

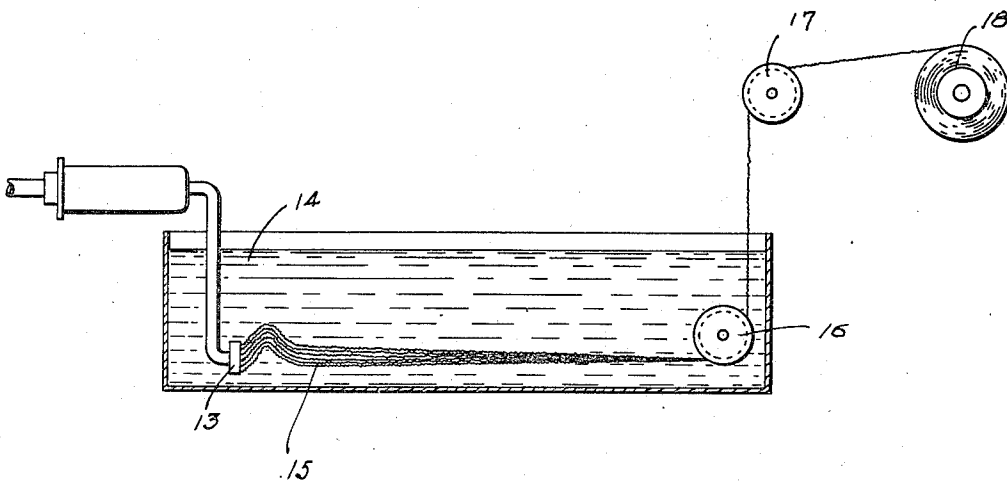
Oct. 6, 1942.

W. H. CHARCH ET AL

2,297,746

PROCESS FOR THE PRODUCTION OF CELLULOSIC STRUCTURES

Filed Oct. 17, 1940



INVENTORS
William Hale Charch
William Frederick Underwood
BY *Louis A. Lieber*
ATTORNEY

UNITED STATES PATENT OFFICE

2,297,746

PROCESS FOR THE PRODUCTION OF
CELLULOSIC STRUCTURES

William Hale Charch and William Frederick
Underwood, Buffalo, N. Y., assignors to E. I.
du Pont de Nemours & Company, Wilmington,
Del., a corporation of Delaware

Application October 17, 1940, Serial No. 361,604

14 Claims. (Cl. 18—54)

This invention relates to improvements in the process of crimp-spinning crimped filaments, threads, and the like from viscose. This application is a continuation, in-part, of our copending application No. 180,976, filed December 21, 1937, now Patent No. 2,249,745.

The above-identified application is directed to new cellulosic filaments which possess inherent and permanent crimps in three dimensions with substantially no molecular orientation in the direction of the fiber axis, said filaments exhibiting good strength, elasticity and other desirable physical characteristics. The above said application is further directed to a new process, hereinafter referred to as a "crimp-spinning" process, for producing such filaments, and yarns composed of such filaments, which comprises forcing fine streams of viscose through the orifices of a spinneret into a coagulating bath which will substantially completely coagulate the filaments before any substantial regeneration thereof; the speed of extrusion, i. e., the jet velocity, being four or more times the rate at which the coagulated filaments are drawn off and removed from the coagulating bath. The filaments, after being removed from the coagulating bath are preferably stretched while in the wet state to improve the character of the crimped filaments.

As set forth in application Serial No. 180,976, the coagulating bath may be a fast coagulating, non-regenerating bath, in which case the filaments will be subjected to regeneration in a second bath. If, on the other hand, the coagulating bath has a slow or delayed regenerating action, the crimped filaments may be spun by a single-bath process.

In the spinning of crimped filaments by the process disclosed in the above said previously filed application, it was found that after spinning a very desirable product for a period of time, the continuation of spinning, even though conducted under exactly the same conditions of spinning, produces a filament with a gradually decreasing amount of crimp. Although this condition is objectionable in a two-bath spinning process, it is particularly objectionable in a single-bath process. In an extreme case in a single-bath spinning process the crimped structure of the spun filaments may disappear in a period of time as short as ten or fifteen minutes. Consequently, heretofore, a two-bath spinning process for the crimp-spinning of filaments has been considered more economical than a single bath process, whereas the reverse should be true.

The above difficulties presented a serious problem to overcome in the crimp-spinning of filaments by the employment of a single coagulating and regenerating bath. In order to solve this problem a very extended search was instituted for a bath composition which would overcome the difficulty. Many different types of spinnerets were investigated with the object of eliminating the said problem. Attempts were made to solve the problem by modifying the viscose spinning solutions, and also by applying an electrical potential to the spinnerets. All of these several attempts, as well as other modifications of the process, failed to eliminate the above said difficulty.

The object and nature of the present invention is to overcome the above mentioned objections in the crimp-spinning of filaments.

It is a further object of this invention to provide an improved crimp-spinning process in which a crimp of uniform character will be imparted to the filaments over an extended period of time.

It is another object of this invention to dissolve, in a crimp-spinning coagulating bath, an agent which will increase the continuity of uniform crimp formation in the filaments produced by a crimp-spinning process.

The present invention will be more clearly understood by reference to the accompanying illustration in which reference numeral 13 designates a filament-forming spinneret submerged in coagulating bath 14. The filaments issuing from the spinneret are passed about guide rollers 16 and 17 and wound onto bobbin 18. The draw-off speed imparted to the filaments 15 by the bobbin does not exceed one-quarter of the jet velocity of the filament-forming composition issuing from the spinneret. As a result, the filaments will be caused to crimp substantially in the manner illustrated.

The objects of the present invention may be accomplished, in general, by crimp-spinning a viscose solution into a coagulating bath with a jet velocity-draw-off ratio of at least four, said coagulating bath having such a character as to substantially completely coagulate the resulting spun filaments before any substantial regeneration thereof, said bath containing, dissolved therein, an agent which will increase the continuity of uniform crimp formation in the resulting spun filaments. Agents suitable for use in accordance with the present invention to increase the period during which uniform crimps are formed in a crimp-spinning process shall

hereinafter be referred to as "crimp-continuity preserving agents."

As "crimp-continuity preserving agents," it has been found that all surface-active agents which are soluble to the extent of at least 0.01%, by weight, in a 5% aqueous sulfuric acid solution are suitable for use in accordance with the present invention. By "surface-active agents" is meant agents which will reduce the surface tension or inter-facial tension of a liquid or liquids. Sulfonic acid salts of aliphatic and aromatic hydrocarbons have been found to be particularly desirable for use as crimp-continuity preserving agents in accordance with the present invention, and are therefore to be preferred over other surface-active agents. All surface-active agents having the above-mentioned solubility characteristics are, however, operative to increase the period during which crimps are formed in filaments in a crimp-spinning process. The surface-active agents may be either cation-active or anion-active. The anion-active materials are preferred largely because of economy, these materials being cheaper, in general, than the known cation-active materials.

The following classes of surface-active agents are found to be very desirable for use as crimp-continuity preserving agents:

Table of surface-active agents which have been found, by test, to be very desirable as crimp-continuity preserving agents. This table indicates the general class of compounds, the trade names of the compounds, together with the name of the manufacturer, and also, as far as possible, the chemical formula of the agent

Formula	Trade name	Manufacturer
CLASS I.—Sulfonated hydrocarbons		
Isopropylated naphthylene sodium sulfonate.	"Alkanol B, HG, SA."	E. I. du Pont.
Tetrahydronaphthylene sodium sulfonate.	"Alkanol S"-----	Do.
Monobutylphenylphenol sodium monosulfonate.		
Dibutylphenylphenol sodium disulfonate.		
Monobutylidiphenyl sodium monosulfonate.		
Sodium abietene sulfonate.		
Alkyl aryl sulfonate.		
	"Invadine B, C, N"	Ciba Co., Inc.
	"Merpentine"-----	E. I. du Pont.
	"Naccolene F"-----	Natl. Aniline & Chem. Co.
	"Nacconol E"-----	Do.
	"Nacconol NR"-----	Do.
	"Nacconol NRL"-----	Do.
	"Nacconol NRSE"-----	Do.
	"Nacconol A"-----	Do.
	"Nekal A, BX"-----	General Dyestuffs Corp.
	"Neomerpin VD"-----	E. I. du Pont.
	"Novonacco"-----	Natl. Aniline & Chem. Co.
	"Novonacco NN"-----	Do.
	"Santomerse"-----	Monsanto Chemical Co.
	"Solvadine NC"-----	Ciba Co., Inc.
	"Ultravon, K, W"-----	Do.
Sulfonated hydrocarbon	"Sulfatate"-----	Glycol Products Co.
CLASS II.—Alcohol sulfate, having 8 or more carbon atoms		
Octyl sodium sulfate.	"Gardinol LS"-----	E. I. du Pont.
Octadecenyl sodium sulfate.	"Gardinol WA"-----	Do.
Dodecyl sodium sulfate	"Drene"-----	Proctor & Gamble.
Fatty alcohol sulfates	"Duponol L-144, WD, LS, ME, OS, WA, T, WS, 80,"	E. I. du Pont.
	"Merpel B, C"-----	Do.
	"Modinal T"-----	Do.
CLASS III.—Cation-active materials		
Cetyl pyridinium bromide.		
Lauryltrimethylammonium bromide (and those compounds described in Polak U. S. Patent 2,125,031).		
Quaternary ammonium salts.	"Triton S-18"-----	Rohm & Haas Co.
The fatty acid salt of trimethylolamino methane.	"Trimethylol aminomethane."	Commercial Solvents Corp.
CLASS IV.—Modified sulfated fatty acid esters		
Oleic acid ester of an aliphatic compound.	"Igepon AP Extra."	General Dyestuffs Corp.
CLASS V.—Derivatives of fatty acid amides		
Fatty acid amides	"Igepon T"-----	General Dyestuffs Corp.
	"Intrammine"-----	Synthetic Chemicals.
	"Sapamine A"-----	Ciba Co., Inc.
CLASS VI.—Sulfonated ethers		
Sulfonated ethers	"Tensol"-----	Synthetic Chem., Inc.
	"Triton W-30"-----	Rohm & Haas Co.
	"Triton 720, 812"-----	Do.
CLASS VII.—Miscellaneous and unclassifiable.		
	"Aresket"-----	Monsanto Chemical Co.
	"Aresklene"-----	Do.
	"Neopen SS"-----	E. I. du Pont.
	"Aerosol OS"-----	American Cyanamide and Chemical Corp.
	"Daconol"-----	S. A. Day Mfg. Co., Inc.
	"Darvan"-----	R. T. Vanderbilt Co., Inc.
	"Daxad"-----	Dewey & Almy Chem. Co.
	"N. S. A. E."-----	Onyx Oil & Chem. Co.
	"Wetsit"-----	Jacques Wolf & U Co.
	"Arylene"-----	Hart Products Corp.
	"Ultra Wet"-----	Atlantic Refining Co.
	"Nopco 1967"-----	Natl. Oil Products Co.
	"Duponol 80"-----	E. I. du Pont.
	"Avirol 80"-----	Do.
	"Cominol"-----	Commonwealth Color & Chemical Co.
	"Detanol"-----	E. I. du Pont.
	"Lanaclarin LM"-----	Commonwealth Color & Chemical Co.
	"Lightning Penetrator X"-----	Onyx Oil & Chem. Co.
	"Mapromin"-----	Proctor & Gamble.
	"Orvus WA"-----	Commonwealth Color & Chemical Co.
	"Soapatol"-----	E. I. du Pont.
	"Fixanol"-----	Do.
	"Product QB"-----	Stanco, Inc.
	"Dipex"-----	A. C. Horn Co.
	"Hornkem 1 and 3"-----	Do.
	"Penetrex"-----	Do.
	"Oleo Glyceryl Sulfate C"-----	Onyx Oil & Chem. Co.
	"Oleo Glyceryl Sulfate O"-----	Do.
	"Onyxol 66"-----	Do.
	"Triton M-25"-----	Rohm & Haas Co.
	"Maprofix"-----	Onyx Oil & Chem. Co.
	"Mapromol"-----	Do.
	"Tergitol Penetrant OS"-----	Carbide & Carbon Chem. Do.
	"Tergitol Penetrant 4, 7"-----	Do.

Admittedly, surface-active agents, such as above disclosed, have previously been added to coagulating baths in conventional viscose spinning processes. Heretofore, however, such ma-

terials have been added to coagulating baths in conventional spinning processes for the spinning of straight filaments. Such surface-active materials have previously been added to coagulating baths for the purpose of obtaining improved spinning of straight filaments, for example, to obtain a faster coagulation of the viscose in the coagulating bath, and for the reduction or elimination of spinneret cratering, or the elimination of clogging of spinneret openings. Spinneret cratering or spinneret clogging, if permitted to exist, will result in broken filaments or the production of small, under-size filaments. Yarns produced under these circumstances are decidedly inferior. In the crimp-spinning of crimp filaments, however, the gradual discontinuation of the formation of crimps in the filaments is not due to slow coagulation of the viscose, or to clogged spinneret openings, or to craters. The problems involved in the crimp-spinning process above described do not result in the breaking of filaments but involve merely a discontinuation of crimp formation. These latter processes are therefore not concerned with the usual factors of spinning straight filaments and therefore it could not be foreseen that the addition of surface-active agents to a coagulating bath of a crimp-spinning process would preserve the continuity of crimp formation in the filaments.

The quantity of surface-active agents to be incorporated in the coagulating bath of a crimp-spinning process is relatively small, based on the total bath ingredients, being of the order of 0.05% up to 0.5%, while larger quantities may be employed if the surface-active agent is sufficiently soluble.

Although the surface-active agents used in accordance with the present invention as crimp-continuity preserving agents will decrease the measured surface tension of the coagulating bath, it is not known what part, if any, the lowering of surface tension may play in preserving the continuity of crimp formation in the spinning process. There is undoubtedly more to the phenomena of preserving the crimp-continuity of spinning than the mere lowering of surface tension. Attention is directed to the fact that in order to impart maximum preservation of crimp-continuity it is necessary to use a quantity of surface-active agent which is distinctly in excess of that quantity merely required to give a maximum lowering of the surface tension of the bath. For example, 0.01% of a certain surface-active material is known to give maximum lowering of the surface tension in a coagulating bath. In order to achieve the desired preservation of crimp-continuity, it was found necessary to employ 5 to 10 times the quantity of a surface-active agent known to give a maximum of the lowering of the surface tension. That is to say, it is necessary to use from 0.05% to 0.1% of the surface-active agent. Generally, quantities of the surface-active agents in excess of 0.01% will effect substantially no further lowering of the surface tension in a coagulating bath, whereas quantities considerably in excess of that amount profoundly affect the length of time in which the spinning process will function to uniformly spin a crimp filament. It has been found, in all cases, that the surface-active agent, for the purposes of the present invention, must be used distinctly in excess of that required to give the maximum lowering of surface tension of the bath.

Attention is further directed to the fact that there is no direct correlation between the extent

of lowering of the surface tension and improvement in the spinning of crimp filaments to be realized by the use of specific surface-active agents. For example, it has been observed that although octyl sodium sulfate will lower the surface tension to nearly 30 dynes per square centimeter as compared to "Nacconol NR," which lowers the surface tension to only 40 dynes per square centimeter, the latter material is superior to the former in preserving the continuity of spinning of crimp filaments. The manner in which surface-active agents function to preserve continuity of spinning of crimp filaments is therefore completely unknown.

It has furthermore been found that the surface-active agents will function to preserve the continuity of crimp formation in filaments to a greater extent if the coagulating bath contains an appreciable quantity of hydrogen sulfide. The presence of at least 28 parts per million by weight of hydrogen sulfide in the bath will not in itself preserve the continuity of crimp formation, but will improve the results to be obtained when using a surface-active agent for this purpose.

As above indicated, a coagulating or setting bath in which crimp filaments of good strength are formed by spinning with a high jet velocity-draw-off ratio must be a fast coagulating bath with no regenerating action, or, alternatively, one with a slow or delayed regenerating action. The preferred coagulating baths, employed in accordance with the present invention, are substantially the same as those described in our copending application above referred to, with the exception that a crimp-continuity preserving agent is added thereto. A fast coagulating non-regenerating bath will comprise a high concentration of a highly soluble salt or a high concentration of a weak acid containing such a small amount of water as to give a strong dehydrating or precipitating action. An aqueous, saturated, or nearly saturated ammonium sulfate bath at approximately 45° C. constitutes an excellent coagulating bath for the present process. Other highly soluble ammonium salts can also be used in high concentrations as well as other coagulating salt solutions such as a saturated sodium sulfate solution.

In a single setting bath spinning process, with which the present invention is most concerned, the setting bath should be slightly acid in character so as to have a slow or delayed regenerating effect on the viscose extruded thereinto. A preferred setting bath for such a process contains at least 5%-10% or more of ammonium sulfate together with sufficient sodium sulfate to make the total salt content of the bath 15%, or greater, of the total bath ingredients including the water. The total salt content of the bath should be at least 2-3 times that of the total acid employed. The total salt content must be higher with high acid content and may be lower with low acid content.

As a general rule, baths which operate to produce filaments which are substantially smooth and non-crenulated, as opposed to the highly crenulated filaments produced by baths conventionally employed in the spinning of straight filament viscose rayon, are suitable for use in accordance with the present invention. The production of substantially smooth and non-crenulated filaments in a bath is a definite indication that the bath is functioning to substantially complete the coagulation of the filaments before any substantial regeneration thereof.

Similar to the process defined in the above

mentioned copending application, it is preferred that the crimpy filaments be subjected to a stretching treatment after the coagulation thereof and while they are still in the wet state. This stretching treatment may be carried out before or after the regeneration of the filaments, preferably after a partial regeneration thereof. The stretching also may be carried out with satisfactory results after coagulating, after washing, or even after drying and then rewetting the yarn. The stretching treatment is followed by a relaxing of the filaments.

The following specific examples are given to illustrate the details of spinning permanently crimpy filaments in accordance with the present invention. It is to be understood that the invention is not to be limited to the specific details set forth in these examples.

Example I

A viscose made from wood pulp containing 7% cellulose and 4% sodium hydroxide at a sodium chloride index of 4.0 was extruded through noble metal alloy spinneret having 100 holes, each hole being 0.002 inch in diameter, into a spinning bath having a composition of 3.38% sulfuric acid, 15.35% ammonium sulfate, 7.85% sodium sulfate, and 0.20% isopropyl naphthalene sodium sulfonate ("Alkanol B"). The temperature of the bath was maintained at about 47° C. The viscose delivery was 20.25 grams per minute and the crimpy yarn formed spontaneously at the spinneret was drawn away from the spinneret at the rate of 504 inches per minute after 48 inches of travel in the bath and was then stretched 75% (based on the straight length of the filaments) between rubber nip rolls followed by relaxation under no tension. After one hour time lag to insure complete regeneration, the crimpy yarn was then washed free from acid and then rinsed with approximately 0.25% sodium carbonate solution at 95° C. in order to remove free sulfur, followed by a wash with soft water to remove residual alkali. The purified yarn was then finished with approximately 0.5% aqueous sulfonated olive oil solution at 60° C. followed by drying in a relaxed condition with the filaments free to shrink.

The yarn possessed a good permanent crimp, was strong and had a smooth or substantially non-crenulated cross section with many of the filaments substantially round.

In this experiment, the ratio of nozzle velocity to the rate at which the thread was drawn away from the spinneret was approximately 7.2. The "Alkanol B" required in the above bath, to attain maximum lowering of the surface tension, is approximately 0.003%, while it was found essential to satisfactorily prolong crimp-spinning to use approximately 0.20% "Alkanol B."

Example II

A viscose prepared from wood pulp containing 7% cellulose and 6% sodium hydroxide at a sodium chloride index of 4.0 was extruded into spinning bath having a composition of 4.69% sulfuric acid, 11.22% ammonium sulfate, 8.8% sodium sulfate, and 0.20% "Alkanol B" at 55° C. Spinning nozzles made from noble metal alloys, having 70 holes, each hole being 0.002 inch in diameter, were used in this example with a viscose delivery of 19.04 grams per minute. The crimpy yarn was formed spontaneously at the spinneret and was drawn away from the spinneret after 48 inches travel in the bath at the

rate of 456 inches per minute, and then stretched 50% between rubber nip rolls, followed by relaxation under no tension. The yarn, after one hour time lag to insure complete regeneration, was washed free from acid, using soft water. The acid free yarn was then rinsed with 0.25% sodium carbonate solution at 95° C. to remove free sulfur and then washed free of alkali, using soft water. The purified yarn was then finished with an aqueous dispersion of 0.5 gram per liter of sulfonated olive oil at 60° C. After removal of excessive liquor in a centrifuge it was then dried in a relaxed condition in order to permit shrinkage of the individual filaments. Using the above spinning conditions, the ratio of nozzle velocity of the viscose to the rate of yarn drawn away from the spinneret was approximately 10.4. In the above spinning bath, the amount of "Alkanol B" required to obtain maximum lowering of the surface tension was approximately 0.0025% while the amount required to obtain a satisfactorily increased crimp-spinning was approximately 0.20%. The resulting yarn possessed a substantially smooth non-crenulated cross section, possessed a good permanent crimp and was strong.

Using the above spinning conditions, it was possible to spin filaments having a satisfactory, uniform crimp for a period of 8 hours.

Example III

Viscose made from cotton linters containing 7% cellulose and 6% sodium hydroxide at a salt index of 3.9 was extruded through noble metal alloy spinnerets having 100 holes, each hole being 0.0025" in diameter, into a spinning bath having a composition of 2% sulfuric acid, 11% ammonium sulfate, 11% sodium sulfate, and 0.20% lauryl trimethyl ammonium bromide at a spinning bath temperature of 43° C. The viscose was extruded through the above spinneret at the rate of 26.9 grams per minute, which represents a nozzle velocity of approximately 3010 inches per minute. The crimpy yarn formed spontaneously at the spinneret was drawn away from the spinneret at the rate of 324 inches per minute through 40 inches of bath travel and then immediately stretched 50% between rubber nip rolls, followed by relaxing under no tension. After one hour time lag to insure complete regeneration of the above filaments they were washed free from acid, desulfured, washed free from alkali, finished and dried in the same manner as in Example II above. The yarn possessed a good permanent crimp and a substantially smooth and non-crenulated cross section. Using the above surface-active material in the spinning bath, it was possible to spin 6 hours or more before failure of formation of satisfactorily crimped filaments, while without the use of the above material, after spinning for from 3 to 10 minutes, satisfactory crimps were no longer formed in the filaments. The ratio of nozzle velocity to the rate at which the yarn was drawn away from the spinneret was approximately 9.29. The amount of surface-active material required to obtain maximum lowering of the surface tension was approximately 0.003% while it was found necessary to use approximately 0.20% of the above material in order to satisfactorily prolong crimp-spinning.

Example IV

Viscose made from wood pulp having the composition 7% cellulose and 6% sodium hydroxide

at a sodium chloride index of 4.0 was extruded at the rate of 140 grams per minute through noble metal alloy spinnerets having 400 holes, each hole being 0.003" in diameter, into a spinning bath having the composition 8.44% H_2SO_4 , 37.65% ammonium sulfate, 2.5% sodium sulfate, and 0.1% "Nacconol NR," at 45° C. The crimpy filaments, which formed spontaneously at the spinneret, were drawn away from the spinneret through 24 inches of bath at the rate of 414 inches per minute, and immediately stretched 59.5% between rubber nip rollers followed by relaxation. After a 3 minute time lag to insure complete regeneration of the filaments, the crimpy yarn was purified, finished and dried in the same manner as the above examples. The resulting dry yarn possessed a good permanent crimp, was strong and had a smooth substantially non-crenulated cross section. The nozzle velocity in this spinning experiment was approximately 2910 inches per minute; hence the ratio of nozzle velocity to the rate at which the filaments were drawn away from the spinneret was approximately 7.03. The amount of "Nacconol NR" in the above bath required to obtain maximum lowering of the surface tension was approximately 0.01% while we found it necessary to add approximately 0.10% in the above spinning both in order to satisfactorily maintain crimp-spinning conditions.

Example V

Viscose made from wood pulp containing 7% cellulose and 4% sodium hydroxide at a sodium chloride index of 4.0 was extruded through noble metal alloy spinnerets having 100 holes, each hole being 0.0025" in diameter, at the rate of 30.6 grams per minute into a bath having the composition 3.42% H_2SO_4 , sufficient sodium sulfate to produce a saturated solution, and 0.2% "Nacconol NR" at 45° C. The crimpy filaments formed spontaneously at the spinneret were drawn away from the spinneret through 60 inches of bath at the rate of 300 inches per minute, and immediately stretched 50% between rubber nip rolls followed by relaxing. After a one hour time lag to insure complete regeneration of the yarn, it was purified, finished and dried in the same manner as in the above example. The dry yarn was strong, well crimped and had a substantially smooth and non-crenulated cross section. The ratio of the rate of extrusion of the viscose to the rate at which the crimpy filaments were drawn away from the spinneret was approximately 11.3. The amount of "Nacconol NR" required to obtain maximum lowering of the surface tension in the above spinning bath is approximately 0.02%, while it was found necessary to have approximately 0.2% "Nacconol NR" present to satisfactorily maintain crimp-spinning conditions.

Crimp-spinning processes, that is, processes in which a crimp is directly spun into the filaments by a high jet velocity-draw-off ratio with a fast coagulating, slow regenerating, or non-regenerating bath, can be made to spin crimpy filaments for a longer continuous period of time by including in the coagulating bath a crimp-continuity preserving agent in accordance with the present invention. The preservation of crimp-formation in the filaments is sufficiently prolonged that single-bath crimp-spinning processes can now be employed in plant production more economically than the two-bath processes as used prior to the present invention.

Since it is obvious that many changes and modifications can be made in the details set forth above without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to these details except as set forth in the following claims.

We claim:

1. The process of crimp-spinning crimpy filaments which comprises extruding viscose, in the form of filaments, into a setting bath which has a sufficiently rapid coagulating action, relative to any regenerating action thereof, to completely coagulate said filaments prior to any substantial regeneration thereof, and drawing said filaments from the point where they are extruded into the bath, the velocity of extrusion being at least 4 times the velocity of the draw-off, said coagulating bath containing dissolved therein, as a crimp-continuity preserving agent, at least 0.05% of surface-active material having a solubility of at least 0.01% by weight in a 5% aqueous solution of sulfuric acid whereby to increase the continuity of uniform crimp formation in the spun filaments.
2. The process of crimp-spinning crimpy filaments as defined in claim 1, in which the setting bath has a coagulating and regenerating action.
3. The process of crimp-spinning crimpy filaments as defined in claim 1, in which the setting bath has a coagulating, non-regenerating action.
4. The process of crimp-spinning crimpy filaments which comprises extruding viscose, in the form of filaments, into a setting bath which has a sufficiently rapid coagulating action, relative to any regenerating action thereof, to completely coagulate said filaments prior to any substantial regeneration thereof, drawing said filaments from the point where they are extruded into the bath, the velocity of extrusion being at least 4 times the velocity of draw-off, said coagulating bath containing dissolved therein, as a crimp-continuity preserving agent, at least 0.05% of a surface-active material having a solubility of at least 0.01% by weight in a 5% aqueous solution of sulfuric acid whereby to increase the continuity of uniform crimp formation in the spun filaments, and stretching said coagulated filaments in the wet state.
5. The process of crimp-spinning crimpy filaments as defined in claim 4, in which the setting bath has a coagulating and regenerating action.
6. The process of crimp-spinning crimpy filaments which comprises extruding viscose in the form of filaments into a setting bath which has a sufficiently rapid coagulating action, relative to any regenerating action thereof, to completely coagulate said filaments prior to any substantial regeneration thereof, drawing said filaments from the point where they are extruded into the bath, the velocity of the extrusion being at least 4 times the velocity of draw-off, said coagulating bath containing dissolved therein, as a crimp-continuity preserving agent, at least 0.05% of a surface-active material having a solubility of at least 0.10% by weight in a 5% aqueous solution of sulfuric acid whereby to increase the continuity of uniform crimp formation in the spun filaments, regenerating said filaments, and stretching the same while they are in the wet state.
7. The process of crimp-spinning crimpy filaments as defined in claim 6, in which the setting bath has a coagulating and regenerating action.
8. The process of crimp-spinning crimpy filaments as defined in claim 1, in which the surface-active material comprises a sulfonic acid salt.

9. The process of crimp-spinning crimpy filaments as defined in claim 1, in which the coagulating bath contains at least 28 parts per million, by weight, of hydrogen sulfide.

10. The process of crimp-spinning crimpy filaments which comprises extruding viscose, in the form of filaments, into a setting bath which has a sufficiently rapid coagulating action, relative to any regenerating action thereof, to completely coagulate said filaments prior to any substantial regeneration thereof, drawing said filaments from the point where they are extruded into the bath, the velocity of extrusion being at least 4 times the velocity of draw-off, stretching said filaments while they are still wet with said coagulating bath liquid, and relaxing said filaments in the absence of tension, said coagulating bath containing dissolved therein, as a crimp-continuity preserving agent, at least 0.05% of a surface-active material having a solubility of at least 0.01% by weight in a 5% aqueous solution of sulfuric acid whereby to increase the continuity of uniform crimp formation in the spun filaments.

11. The process of crimp-spinning crimpy filaments as defined in claim 10, in which the setting bath has a coagulating and regenerating action.

12. The process of crimp-spinning crimpy filaments as defined in claim 10, in which the sur-

face-active material comprises a sulfonic acid salt.

13. The process of crimp-spinning crimpy filaments which comprises extruding viscose, in the form of filaments, into a coagulating and regenerating bath which has a sufficiently rapid coagulating action, relative to the regenerating action thereof, to completely coagulate said filaments prior to any substantial regeneration thereof, drawing said filaments from the point where they are extruded into the bath, the velocity of extrusion being at least 4 times the velocity of draw-off, stretching said filaments while still wet with coagulating bath liquid, relaxing said filaments in the absence of tension for a sufficient period of time to insure complete regeneration by the coagulating bath liquid contained therein, said coagulating bath containing dissolved therein, as a crimp-continuity preserving agent, at least 0.05% of a surface-active material having a solubility of at least 0.01% by weight in a 5% aqueous solution of sulfuric acid whereby to increase the continuity of uniform crimp formation in the spun filaments.

14. The process of crimp-spinning crimpy filaments as defined in claim 13, in which the surface-active material comprises a sulfonic acid salt.

WILLIAM HALE CHARCH.

WILLIAM FREDERICK UNDERWOOD.