kogyo Kabushiki Kaisha, Osaka, Japan, a corporation of Japan

Filed July 25, 1960, Ser. No. 45,051
Claims priority, application Japan Aug. 3, 1959
3 Claims. (Cl. 18—8)

This invention relates to a stretch-spinning process of cuprammonium rayon and more particularly to an improved method for manufacturing cuprammonium filament yarn by discharging thread forming material in liquid form spinning solution into a stream of coagulating liquid flow.

An object of the present invention is to provide a method for producing cuprammonium rayon which is superior in both tenacity and elongation, particularly in the latter.

Another object of the present invention is to provide a method for producing cuprammonium rayon having homogeneous sectional structure and easy resin finishing character.

The other objects will appear from the following description of an illustrative embodiment of the invention taken together with the accompanying drawings.

FIGURE 1 of the accompanying drawings is a sectional view of an apparatus for producing rayon filament yarn suitable for carrying out the present invention.

FIGURE 2 shows the relation of filament diameter (μ) and path distance from spinneret (cm.), a solid line representing the aforesaid relation according to the present invention and a dotted line representing the same according to a conventional process.

FIGURE 3 shows a typical conventional funnel type apparatus.

In the art as hitherto practised, a cuprammonium spinning solution is conventionally discharged into a rapidly flowing stream coagulating liquid and stretched or drawn due to the flow of the stream while being of partially coagulated form, and the partial coagulate is then further stretched or drawn in the rapid water stream due to drawing from the outside. The last stretching or drawing is effected when and after the spinning solution was substantially coagulated, with the result of increasing tenacity of the final thread but reducing elongation. Such a final thread is so brittle that it is not suitable for resin finishing. This is a major drawback of conventionally produced cuprammonium rayon threads.

According to the present invention, a cuprammonium spinning solution is discharged into a coagulating liquid funnel, and stretched or drawn, immediately after the discharging and while the spinning solution is not yet completely coagulated, but still in suitable state

$$(5\times10^3—25\times10^3 \text{ poise})$$

for strong stretching, by rapid flow of the coagulating liquid whereupon the spinning water is suddenly led into an expanded path thereby greatly reducing the rate of motion of the filament which is formed by coagulation of the spinning solution. In the expanded path, coagulation of the filament is completed due to turbulent diffusion of ammonia while the filament is loose or relaxed, whereafter the completely coagulated filament is drawn out by an external drawing. A main difference between the prior art and the process according to the invention consists in that, after substantial coagulation has been attained, the filament remains for a time, until coagulation is complete, in a slowly flowing stream of coagulating water and is not subjected to further drawing would be the case in the conventional process.

The present invention shall now be described in more detail, having reference to the accompanying drawings. In FIG. 1, reference numeral **6** designates spinneret mounting means, **4** is a spinneret, and **1** is a conical tube located below and distanced from the spinneret **4** to provide sufficient space for introducing coagulating liquid. Spinning solution is fed from an upper feed pipe **5** to means **6** and is discharged through a number of fine holes into the spinning water. The coagulating liquid is fed from an upper inlet **7**, and flows into conical tube **1** together with the spinning solution. The spinning solution and the coagulating liquid flow downward passing through a conical tube zone and on through a straight long tube **2** of smaller diameter, wherein the spinning solution is not yet completely coagulated but sufficiently so that it can be drawn to its maximum possible length by the torrent of the coagulating liquid and that it can be drawn out of tube **2** by the pull applied from the outside reeling or winding device, reference numeral **3** designates a coagulation tube such as an expanded straight tube the diameter of which is greater than that of tube **2**, and the lower end of the tube **2** is inserted into the closed upper end of tube **3**. The torrent of the coagulating liquid in tube **2** enters suddenly the coagulation tube **3** whereby its flowing speed is suddenly and greatly reduced. The filament drawn to the utmost extent is loosed or relaxed and is completely coagulated. The filament in tube **3** is taken up by the aforesaid drawing applied from the outside reeling or winding device without being subjected to further stretching. In the preferred embodiment of the apparatus, the ratio of the diameter of tube **2** to the diameter at the upper end of conical tube **1** is $d_2:d_1$, ½ to ¼, and the length of conical tube **1**, l_1 and the length of straight tube **2**, l_2, may be 4–20 cm. and 3–25 cm. respectively when d_1 is 1.5–3.0 cm. and d_2 is 0.5–1.0 cm. and the diameter of tube 3, d_3 is also preferable to be 1.2–4 times d_2. As will be clear from FIGURE 2, the diameter of the thread is reduced greatly until the point where the spinning solution leaves the lower end of tube **2** and then it is slowly reduced in tube **3** while releasing ammonia, whereby destruction of the cross sectional area of thread is prevented and the thread is provided with uniform sectional structure. In contrast thereto, according to conventional processes, the thread is drawn even in tube **3**, whereby the thread is subjected to severe deformation and is given a heterogeneous sectional structure.

The following examples will serve to understand the present invention, given a comparison wtih a case wherein conventional funnels are used. The spinning solution contained 10% by weight of cellulose, 7.3% by weight of ammonia and 3.65% by weight of copper.

I. SKEIN SPINNING

No.	Conical tube		Straight long tube		Coagulating tube	Titer, denier	Tenacity Dry, gr./den.	Elongation, Dry, percent	Tenacity wet, gr./den.	Elongation, Wet, percent
	l_1 (cm)	d_1 (cm)	l_2 (cm)	d_2 (cm)	d_3 (cm)					
1[1]	24.5	3.5	14.0	1.9	----------	30	1.97	11.4	1.21	15.1
2	4.0	2.0	15.0	0.6	1.9	30	2.52	13.8	1.82	20.2
3	8.0	2.0	10.0	0.75	1.9	30	2.54	15.3	1.86	21.0
4	8.0	1.8	10.0	0.5	1.9	30	2.56	12.9	1.78	18.1
5	8.0	3.0	10.0	0.9	1.9	30	2.30	12.5	1.47	15.8
6	11.0	2.0	10.0	0.6	1.9	30	2.31	14.6	1.68	20.8
7[1]	15.0	1.5	10.0	0.75	1.9	30	2.35	13.6	1.57	19.0
8	24.5	3.5	14.0	1.9	----------	40	1.98	12.9	1.22	17.3
9	4.0	2.0	9.0	0.7	1.9	40	2.46	12.5	1.63	17.0
10	8.0	1.8	10.0	0.5	1.9	40	2.36	14.1	1.67	21.2
11	11.0	2.0	10.0	0.7	1.9	40	2.51	14.2	1.36	20.2
12[1]	24.5	3.5	14.0	1.9	----------	60	1.96	12.1	1.12	15.8
13	4.0	2.0	6.5	0.7	1.9	60	2.27	15.6	1.52	20.6
14	8.0	2.0	6.5	0.7	1.9	60	2.48	16.3	1.58	21.2
15	11.0	2.0	6.5	0.7	1.9	60	2.49	15.3	1.53	20.7

[1] An apparatus shown in FIGURE 3 was used.

II. CONTINUOUS SPINNING

No.	Conical tube		Straight long tube		Coagulating tube	Titer, denier	Tenacity Dry, gr./den.	Elongation, Dry, percent	Tenacity wet, gr./den.	Elongation, Wet, percent
	l_1 (cm)	d_1 (cm)	l_2 (cm)	d_2 (cm)	d_3 (cm)					
1	18	3.0	20	0.7	2.0	50	2.79	11.0	1.77	20.4
2	18	3.0	25	0.7	2.0	50	2.92	9.4	1.72	21.2
3	18	3.0	15	0.8	2.0	50	2.94	9.0	1.52	16.0
4	18	3.0	25	0.8	2.0	50	2.62	7.0	1.55	21.8
5	14	3.0	15	0.8	2.0	50	2.80	8.0	1.61	21.4
6[1]	24.5	5.0	27	2.0	2.0	50	2.54	4.3	1.33	14.4

[1] An apparatus shown in FIGURE 3 was used.

Dotted and solid lines in FIGURE 2 correspond respectively to Nos. 1 and 3 of the above table I.

III. FILAMENT YARN (40 DEN) RESIN FINISHED

	Conventional filament					Present filament				
Resin conc., percent in bath	------	8	12	16	20	------	8	12	16	20
Resin, percent attached	------	3.4	4.5	5.4	6.5	------	3.8	4.6	5.6	6.8
Tenacity:										
Dry, g.	79.0	89.4	90.0	90.2	85.6	98.6	112.0	110.8	119.5	124.6
Wet, g.	47.6	55.4	58.6	62.2	65.8	65.8	72.0	73.8	78.6	86.8
Elongation:										
Dry, percent	12.0	10.0	10.0	8.0	7.4	13.2	10.5	11.1	9.2	9.9
Wet, percent	19.3	16.8	15.5	15.0	13.8	20.0	18.0	17.9	16.7	15.0

We claim:

1. In an apparatus for the stretch spinning of cuprammonium rayon, in combination, a first, descending coagulating and stretching tube means having an upper end adapted for introduction thereinto of cuprammonium spinning solution and a stream of coagulating liquid, for simultaneous flow through said tube means in the same direction, said first tube means in the upper part thereof having a diameter d_1 and a length l_1; and being tapered in the remaining lower part toward the lower end thereof which has a narrower diameter d_2; a second, descending coagulating and stretching tube means connected with its upper end to the lower end of said first tube means, said second tube means having a length l_2 and being substantially of said diameter d_2 throughout said length l_2, and a third coagulating tube means having an enclosed upper end into which the lower end of said second tube means opens unrestrictedly and a further opening at the lower end, said third tube means having a substantially constant diameter d_3; l_1:l_2 ranging from (4 to 20):(3 to 25); l_1:d_1 ranging from 4:1.5 to 20:3.0; l_2:d_2 ranging from 3:0.5 to 25:10; d_1:d_2 ranging from 2:1 to 4:1, and d_3 being substantially larger than d_2, whereby completely coagulated and stretched filament is discharged from said opening at the lower end of said third tube means.

2. A process for stretch-spinning of cuprammonium rayon, which comprises

(*a*) extruding a cuprammonium spinning solution simultaneously with a coagulating liquid into a stretch coagulation zone having an intermediate region wherein the diameter of said zone transverse to the direction of flow of said solution and liquid is substantially narrowed, whereby the flow velocity of said coagulating liquid in the remaining part of said zone, subsequent to said narrowed region, is made greater than the flow velocity of the coagulate being formed by the contact of said cuprammonium solution with said coagulating liquid, and said coagulate is thereby being stretched, and

(*b*) passing said coagulating liquid and coagulate therein, with the same direction of flow and prior to completion of the coagulation, unrestrictedly into a further coagulation zone wherein the flow rate of the coagulating liquid is suddenly diminished, thereby permitting completion of the coagulation of the coagulate while the latter is in a relaxed state.

3. A process for stretch-spinning of cuprammonium rayon, which comprises

(*a*) extruding a cuprammonium spinning solution simultaneously with a coagulating liquid into a stretch coagulation zone having an intermediate region wherein the diameter of said zone transverse to the direction of flow of said solution and liquid is substantially narrowed, whereby the flow velocity of said coagulating liquid in the remaining part of said zone, subsequent to said narrowed region, is made greater than the flow velocity of the coagulate being formed by the contact of said cuprammonium solution with said coagulating liquid, and said coagulate is thereby

being stretched when said coagulate has a viscosity of about 5000 to 25,000 poises, and

(*b*) passing said coagulating liquid and coagulate therein, with the same direction of flow and prior to completion of the coagulation, unrestrictedly into a further coagulation zone wherein the flow rate of the coagulating liquid is suddenly diminished, thereby permitting completion of the coagulation of the coagulate while the latter is in a relaxed state.

References Cited in the file of this patent

UNITED STATES PATENTS

1,590,600 Taylor ---------- June 29, 1926
2,828,497 Hartmann et al. ---------- Oct. 20, 1931