Patented July 25, 1950

2,516,562

UNITED STATES PATENT OFFICE

2,516,562

PROCESS OF MAKING ARTIFICIAL WOOL FROM NYLON FIBERS

Boynton Graham, Claymont, Del., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

No Drawing. Application December 3, 1946, Serial No. 713,851

2 Claims. (Cl. 8—115.5)

1

This invention relates to textile materials and more particularly to fluffy nylon filaments having wool-like properties, and to a process for their preparation.

The synthetic linear polyamides known as nylon and the production of fibers and fabrics therefrom have been described in a number of United States patents and in particular in 2,071,250, 2,071,253 and 2,130,948. These fiber-forming polycarbonamides described in the previously mentioned patents are obtained by several methods, for example, by self-polymerization of a monoaminomonocarboxylic acid, e. g., 6-aminocaproic acid or 12-aminostearic acid, by reacting in substantially equimolecular proportions a dibasic acid, e. g., adipic or sebacic acid with a diamine, e. g., hexamethylenediamine or with a monoaminomonohydric alcohol, e. g., monoethanolamine, it being understood that these reactants can be replaced by their equivalent amide-forming derivatives. These linear polyamides also include polyesteramides, for example, those obtained by admixture with other linear polymer-forming reactants, such as glycol-dibasic acid mixtures, or mixtures of hydroxy acids, with the previously mentioned polyamide-forming reactants. The polyesteramides, when used in the practice of the invention described herein, should contain a ratio of amide groups to ester groups of at least 10%, and preferably 50% or more, amide groups. On hydrolysis with hydrochloric acid the amino acid polymers yield the amino acid hydrochloride, the diamine-dibasic acid polymers yield the diamine hydrochloride and the dibasic carboxylic acid, and the amino alcohol-dibasic acid polymers yield the amino alcohol hydrochloride and the dibasic carboxylic acid. In these polyamides the average number of carbon atoms separating the amide groups is at least 2 and usually less than 12. The preferred polyamides have an intrinsic viscosity of at least 0.4 (defined as in Patent 2,130,948) and a unit length (defined in Patents 2,071,253 and 2,130,948) of at least 7.

The nylons used in the practice of the present invention are those obtained from reactants of the above mentioned kind which yield polyamides having hydrogen-bearing amide groups. These polyamides are insoluble in water but are soluble in phenols. The fibers used are oriented to at least the extent given by a stretching or draw ratio of at least 3.5:1, but usually as high as possible to give high tenacity filaments.

The nylon fabrics, in the absence of special treatment of the fiber or fabric, are deficient in

2

wool-like feel and appearance. These properties are desired in nylon fabrics to be used as garments which should have a soft warm feel. Fluffiness has been obtained in nylon by crimping as described in U. S. Patents 2,197,896 and 2,287,099. These treatments are not entirely satisfactory because the products do not exhibit the random crimp, high residual elongation and good dye receptivity characteristic of wool.

This invention has as an object improved nylon filaments having wool-like crimp, elongation and dye receptivity. A further object is a method for obtaining wool-like filaments of this kind. Other objects will appear hereinafter.

The above objects are accomplished by heating while in relaxed condition oriented nylon filaments, which have been treated to render them insoluble in solvents in which the nylon was initially soluble, to a temperature of from 10° C. to 50° C. below the softening point of the treated filaments until the filaments have shrunk at least 20% and preferably at least 50% of their original oriented length.

The insolubilized filaments are best obtained by treating oriented filaments which comprise nylon containing hydrogen-bearing amide groups with formaldehyde in the presence of an acidic catalyst which gives a pH below 3 in saturated aqueous solution. In this method, the oriented filaments are heated with the formaldehyde until at least 0.25 and preferably 1 to 5% formaldehyde is combined with the nylon, a suitable procedure consisting in heating the filaments with acidic aqueous Formalin solution, e. g., for 10 minutes at 80° to 85° C. in 37% formaldehyde containing an acidic catalyst such as 1% ammonium chloride, followed by drying and baking, e. g., for 15 minutes at 140° C. Another method of obtaining the insolubilization of the polyamide filament is by treating the oriented polyamide filament with an aqueous solution containing 0.1–10% of a non-volatile acid or salt which gives a pH below 3 in saturated aqueous solution. Such acid catalysts include oxalic acid, maleic acid, para-toluene-sulfonic acid and salts such as ammonium chloride and calcium chloride. The filaments are removed from the acid catalyst solution, dried, and are subsequently treated with formaldehyde, preferably in the anhydrous condition as a vapor at temperatures of from 100–250° C. and suitably at 130–150° C. The products obtained by such process may be rinsed with water to remove residual acid catalyst. The filaments obtained are insoluble in such solvents as metacresol commonly applied to dissolve the initial polyamide.

The insoluble polyamide filaments are heated to temperatures 10–50° C. below their softening point while maintained in relaxed condition, that is, in such a condition that the filaments can shrink to an extent of at least 20% and preferably at least 50%. The temperature of treatment is approximately that of the original melting point prior to insolubilization, although it may be slightly higher and is dependent upon the particular fiber-forming polyamide employed. The heating may be carried out over a heated bar, plate or slot, in a hot liquid, in hot air, or by such a suitable method as to heat the fiber to the required temperature. The time of heating is at least a few seconds and may be a minute or more.

The following example further illustrates the practice of this invention.

Example

Oriented filaments of polyhexamethyleneadipamide were soaked for 16 hours at room temperature in a 1% aqueous ammonium chloride solution, excess solution was removed, and the filaments were dried at room temperature. The filaments were then treated with anhydrous formaldehyde vapor for 15 minutes at 150° C. The filaments after washing and drying were difficultly fusible and insoluble in cresol whereas the initial filaments were soluble and melted at 250° C. The filaments were heated for about 3 seconds by passing in a freely relaxed condition over a metal bar heated at a temperature of 260° C. The filaments obtained were shrunk about 75% and were fluffy and wool-like in appearance. Dye receptivity was improved, the treated filaments exhibiting wool-like dye receptivity. The shrunken filaments could be elongated about 120%, whereas untreated nylon can be elongated only 15–20%.

Further examples of particular polyamide-forming reactants that can be used in making the nylon fibers are mono-amino-carboxylic acids such as 6-aminocaproic and 12-aminostearic acid; dicarboxylic acids such as glutaric, adipic, sebacic; diamines such as hexamethylenediamine, and meta- and para-phenylenediamine.

The optimum temperature for heat shrinking of the insoluble polyamide, as has been noted previously, corresponds approximately to the melting point of the original polymer which for many of the fiber-forming polyamides is approximately 200–250° C. The time is dependent upon the type of heating and must be sufficient for the filament to be heated to the temperature previously mentioned. The time will depend on filament denier, the minimum time being that required to raise the core of the filament to the shrinking temperature. Thus, for a low denier

filament with efficient heat transfer, one second is sufficient. For a high denier bristle, 60 seconds may be required. In all cases, longer times are operable, but prolonged heating (above 5 minutes) which causes degradation should be avoided. It is to be understood that the shrinkage may be mechanically restrained to any value less than the maximum obtainable, but greater than at least 20%, to get better tenacity, but with lowered fluffiness, elongation and dye receptivity.

The yarns obtained by the process described above are useful in the preparation of garments which have a soft warm feel. The filaments may be blended with wool, cellulose, rayon or other filamentary material in the preparation of fabrics.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A process for obtaining improved nylon filaments having a wool-like crimp, said process comprising heating with inert dry heat in relaxed condition to a temperature of from 10° C. to 50° C. below its melting point, an oriented nylon filament which has been subjected to treatment with formaldehyde rendering it insoluble in meta cresol, and continuing said heating until the filament has shrunk at least 50% of its original length, said nylon filament after said treatment with formaldehyde containing at least 0.25% combined formaldehyde, and prior to said treatment being insoluble in water, soluble in meta cresol, and having a melting point above 200° C.

2. The process set forth in claim 1 in which said nylon is polyhexamethylene adipamide.

BOYNTON GRAHAM.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,177,637	Coffman	Oct. 31, 1939
2,199,411	Lewis	May 7, 1940
2,217,113	Hardy	Oct. 8, 1940
2,249,756	Finzel	July 22, 1941
2,346,208	Conaway	Apr. 11, 1944
2,365,931	Benger	Dec. 26, 1944

FOREIGN PATENTS

Number	Country	Date
519,038	Great Britain	Mar. 14, 1940
559,369	Great Britain	Feb. 16, 1944
562,555	Great Britain	July 6, 1944