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PRODUCTION OF FILAMENTS

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This invention relates to the production of filaments of synthetic linear condensation polymers and, more particularly, to a method of improving the properties of filament formed by the melt-spinning of polymeric hexamethylene adipamide, a specific synthetic linear condensation polymer.

By the term "synthetic linear condensation polymers" is meant new polymeric materials such as described in United States Patents 2,071,250 and 2,071,253 and in United States application, Serial No. 136,031, filed April 9, 1937 now Patent No. 2,130,948, all of W. H. Carothers. These polymers are capable of being formed by melt-spinning into filaments, ribbons, and the like, which possess the unusual property of being capable of being "cold-drawn," i. e., drawn or elongated under application of stress in the solid state, with resulting orientation of internal structure and corresponding marked benefits to their mechanical properties.

In a copending application for United States Patent filed by G. D. Graves on February 15, 1937, entitled "Synthetic polymers and shaped articles therefrom," Serial No. 125,926, now Patent No. 2,212,772 is disclosed and claimed a step, conveniently designated "quenching," which improves very materially the strength, toughness, and pliability of filaments and the like thus spun and drawn. The step of quenching comprises essentially bringing the filaments and the like, immediately upon their formation from molten polymer, into contact with a non-solvent liquid maintained at such temperature as to effect rapid chilling of the filament and the like. The procedure disclosed in this Graves application is broadly applicable to synthetic linear condensation polymers and contemplates the use of a quenching liquid maintained at a temperature within a wide range although favoring a temperature somewhat below ordinary temperature to insure a wide difference between the temperature of the freshly spun polymer and the quenching liquid.

In certain uses of filaments of synthetic linear condensation polymers, particularly to coarser filaments, it is of great importance that the filament be substantially perfectly circular in cross section and it is, therefore, important to produce such filament with a minimum of deviation in cross sectional shape from that of the circular orifice from which it has been spun. With some of these polymers, it appears that the production of filaments of circular cross section may readily be accomplished by following the teachings of the prior art but this has not been the case with respect to filaments of polymeric hexamethylene adipamide.

An object of the present invention is to provide a process of manufacturing polymeric hexamethylene adipamide filaments which are substantially perfectly circular in cross section. A further object is to provide such a process which will produce filaments in no way impaired with respect to their other properties. Other objects of the invention will be apparent from the description given hereinafter.

The above objects are accomplished according to the present invention by extruding molten polymeric hexamethylene adipamide through a circular orifice into a quenching liquid having substantially no solvent action on the polymer and maintained at a temperature of at least 35° C. Preferably, the quenching liquid is water and, for practical operation, it should be kept at a temperature of 35°–45° C. The filament is subsequently cold-drawn after being thoroughly soaked in water or many be cold-drawn at a temperature somewhat above room temperature.

It has been observed that, if polymeric hexamethylene adipamide filaments are quenched in water or the like maintained at a relatively low temperature in order that the difference between the temperature of the extruded filament and that of the water may be very great, the resulting filament is, to a greater or lesser degree, "flat," that is, ellipsoidal. This deviation in the cross section of the filament from circular is frequently highly objectionable. Peculiarly, this difficulty is not encountered in the manufacture of certain of the synthetic linear condensation polymers; in fact, polymeric hexamethylene adipamide may be unique in this respect.

The present invention resides in the discovery that, if extruded filaments of polymeric hexamethylene adipamide are quenched in a non-solvent quenching liquid maintained at a temperature of 35° C. or more, the cross sectional shape of the extruded filament is not changed but retains the shape of the orifice through which the molten polymer was extruded. This is especially observable in the manufacture of coarser filaments and it is the manufacture of such filaments to which the present invention is most advantageously applicable. Because of this discovery, the manufacture of a filament of substantially circular cross section can be assured by extruding the molten polymeric hexamethylene adipamide through a circular orifice into a quenching liquid held at a temperature of at least 35° C.

Since the use of such temperature for quenching adds somewhat to the difficulty of the usual subsequent operation of cold-drawing, the filament thus quenched is preferably soaked in water before being cold-drawn, or is cold-drawn while at a temperature somewhat above room temperature, either of which techniques facilitates the cold-drawing.

Soaking for about 24 hours in water at room temperature is ordinarily sufficient to overcome any difficulty in cold-drawing but the allowance of a longer time, such as two days, does no harm and may be preferable in commercial practice to ensure thorough contact of water with the inner layers of a large spool of filament and a longer period of soaking may be required in the case of filaments of especially large diameter.

While the quenching liquid may be held at temperatures considerably above 35° C. in so far as obtaining a polymeric hexamethylene adipamide filament of cross sectional shape substantially identical with that of the orifice through which the polymer is extruded, is concerned, it is preferred that the temperature of the quenching liquid should not exceed 45° C.

The effectiveness of the procedure of the present invention is readily illustrated by extruding polymeric hexamethylene adipamide filaments into water as the quenching liquid at various temperatures. Molten polymeric hexamethylene adipamide, of melt viscosity 700 poises, was extruded through a circular orifice of 0.060″ diameter at a rate of 315 feet per minute into a quenching bath of water at a distance of 3.5″ from the orifice. Sufficient filament was extruded with the quenching liquid held at various temperatures to permit of the carrying out of subsequent operations under commercial conditions. Specimens of filaments thus produced were drawn by hand, i. e., stretched by hand until they ceased to yield, without any intermediate treatment. Filaments quenched in the quenching liquid maintained at 40° C. or less could be drawn satisfactorily in this manner.

Other specimens of the filaments thus produced were soaked in water at 20° C. for 24 hours and then cold-drawn under uniform conditions of commercial mechanical equipment. The percentage of elongation imposed upon each filament was about 260%, i. e., every 100 feet was drawn to 360 feet. Difficulty was encountered in drawing the filament specimens quenched at 40° C. and higher. By soaking 48 hours, filament specimens quenched at temperatures up to 60° C. could be satisfactorily cold-drawn and, by increasing the soaking another 24 hours, the filament specimens quenched at 70° C. could be satisfactorily cold-drawn.

Twenty measurements were made of maximum and minimum diameters of each of the filaments soaked in water and drawn, in order to secure the numerical measure of their deviations from circularity. The results obtained are tabulated below with the figures there given with respect to the deviation being in each case the difference between average maximum and minimum diameters expressed as a percentage of the average mean diameter:

Temperature of quenching liquid	Time of soaking before drawing	Deviation from circularity
° *C.*	*Days*	*Percent*
12	1	31.6
20	1	21.1
25	1	12.8
30	1	7.2
35	1	1.9
40)	1)	1.7)
40)	2)	2.9)
50	2	1.4
60	2	1.4
70)	2)	1.3)
70)	3)	1.6)
80)	2)	2.4)
80)	3)	1.9)

From this table it is apparent that "flatness" of the cross sectional shape of the filament increased as the temperature of the quenching liquid was decreased from about 35° C. On the other hand, it is clearly shown that filaments quenched at temperatures from 35° C. upward uniformly exhibit a deviation from circularity so slight as to be hardly greater than the experimental error of the measurements (i. e. 0.00025″ in 0.018″, or 1.4%).

It is not preferred to exceed a temperature of 45° C. for the quenching liquid because the filaments quenched above that temperature become more difficult to cold-draw while no advantage accrues from the use of higher temperatures. Also, there may be a certain falling off of various desirable physical properties of cold-drawn filaments quenched at temperatures above 45° C. such as the tensile strength of the filaments and their "recovery," i. e., extent of their ability to straighten after having been severely bent.

In carrying out the present invention the molten polymer should be extruded into the quenching liquid without any prolonged cooling of the extruded polymer intervening. That is, the polymer may be molten when it contacts the quenching liquid or may be set or partially set due to the preliminary cooling action of the air but the temperature of the polymer should not be materially below the melting point of the polymer. Thus, in the case of polymeric hexamethylene adipamide, which has a melting point (in the absence of oxygen) of about 263° C., it is desirable that the temperature of the polymer be above about 245° C. as it enters the quenching liquid.

The liquid used to quench the polymer should preferably be chemically inert toward the polymer and should not have an appreciable solvent action thereon under the conditions of quenching. Water meets these requirements and, in addition, is readily available and has a high specific heat. It is, therefore, the preferred quenching medium. However, a large variety of other liquids may be employed such as alcohols, ketones, ethers, esters, hydrocarbons, and chlorinated hydrocarbons. The quenching liquid may consist of a mixture of compounds. Also, it may contain wetting agents, dyes, and the like.

It is optional whether the quenching liquid is used in the form of a bath or a spray although the former is apt to be more convenient. The temperature of the liquid may be regulated or kept constant by any suitable means.

After the filament has been quenched, the cold-drawing is facilitated by first soaking the filament in water, the duration of said soaking being regulated to avoid any difficulty in the cold-drawing. Usually a period of 24 hours will be sufficient but, as shown hereinbefore, a soaking of 48 hours or even longer may be desirable where the filament has been quenched in a liquid maintained at a relatively high temperature. Rather than soaking the filament in water, it may be warmed to a temperature of from about 50° C. to about 100° C. to facilitate cold-drawing.

The present invention provides a method of ensuring the substantial circularity of cross sections of filaments of polymeric hexamethylene extruded from a circular orifice and is of great practical value in the production of filaments of this material for uses in which circularity of the filament is of prime importance. This circularity of the filament is achieved in accordance with this invention at some sacrifice of tensile strength but without significant impairment of the other important properties of the filament.

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 the invention is not limited to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. Process of manufacturing filaments of polymeric hexamethylene adipamide of circular cross section, which comprises extruding molten polymer through a circular orifice into a quenching liquid having substantially no solvent action on said polymer and maintained at a temperature of 35° C. to 45° C.

2. Process of manufacturing filaments of polymeric hexamethylene adipamide of circular cross section, which comprises extruding molten polymer through a circular orifice into water maintained at a temperature of 35° C. to 45° C.

3. Process of manufacturing filaments of polymeric hexamethylene adipamide of circular cross section, which comprises extruding molten polymer through a circular orifice into water maintained at a temperature of 35° C. to 45° C., thereafter soaking the filaments thus formed in water, and thereafter cold-drawing said filaments.

4. Process of manufacturing filaments of polymeric hexamethylene adipamide of circular cross section, which comprises extruding molten polymer through a circular orifice into water maintained at a temperature of 35° C. to 45° C., thereafter soaking the filaments thus formed in water at about 20° C. for at least 24 hours, and thereafter cold-drawing said filaments.

5. Process of manufacturing filaments of polymeric hexamethylene adipamide of circular cross section, which comprises extruding molten polymer through a circular orifice into water maintained at a temperature of 35° C. to 45° C., thereafter heating the filaments thus formed to a temperature of 50° C. to 100° C., and drawing said filaments while at approximately said temperature.

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