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2,296,202

PROCESS OF MAKING ARTIFICIAL WOOL

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Fig. 1.

Fig. 2.

Fig. 4.

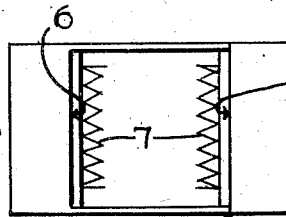
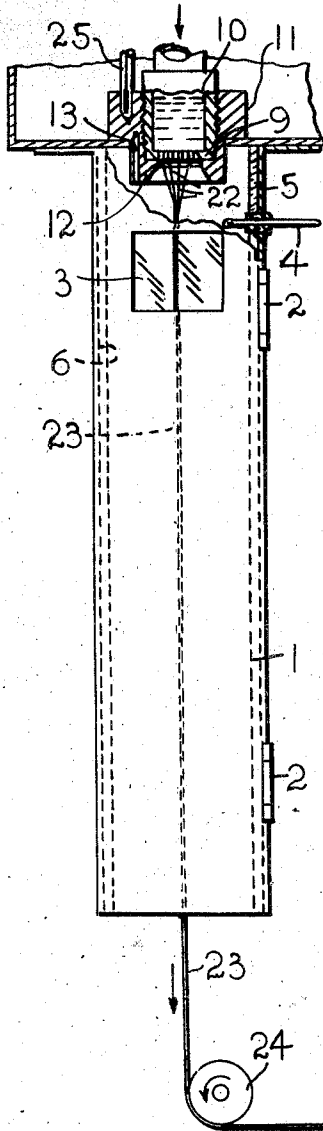
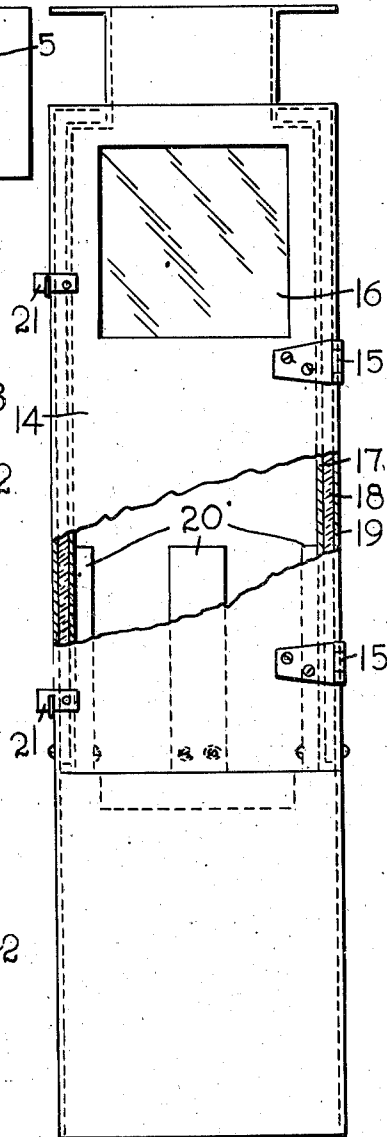
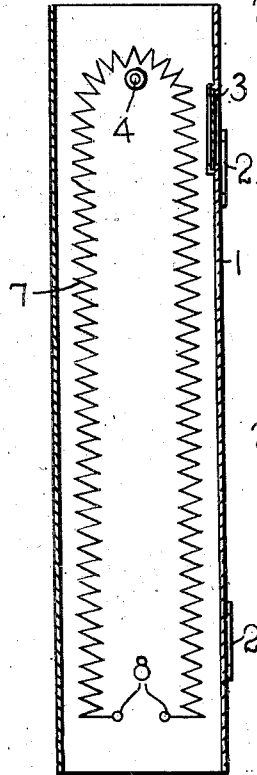


Fig. 3.



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PROCESS OF MAKING ARTIFICIAL WOOL

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8 Claims. (Cl. 18-54)

This invention relates to synthetic fibers and more particularly to artificial wool.

This invention is an improvement in the method of making artificial wool from synthetic linear superpolymers as described in application Serial No. 183,922 filed January 7, 1938, by J. B. Miles and myself. The invention in that application is based on the discovery that synthetic linear polymer filaments when cold drawn while wet with water or other mild swelling agent, and then dried while in relaxed condition crimp spontaneously to a product which is remarkably wool-like. This synthetic wool is superior to those heretofore known in strength, especially wet strength, crimp retentivity both wet and dry, elasticity, heat stability, and heat insulating properties. These crimped fibers were obtained by a process wherein suitably treated wet fibers crimped spontaneously during drying in the relaxed condition. One method for rendering the fibers susceptible to spontaneous crimping consisted in giving undrawn or partly drawn wet filaments of the polymer a heat treatment ("conversion") in a suitable medium such as oil, followed by further cold drawing. Another method consisted in simply preparing filaments of the polymer by melt spinning, followed by wetting and cold drawing the filaments. This last mentioned method, which is referred to as the "direct spinning process," in addition to giving a better product, would be the more economical and convenient process were it not for certain restrictions which have seriously handicapped this method. The most serious of the restrictions are that the process requires the use of polymers of low viscosity, which means that the fibers have a lower tensile strength than those made from higher viscosity polymers, and the process requires lower spinning temperatures than are consistent with efficient melt spinning. A further disadvantage is that it is difficult to reproduce results, i. e. obtain products of uniform quality from run to run.

This invention has as an object a new and improved method for the manufacture of artificial wool. A further object is the preparation of crimped fibers by an improved "direct spinning process" which is free from the disadvantages noted above. More specifically an object of this invention is to provide a process for melt spinning fiber-forming synthetic linear polymers susceptible to cold drawing, whether of low or high viscosity, which will impart spontaneous crimping properties to the resultant filaments. Other objects will appear hereinafter.

The above objects are accomplished by spinning from melt a synthetic linear polymer, which can be formed into filaments susceptible to cold drawing, e. g. crystalline synthetic fiber-forming linear polymers, and preventing the extruding filaments from cooling rapidly for an appreciable time after they have been extruded from the molten polymer. The filaments thus formed are then treated with a mild swelling agent therefor, e. g. water, cold drawn while wet, and dried in the relaxed condition, whereupon they crimp spontaneously.

This invention has resulted from a theory I have formulated that the ability of a synthetic linear polymer filament to crimp spontaneously after being cold-drawn wet and allowed to dry is dependent upon large differences in the sizes of the crystals in the filament. This theory is based on the following observations: An examination of a cross-sectional photo-micrograph of yarn, which is spun from low viscosity polyhexamethylene adipamide at the usual spinning temperature (for instance about 283° C. in the melt and 270° C. at the spinneret) and which is incapable of spontaneous crimping, revealed that the crystallites in the filaments of the yarn are so small as to be invisible and are presumably of relatively uniform size. On the other hand, a similar photo-micrograph of yarn spun from the same polymer but at the lower spinning temperature required to produce a spontaneous crimpable yarn revealed that crystal growth had proceeded to the point where many of the crystals were visible under the microscope and that the crystals were extremely non-uniform in size throughout the individual filaments. I observed further that when a medium or high viscosity polymer is melt spun in the usual method, even though the lowest practical temperature is used, the filaments are not spontaneously crimpable to any appreciable extent and appear under the microscope similar to those in a yarn obtained from low viscosity polymer at high temperature. I discovered further that low, medium, or even high viscosity polymer, when spun from melt under the conditions described below for controlling the rate of cooling gave spontaneously crimpable filaments which showed marked non-uniformity of crystal size when examined under the microscope.

The slow cooling of the extruded filaments brought about by the practice of this invention allows for the non-uniform crystal growth which I have found to characterize the spontaneously crimpable filaments. The most effective and

preferred method of practicing my invention consists in passing the filaments directly after extrusion from melt, i. e. on leaving the spinneret, through a zone of gas or vapor, the temperature of which is well above room temperature, namely, at least 75° C., but not higher than about 25° C. below the melting point of the polymer. The preferred temperature and length of the heated zone will depend upon such factors as the melting point of the polymer, the viscosity of the polymer, the denier of the filaments spun, and the speed of spinning. If the polymer viscosity or the spinning temperature or both are only slightly above those best suited for the production of spontaneously crimpable filaments, the heated zone may be relatively short and of low temperature. If, on the other extreme, the polymer viscosity or the spinning temperature or both are far above those best suited for the production of spontaneously crimpable yarn, the heated zone must be longer and of a higher temperature in order to effect the desired results. The most suitable length and temperature for the heating zone are also dependent upon the spinning wind-up speed and the filament denier. As the spinning wind-up speed is increased, other things being constant, it is desirable to lengthen the heating zone or increase its temperature, or both. On the other hand, as the filament denier is increased the necessity for retarded cooling in order to produce spontaneous crimpability is decreased. In most instances the temperature will be within the range of from 75° C. up to about 25° C. below the melting point of the polymer, although in rare instances it may be in the range from 50° C. up to 10° C. below the melting point of the polymer. The length of the heated zone through which the filament passes will in general be such that the filament will be exposed to the heated zone for from 0.02 second to 0.2 second but in some cases the time of exposure may range from 0.01 to 1.0 second. Depending on the spinning speed the length of the heated zone will generally range from 1 to 5 feet. The heated zone need not have a uniform temperature, in fact, it is desirable that the portion of the zone close to the spinneret have a higher temperature than the portion farthest removed from the spinneret. The filamentary stream of molten filaments solidify during their passage through the heated zone.

Suitable apparatus for carrying out my invention is shown in the accompanying drawing in which

Fig. 1 is a front elevation of a heated chimney with a spinneret associated therewith;

Fig. 2 is a plan view of the chimney shown in Fig. 1;

Fig. 3 is a longitudinal sectional view; and

Fig. 4 is a front elevation of another form of chimney.

The chimney shown in Figs. 1, 2 and 3 is about 18" high and is 4" square and is constructed of a suitable metal such as galvanized iron. The entire front consists of a door 1 hinged at 2 and provided with a 2 x 2" window 3. A similar window may be mounted in the opposite wall. The chimney about 2" from its top is provided with an inlet for a thermometer 4. On the inner side of each of the opposite walls 5 and 6 are resistance coils 7. The top of each coil is about 2" from the top of the chimney and the terminal ends 8 of the coil about the same distance from the bottom. The resistance

coil is designed to carry 50, 100 or 200 watts for heating. Any temperature may be obtained between room temperature and 190° C. within 2" from the top. The temperature at approximately 4" from the bottom may vary between room temperature and 75° C.

The upper end of the chimney fits tightly against the bottom of the spinneret assembly of the melt spinning machine. The spinneret assembly includes a spinneret plate 9 forming the bottom of a drum 10 into which is fed the molten polymer from the associated parts of the melt spinning machine (not shown) above. Around the drum is threaded the spinneret block 11 in the form of the nut 11 provided with lugs 12 which abut the spinneret plate 9 and with a thermocouple well 13 adjacent the spinneret plate. The body of the spinneret block 11 is also provided with an inlet for a thermometer 25. The filaments 22 extruded through the plurality of holes in the spinneret plate unite to a single yarn 23 which is drawn around the roll 24 by a wind-up roll not shown.

Fig. 4 shows a chimney of similar construction having a door 14 hinged at 15 and provided with latches 21 and window 16. This chimney is about 7" square and 24" long. This chimney as well as that illustrated in Figs. 1-3, is preferably of insulated construction which may consist of an inner wall 17, and insulating material 18 between the inner wall 17 and outer wall 19. Strip heaters 20 of conventional type are located on three sides of the chimney near the bottom. By this construction the operator may obtain practically uniform temperature throughout the chimney.

Due to the difficulties involved it is not practical to determine the actual temperature of the molten filaments as they leave the spinneret. The spinneret temperatures given below are those obtained from the thermocouple in the well 13 shown in the spinneret block near the spinneret. This temperature is believed to be somewhat below that of the molten polymer passing through the spinneret orifices. The melter temperature is that of a grid (not shown in the figures) which melts the polymer above the spinneret and spinneret block and from which the molten polymer flows down through the spinneret block to the spinneret.

This invention is further illustrated in the examples given below. In Examples I-V, inclusive, the linear polymer is the polyamide, polyhexamethylene adipamide, and in Example VI it is polydecamethylene adipamide. The chimney shown in Figs. 1 to 3 was used in the first four examples and that shown in Fig. 4 in the last two examples.

Example I

Polymer having a melt viscosity of 200 poises at 285° C. was spun under the following conditions: Melter temperature, 270° C.; spinneret temperature, 262° C.; heating zone temperature 2 inches below the spinneret, 180° C.; spinning wind-up speed, 800 ft./minute; yarn spun, 70 denier, 5 filaments. The yarn obtained in this way was soaked in water and cold drawn 200% (i. e. to three times its original length) while still wet. On drying the yarn under no tension it crimped spontaneously to give a highly crimped wool-like yarn. When the same polymer was spun and treated under essentially the same conditions, except that no heating chimney was used, it showed no crimping properties.

Example II

The spinning conditions were as follows: melt viscosity of the polymer, 600 poises at 285° C.; melter temperature 269° C.; spinneret temperature, 258° C.; heating chimney temperature two inches from the spinneret, 180° C.; heating chimney temperature 14 inches from the spinneret, 70° C.; spinning wind-up speed, 800 ft./minute; yarn spun, 70 denier, 5 filaments. The resultant yarn, when cold drawn 200% while wet with water and allowed to dry under no tension, showed excellent spontaneous crimping properties. When the same polymer was spun without the heated zone but otherwise treated under the same conditions, it showed practically no spontaneous crimping properties.

Example III

The spinning conditions were as follows: melt viscosity of the polymer, 130 poises at 285° C.; melter temperature, 279° C.; spinneret temperature, 267° C.; chimney temperature 2 inches below the spinneret, 215° C.; spinning wind-up speed, 1200 ft./minute; yarn spun, 224 denier, 15 filaments. The yarn produced in this way showed excellent crimping properties when cold drawn while wet with a swelling agent followed by drying in the relaxed condition. Yarn spun from the same polymer without the use of the heated zone but otherwise treated in the same manner, showed very poor crimping properties.

Example IV

The spinning conditions were as follows: melt viscosity of the polymer, 130 poises at 285° C.; melter temperature, 276° C.; spinneret temperature, 266° C.; heated zone temperature 2 inches below spinneret, 170° C.; spinning wind-up speed, 2200 ft./minute; yarn spun, 92 denier, 15 filaments. This yarn when cold drawn while wet with water and allowed to dry showed very good crimping properties.

Example V

The spinning conditions were as follows: melt viscosity of the polymer, 160 poises at 285° C.; melter temperature, 278° C.; spinneret temperature, 265° C.; spinning wind-up speed, 1200 ft./minute; yarn spun, 270 denier, 10 filaments. The chimney was used with temperature of 75° C. throughout its length. The yarn spun showed excellent spontaneous crimping properties when cold drawn while wet with water and then dried in the relaxed condition.

Example VI

The spinning conditions were as follows: melter temperature, 255° C.; spinneret temperature, 240° C.; spinning wind-up speed, 900 ft./minute; yarn spun, 150 denier, 10 filaments. The heated zone had a temperature of 125° C. throughout its length. The yarn spun showed excellent spontaneous crimping properties when cold drawn while wet with water followed by drying under no tension.

This invention is in general applicable to any synthetic polymer capable of being spun from melt into filaments which can be cold drawn. Examples of polymers of this kind are given in U. S. Patents 2,071,250 and 2,153,553 and in the above mentioned application. These polymers can be spun into filaments which yield oriented fibers upon application of stress herein referred to as "cold-drawing." Cold drawn fibers show definite orientation along the fiber axis where-

as the undrawn filaments are substantially un-oriented. The term "filament" as used herein refers to both oriented and unoriented threads which are drawn from the polymer regardless of whether the threads are long (continuous) or short (staple), while the term "fiber" refers more specifically to the oriented filaments. Particularly useful are the polymers obtainable from self-polymerization of amino acids or their amide-forming derivatives, e. g. the lactams, or by reacting a diamine with a dibasic carboxylic acid or its amide-forming derivative. These polymers are described in U. S. Patents 2,071,253 and 2,130,948. It is to be understood that the term "polymer" includes interpolymers obtained from a mixture of different linear polymer-forming reactants and that the term "polyamide" includes interpolyamides and all linear polymers containing a plurality of amide groups as an integral part of the main chain of atoms making up the polymer chain, as for instance the ester-amide interpolymers. Specific examples of polymers other than that mentioned in the examples which are of particular value in the practice of this invention are polypentamethylene sebacamide, polyhexamethylene sebacamide, poly-p-xylylene sebacamide, and polymeric 6-aminocaproic acid. The polymers may be modified with plasticizers, dyes, pigments, inert inorganic materials, resins, etc. as desired.

The optimum degree to which the filaments are cold drawn prior to crimping will differ with different polymers. Preferably the filaments are not cold drawn to the maximum extent possible. For most filaments, the optimum degree of cold drawing will vary from 150 to 300%. The cold drawing can be done in stages if desired. As already indicated, the filaments are cold drawn while wet with a mild swelling agent therefor. The preferred swelling agents are low-boiling, hydroxy-containing compounds such as water and the lower boiling aliphatic alcohols, e. g. methanol, ethanol, propanol, isopropanol, and isobutanol.

Wool-like synthetic polymer filaments can be prepared of almost any diameter by this process, but filaments having deniers in the neighborhood of 2 to 25 are most useful. The crimps produced in these filaments by the spontaneous crimping process are very irregular and wool-like in appearance. Some of them are large, some small, and they may be either helical or wave-like in appearance. In general, it may be said that the number of crimps per inch, the fineness of the crimps, and their permanence can be made equal to or better than that of natural wool. The crimp retentivity of the product can be increased by treatment with a hot, nonsolvent swelling agent, e. g. hot water or steam.

In the above examples the melt viscosity of the polymers varied from 130 poises to 600 poises. Although polymers having melt viscosities in this general range are most useful, the invention can be applied to polymers having an even wider viscosity range, e. g., from 50 poises to 1000 poises. The intrinsic viscosity of the polymers can also vary over a wide range, e. g. from 0.5 to 1.5. Spinning speeds ranging from 800 to 2200 feet per minute were used in the examples but the process is not limited to spinning speeds within this range. The spinning temperatures used may likewise vary widely, depending upon the melting point and thermal stability of the polymer. As previously stated, the

higher the spinning temperature, other things being equal, the more drastic the measures described herein must be in order to produce spontaneous crimpability. The filaments, after they have passed through the heated zone and before they have been crimped, can if desired be treated with steam, oil or a spinning finish without affecting materially the subsequent crimping operation.

The type of apparatus used to produce the heated zone is not critical. The essential requirement is that a heated zone be produced. This may even be accomplished by enclosing the area below the spinneret in such a way that the air within is warmed by the spinning unit itself or by the molten polymer as it leaves the spinneret. The atmosphere of the heated zone may be air or any inert gas or vapor, e. g. nitrogen, carbon dioxide, or carbon tetrachloride.

By practicing the slow cooling technic of this invention it is possible to produce spontaneous crimpable yarn from polymers which would not produce such yarn under otherwise comparable conditions. For example, a low viscosity polymer can be spun at advantageously higher temperatures than would otherwise be permissible, or a higher viscosity polymer may be used than would ordinarily give the desired results. Or both of these conditions are permissible when the process of this invention is applied. In other words, the spinning temperature and the viscosity of the polymer are much less critical than in the "direct spinning method" described in application Serial No. 183,922.

The wool filaments prepared by the process of this invention resemble those described in the above mentioned application. That is, they have good elastic properties, show fiber orientation when examined by X-rays, are quite insensitive to moisture, have good resistance to solvents and chemical reagents, are mothproof, have excellent crimp retentivity and show little tendency to lose strength on aging. The process by which the artificial fibers of this invention are made is of such a character that modifying agents, for example, delusterants and plasticizers, can be readily incorporated therewith. The fibers of this invention can be made either as long (continuous) fibers or can be cut into short (staple) fibers. They are useful for making knitted or woven fabrics, rugs, felts, and the like. If desired, the fibers and yarns can be mixed with other fibers and yarns, for example, viscose rayon, acetate rayon, cotton, silk, and wool.

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. In a process for making crimped filaments from a melt-spinnable, crystalline synthetic linear polymer, the step comprising extruding the molten polymer through a spinneret into a heated zone, of gaseous medium which is maintained at a temperature of at least 50° C. but

not greater than about 10° C. below the melting point of the polymer, and controlling the temperature of said heated zone and the time of passage of the extruded polymer therethrough to cool the extruded polymer at a gradual rate, and thereby inducing the formation of crystals of nonuniform size in the resultant filaments.

2. A process for making filaments of fiber-forming synthetic linear polymers spontaneously crimpable upon cold drawing them while wet with a mild swelling agent and then drying them in the relaxed condition, said process comprising extruding the molten polymer through a spinneret, and preventing rapid cooling of the resultant filaments by passing them after leaving the spinneret and before and during solidification through a heated zone of gaseous medium having a temperature of at least 50° C. but not greater than about 10° C. below the melting point of the polymer.

3. The process for making filaments of fiber-forming synthetic linear polymers spontaneously crimpable upon cold drawing them while wet with water and then drying them in the relaxed condition, said process comprising extruding the molten polymer through a spinneret in filamentary form, and preventing rapid cooling of the filaments by passing them, after leaving the spinneret and before and during solidification, through a heated zone of gaseous medium for a time of from about .02 second to 0.2 second, said zone having a temperature of at least 75° C. but not higher than 25° C. below the melting point of the polymer.

4. A process for producing spontaneously crimpable filaments which comprises extruding molten synthetic linear fiber-forming polymer through a spinneret, preventing rapid cooling of the resultant filaments by passing them, after leaving the spinneret and before and during solidification, through a zone of gaseous medium having a temperature of at least about 75° C. but not greater than about 25° C. below the melting point of the polymer for a time from about .02 second to about 0.2 second, wetting the filaments with water, cold drawing the wet filaments, and drying the filaments in a relaxed condition.

5. The process set forth in claim 2 in which said polymer is a polyamide.

6. The process set forth in claim 4 in which said polymer is a polyamide.

7. The process as set forth in claim 4, in which said polymer is polyhexamethylene adipamide.

8. In a process for producing crimped filaments, the steps comprising extruding molten synthetic linear fiber-forming polymer through a spinneret, preventing rapid cooling of the resultant filaments by passing them, after leaving the spinneret and before and during solidification, through a heated zone of gaseous medium the temperature of which is at least 50° C. but not greater than about 10° C. below the melting point of the polymer, the temperature of said heated zone becoming progressively lower as the distance from the spinneret increases.

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