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2,805,464

FIBER TREATMENT

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No Drawing. Application January 18, 1955,
Serial No. 482,654

6 Claims. (Cl. 28—76)

This invention relates to a process for improving the loft or bulk of acrylonitrile copolymer yarns without substantially lessening the desirable hand of the yarns and fabrics made therefrom.

Crimped staple fibers produced from dry spun acrylonitrile homopolymer tow may be converted into spun yarn and fabricated into very satisfactory knitwear. However, crimped staple fiber prepared from acrylonitrile/methyl acrylate copolymers, such as copolymers comprised of 85% to 96% acrylonitrile and 4% to 15% methylacrylate, when processed in like manner, results in hungry, undesirable knitwear due to a very substantial loss in bulk during processing. By comparative measurements, this loss was found to be as much as 25%, which meant that heavier than normal yarns had to be made and knitted with an extremely tight stitch, resulting in substantially greater cost per knit piece.

Many attempts have been made to retain the initial bulk or crimp in the fibers, but invariably methods that produce good crimp retention and therefore satisfactory loft or bulk in the knit goods produced therefrom resulted in an undesirable harsh handle or an undesirable soft, limp handle that rendered the goods unacceptable. Processes previously developed for other hydrophobic synthetic fibers, such as dry heat setting, were not applicable to the processing of acrylonitrile/methyl acrylate copolymer yarns, filaments or staple without encountering unacceptable harshness or unacceptable soft, limp handle. This is true also for hot water or steam treatments, even though these, like the dry heat setting process, involve relaxing.

It is, therefore, an object of this invention to provide an improved process for after-treating fibers of acrylonitrile copolymers that have been drawn and crimped to render the crimp more permanent. Another object is the provision of such a process in which the desirable handle originally inherent in the drawn crimped fibers is retained. Other objects will be apparent from the description that follows.

The objects of this invention are accomplished by subjecting drawn, crimped fibers of acrylonitrile copolymer derived from at least about 85% acrylonitrile, while they are in a relaxed state, to dry heat, the temperature being from about 300° F. to about 350° F., until the fibers attain a temperature of at least about 300° F. and then treating the resultant, heat-relaxed fibers with hot water, in liquid or vapor form, while the fibers are in a relaxed or free-to-relax or no more than a partially restrained state, the temperature of the water being from about 212° F. to about 280° F. The duration of the treatments in each case is usually several minutes, for example generally about ten minutes. As examples are copolymers containing at least 85% acrylonitrile and from 4% to 15% methyl acrylate in the polymer molecule the objectives are readily attained using such polymers by subjecting them first to dry heat relaxing within a temperature range of from 300° F. to 350° F. and preferably at

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a temperature of about 320° F. until the fiber itself attains a temperature of at least about 300° F. and thereafter treating the heat-relaxed fibers with hot water within the temperature range of 212° F. to 240° F. for a period of about ten minutes. The hot water treatment may be applied to filaments which are completely loose and free-to-relax and to semi-restrained filaments or fibers as in a stock-dye kettle wherein the fibers tend to pack and mat together. Thus, the fibers may be in the form of continuous filaments, freely relaxed staple or partially packed staple that prevents complete relaxation, although in no case should the filaments or fibers be under any substantial tension. The process of this invention has its greatest utility in its application to staple that becomes partially packed in the hot water treatment or is in a partially restrained state as opposed to fibers which are completely loose.

It is indeed surprising that this simple combination of steps has such a pronounced effect on the fibers. Neither hot water treatment alone nor hot air relaxing alone will produce substantially as much improvement in bulk density, i. e. crimp retentivity, which shows up as bulk improvement in the finished goods, as will the combination of treatments of this invention. Hot water treatment followed by hot air relaxing did not yield as great bulk improvement and again the handle was undesirably harsh. So far as bulk improvement is concerned, the dry hot air treatment and the hot water treatment are not additive, but in combination, they both appear to contribute to the desired high bulk density and at the same time produce fibers which are convertible into yarns and fabrics with a very pleasing soft handle about equal to the control yarns which were not subjected to either hot air or hot water treatment.

The following examples which compare results obtained under various conditions of treatment are given to illustrate this invention further without any intention of limiting the invention thereby. The controls, of course, have not been processed and have the original pleasant handle.

EXAMPLES

A fiber-forming acrylonitrile copolymer of about 94% acrylonitrile and 6% methyl acrylate dissolved in N,N-dimethylformamide was spun into multifilament tows by the dry or evaporative process. These tows were washed and drawn about 4X to yield two sets of fiber deniers (2 denier and 3 denier) and while in the form of continuous filament tows were crimped with the aid of steam in a stuffer box crimper of the type shown in Hitt U. S. Patent No. 2,311,174 and then cut into 2½ inch staple. A portion of each production was subjected to hot air drying for about ten minutes at a temperature of 320° F. in the relaxed condition while another portion was dried under normal conditions (at about 260° F.). Fifty (50 pound lots of both 2 denier and 3 denier staple so prepared, with or without a hot water treatment under partial restraint, i. e. in a semi-packed condition in a stock dye kettle, as is recorded in the table that follows, were spun into 2/26 worsted count yarns with 10 turns per inch of Z twist in the singles and 5 turns per inch of S twist in plying. The many yarn samples were suitably finished and optimum garments were knit and fabricated from each item on a 12 cut circular jersey knitting machine. The garments were dyed in an open paddle type of machine at near the boil, i. e. 205° F. to 210° F. From these garments, tests were made on each item for fabric thickness, fabric weight, bulk density and handle. The dyeing test is applied to determine if the fibers or fabricated structures retain the desired loft and handle.

Table

50# Fiber Samples	Fiber Denier	Treatment	Fabric Thickness, Inches	Fabric Weight, oz./sq. yd.	Bulk Density, cc./gm.	Percent Increase Bulk Density	Handle
1.....	2	Control—260° F.....	0.0489	7.66	4.78	Control	Satisfactory.
2.....	2	H. W.—212° F. ¹	0.0487	6.52	5.60	17.2	Harsh.
3.....	2	H. W.—240° F.....	0.0529	6.76	5.86	22.6	Do.
4.....	2	H. A.—320° F. ²	0.0444	5.88	5.66	18.4	Too soft, limp.
5.....	2	H. W.—212° F.; H. A.—300° F.....	0.0450	6.10	5.50	15.1	Harsh.
6.....	2	H. W.—240° F.; H. A.—300° F.....	0.0512	7.10	5.40	13.0	Do.
7.....	2	H. A.—320° F.; H. W.—212° F.....	0.0446	5.85	5.70	19.3	Satisfactory.
8.....	2	H. A.—320° F.; H. W.—240° F.....	0.0504	6.29	6.00	25.5	Do.
9.....	2	H. A.—320° F.; 15# Steam ³	0.0478	6.20	5.80	21.3	Do.
11.....	3	Control—260° F.....	0.0450	6.30	5.35	Control	Do.
12.....	3	H. W.—212° F.....	0.0542	6.89	5.89	10.1	Harsh.
13.....	3	H. W.—240° F.....	0.0558	6.66	6.36	18.9	Do.
14.....	3	H. A.—320° F.....	0.0452	5.70	6.00	12.1	Too soft, limp.
15.....	3	H. W.—212° F.; H. A.—300° F.....	0.0456	5.60	6.10	14.0	Harsh.
16.....	3	H. W.—240° F.; H. A.—300° F.....	0.0522	6.50	6.00	12.1	Do.
17.....	3	H. A.—320° F.; H. W.—212° F.....	0.0470	5.50	6.50	21.5	Satisfactory.
18.....	3	H. A.—320° F.; H. W.—240° F.....	0.0530	6.10	6.50	21.6	Do.

¹ H. W.=hot water.² H. A.=hot air.³ 15# Steam=steam treatment for 10 minutes at 15 pounds/square inch gauge.

Handle is a property difficult to rate for there is no precise test and rating is a matter of opinion. In these tests, people skilled in the art passed on each of the fabrics and the handle of those samples rated satisfactory was satisfactory to all the testers. In the case of the samples rated harsh or too soft and limp, the degree of harshness or limpness may have varied with individuals, but, again, all the testers were agreed that these samples were harsher or softer and more limp than desired and should not be considered acceptable.

Similar successful results are obtained using fibers made from a copolymer of acrylonitrile, methyl acrylate and potassium styrenesulfonate (94/5.9/0.1).

The polymers used in fiber form in this invention are polymers containing at least about 85% acrylonitrile and up to about 15% of at least one other monomer copolymerizable with acrylonitrile. Such monomers include methyl vinyl ketone, styrene, acrylic acid and methacrylic acid and their esters or other derivatives, isobutylene, butadiene, sulfoarylethylenes, such as those disclosed in Millhiser French Patent 1,106,816, and such other copolymers as those disclosed in such patents as U. S. Nos. 2,404,714; 2,404,727 and No. 2,486,241.

Standard equipment may be employed in this invention; of course, in those treatments involving water at a temperature above its boiling point, closed pressure systems, such as a pressure kettle, will be used. In the dry heat treatment, inert atmospheres such as air, nitrogen, carbon dioxide and the like are used. Using such atmospheres and the relatively short exposure times, no adverse discoloration of the fibers occurs.

The time involved in the heat-relaxing step should be short, being only a matter of a few minutes. Generally, only such time as is needed to bring the fibers up to a temperature of at least about 300° F. is used. This takes around ten minutes in most instances. The time in the hot water or steam treatment may be varied from a few minutes to several hours, if desired, with a treatment of about ten minutes usually being sufficient to effect the desired results. Both steps are applied to fibers which are in a free-to-relax condition. The temperature used in the hot water treatment may vary from about

212° F. to about 280° F. with temperatures in the range of 212° F. to 240° F. being preferred.

By the process of this invention, the crimp in oriented, crimped acrylonitrile copolymer yarns is retained. Bulk density and handle are very satisfactory, and the knitting characteristics, fabric resilience and garment dimensional stability are commercially acceptable, the orientation, tenacity and similar properties of the treated fibers being retained. While the process can be used for unoriented, crimped fibers, it is generally used with the oriented fibers since they are commercially used for their greater strength.

Any departure from the above description which conforms to the present invention is intended to be included within the scope of the claims below.

We claim:

1. A process for treating crimped fibers made from acrylonitrile copolymers derived from at least about 85% of acrylonitrile which comprises subjecting the said fibers while they are substantially free to relax to dry heat at a temperature of from about 300° F. to about 350° F. until the said fibers attain a temperature of at least about 300° F. and treating the resultant, heat-relaxed fibers with water at a temperature of from about 212° F. to about 280° F. while said fibers are substantially free of tension.

2. A process in accordance with claim 1 wherein said treatment with water endures for about ten minutes.

3. A process in accordance with claim 1 wherein said copolymer is a copolymer of acrylonitrile and methyl acrylate.

4. A process in accordance with claim 1 wherein said copolymer is derived from acrylonitrile, methyl acrylate and sulfoarylethylene.

5. A process in accordance with claim 1 wherein the temperature of the water is in the range of from about 212° F. to about 240° F.

6. A process in accordance with claim 5 in which the treatment with water endures for several minutes.

No references cited.