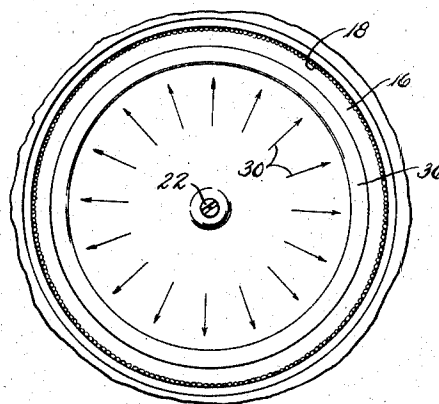
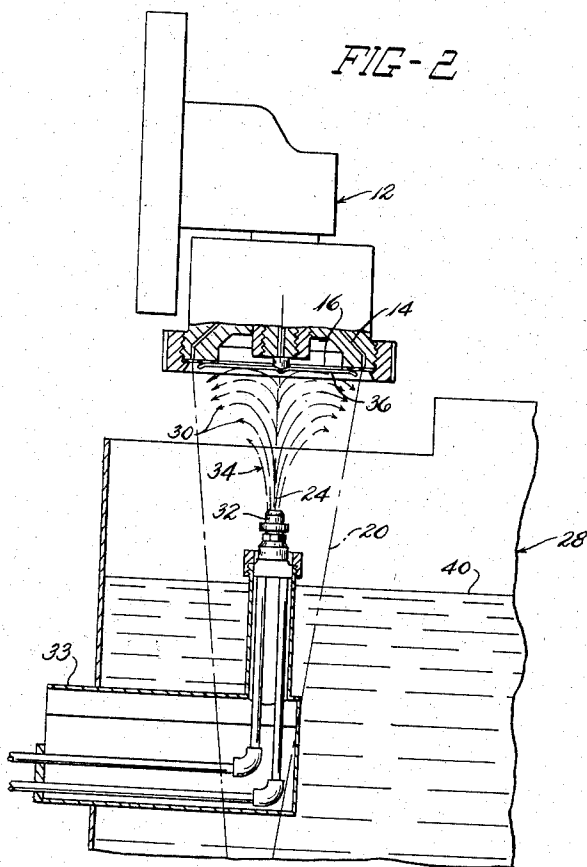
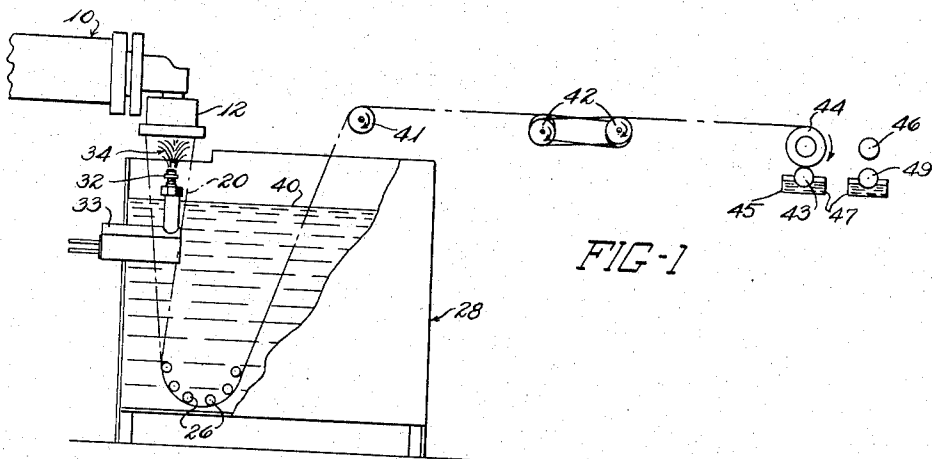


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METHOD FOR PRODUCING CRIMPED FILAMENTS

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METHOD FOR PRODUCING CRIMPED FILAMENTS

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This invention relates to the production of fine crimped filaments of crystalline resins such as the vinylidene chloride, nylon, polyethylene, polydichlorobutadiene and other crystalline high polymeric resinous materials.

It is known that crimped, wool-like filaments may be manufactured from the crystalline resins by melt-extruding an array of filaments blowing cold air transversely of the filaments after they emerge from the die, quenching the filaments in a coolant, and cold-stretching the filaments to convert them to the oriented-crystalline state. After the cold-stretching, the tension is released from the filaments and the uneven strains therein cause the filaments to retract slightly and become crimped and curly. This relaxation is not as rapid as might be desired, apparently (in the light of the present invention) due to the clinging together of the filaments while still wet from the quenching bath; usually the retraction is not complete until the filaments have completely dried. This is both time consuming and also results in a less complete retraction and crimping of the filaments.

Accordingly, it is an object of this invention to provide a novel and improved method for the manufacture of crimped filaments from crystalline resins.

Another object is to provide a process of the type above described in which the filaments are exposed to a cold air blast, quenched, oriented, and relaxed, in which the relaxation of the filaments takes place substantially immediately upon removal of tension therefrom.

A further object is to provide such a process which will result in filaments having improved and more pronounced crimp.

The invention will be described in connection with the annexed drawing, in which:

Fig. 1 is a general diagrammatic view of the apparatus employed in this invention;

Fig. 2 is a detailed side view of the extruding die and associated equipment employed in this invention; and

Fig. 3 is a sectional view along line 3—3 of Fig. 2.

SYNOPSIS OF THE INVENTION

The above and other objects are secured, in accordance with this invention, in a process of the general type described in which a solution of a surface-active agent is distributed upon the filaments at a point subsequent to the orientation of the filaments, and in which the tension on the filaments is thereafter relaxed. The filaments relax and assume a kinked formation immediately upon release of the tension and while still wet, whereas filaments produced by otherwise similar processes, in which no surface-active material is applied, require a substantial time to assume the kinked configuration. The more rapid relaxation and kinking obtained in accordance with this invention avoids the necessity for providing detention facilities and space for the filaments while they are relaxing. Also the more rapid relaxation of the filaments obtained in accordance with this invention results in a more pronounced and resilient kinking of the filaments; the delay in relaxation incurred with

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the usual processes appears to fix the strains in the filaments so that the relaxation and kinking are relatively incomplete.

THE CRYSTALLINE RESINS

The resins employed in the manufacture of kinked filaments in accordance with this invention are of the special class known as "crystalline" resins. These resins are fusible to form very fluid melts. In the solid state, the long molecular chains of these resins tend to associate together locally to produce crystalline regions. If these resins are formed into filaments, as for instance by extrusion in the heat-softened state, and the filaments are thereafter subjected to stretching, these crystalline aggregates become aligned lengthwise in the direction of stretching and impart a high degree of strength and flexibility to the resultant filaments. Resins of this type are well known, and include for instance the vinylidene chloride resins, which are polymers of vinylidene chloride and copolymers thereof with up to 20% of other unsaturated compounds copolymerizable therewith, such for example, as vinyl chloride, vinyl fluoride, vinyl acetate, styrene, esters of acrylic and methacrylic acids such as methyl methacrylate, and ethyl methacrylate, acrylonitrile, vinyl type ethers and ketones such as methyl vinyl ether, methyl vinyl ketone and related compounds such as methyl isopropenyl ketone and the like. For a more complete list of compounds known to copolymerize with vinylidene chloride to produce crystalline resins which can be processed as described herein see Krczil "Kurzes Handbuch der Polymerisation Technik," vol. II, "Mehrstoff Polymerization," Edwards Bros. Inc., page 739, the items indented under "Vinyliden chlorid." These vinylidene chloride resins are particularly adapted for use in this invention, and outstanding results have been obtained therewith. However, the invention is also applicable to the processing into curly crimped filaments of other crystalline resins, such for instance as the nylon resins, which are high molecular weight condensation products of dicarboxylic acids with diamines or of amino acids alone or of mixtures of amino acids with dicarboxylic acids and diamines; aromatic polyester resins such as "Terylene" or "Dacron" which are condensation products of terephthalic acid and ethylene glycol; polyethylene; and polymers of 2,3-dichlorobutadiene-1,3 such as described in the patent to Kuhn 2,451,195. All of these resins may be processed in accordance with this invention into crimped fibers having excellent curliness, loft and feel.

As noted hereinabove, it is known that kinked filaments of crystalline resins may be produced by melt-extruding filaments of the resins, blowing cold air upon the freshly extruded filaments, and quenching, orienting and relaxing the filaments. The process may take various forms; for instance, air may be blown upon the filaments from all sides as they emerge from the extrusion die. It is preferable, however, to blow the air upon one side only of each filament. An advantageous means for achieving this purpose consists in an arrangement in which the extruding dies have the orifices thereof arranged in a circle in a horizontal plane with the orifices opening downwardly, normal to the plane of said circle. The emerging filaments are conducted vertically downwardly from said orifices, so that they form a substantially cylindrical array—i. e., at the orifice the filaments are all substantially parallel to each other and equidistant from and substantially parallel to an axis drawn perpendicular with the center of the circle upon which the orifices of the die are disposed. This is best seen in the drawing, which shows a conventional screw type extruding machine 10 which takes raw granular resin, melts it and forces it into an extruding head 12. As shown in Fig. 2,

3 this extruding head comprises a conical distributing passage 14 into which the molten resin is forced by the extruding machine 10, which receiving passage leads downward to the periphery of a die 16. The face of this die 16 is disposed in a horizontal plane and is provided with a large number of die orifices 18 communicating with the conical passage 14 from which they receive the molten resin under pressure and extrude it as filaments 20. These orifices are arranged in a circle having its center at the center 22 of the die 16, and it will be seen that the filaments 20 are extruded downwardly in a substantially vertical direction equidistant from an axis 24 drawn through the center 22 and perpendicular to the face of the die 16.

It will be understood that the description of the filaments 20 as being in a "cylindrical" array is approximate only: they will be deflected outwardly by cooling air as described below. Countervailing this, the filaments will usually also be drawn inwardly into a frusto-conical array, since they are gathered together in passing over the guides 26 in the quenching bath 28. For all practical purposes, however, the array of filaments may be designated "cylindrical."

Means are provided for directing a flow of cooling air or other gas radially in all directions away from the axis of the cylindrical array of extruded filaments and outwardly past the extruded filaments. This flow of cooling air is indicated by the arrows 30. As shown, this means comprises an air jet 32 mounted upon a bracket arm 33 attached to the wall of a quenching tank into which the filaments ultimately fall. This air jet is directed upwardly and concentrically of the cylindrical array of filaments so as to project a stream of air (indicated by the arrows 34) against a discoidal deflector plate 36 which is suspended from the extrusion die 16 in a position immediately below the die and coaxial of the cylindrical array of filaments. The air from the jet impinging upon this deflector plate is deflected radially outwardly and away from the axis 24 of the cylindrical array of filaments in all directions past the freshly extruded filaments 20. This air flow effects two purposes; first it rapidly chills the skin surface of the filaments and does so in a non-uniform manner, the greatest cooling effect being on that side of any given filament facing the axis of the cylindrical array of filaments thereby setting up uneven strains in the filaments; likewise, the flow of air appears to induce vibrations in the freshly extruded filaments, setting up varying strains in the filaments distributed periodically lengthwise of the filaments.

The filaments after passing the outwardly-directed flow 30 of cooling air, drop into a tank 28 of cooling water 40. The filaments thereby become chilled and completely solidified, whereby the strains induced as above discussed become preserved therein. The filaments then pass down through the bath under a set of guide pins 26 and thence upward through and out of the bath to and over a guide roll 41 to a pair of drawing rolls 42. The drawing rolls are driven at a peripheral speed in excess of the linear velocity at which the filaments are extruded; due to the resistance of the water 40 and to some extent the friction of the pins 26, a considerable tension is induced in the filaments and they are stretched to several times their extruded length during their passage through the bath and to the drawing rolls, whereby the filaments are drawn into an oriented crystalline form. The filaments pass from the drawing rolls 42 to a windup drum 44 upon which the filaments are accumulated as a skein.

A revolving roll 43 dipping into a trough 45 containing a solution or emulsion 47 of a surface-active agent bears against the windup drum so as to apply a regulated amount of the surface-active agent to the filaments as they accumulate upon the drum. The surface-active agent solution (or emulsion) may also be applied in a number of other ways. For example, a constant regulated

stream may be applied from an overhead reservoir directly onto the filament as it is wound upon drum 44. Alternatively the solution may be pumped from a reservoir and applied directly. In another modification the filament may pass over an applicator arrangement similar to the assembly 43, 45 prior to reaching drum 44. When a sufficiently large skein of filaments has accumulated on the drum, the filament is cut and transferred to a second windup roll 46 so as to be wound and accumulated upon the second windup roll. The accumulated filament is then stripped from the first windup roll. Immediately upon removal of the tension, the stripped filaments contract and, due to the uneven strains set up by the action of the air flow to which they were subjected between the extruder and the quenching bath, this contraction takes place with the formation of kinks and curls which are quite sharp and close. This is in sharp contrast to the results obtained when the identical process is carried out without the application of the surface-active agent solution 47; in such case the filaments must be hung up in a relaxed condition for several hours in order to develop the kinks.

Reverting to the second windup drum 46, an applicator roll 49 similar to the applicator roll 43 bears against the windup drum and is arranged to apply the solution 47 of the wetting agent to the filaments as they accumulate on the drum. When a convenient amount of filament has accumulated upon the drum 46, the filament is again cut and transferred back to the first windup drum 44, and the accumulated filament stripped from the drum 46, and so on, the filament being accumulated alternately upon the two drums 46.

As to the surface-active agent applied by the rolls 43 and 49, this may be any of a wide variety of such materials. For instance the ordinary alkali metal and amine fatty acid soaps such as sodium laurate, sodium myristate, sodium palmitate, potassium oleate, potassium stearate, triethanolamine stearate and the like may be employed. Likewise there may be employed sulfate- and sulfonate-group-containing surface-active agents, such as the sulfonated fatty oils and fatty acids, anionic phosphated alcohols, the alkyl sulfosuccinates, the fatty alcohol sulfates and the like, as exemplified in turkey red oil, sulfated castor oil fatty acids, sulfated coconut oil alcohols, di-(2-ethyl hexyl) sulfosuccinate, sodium dodecyl naphthalene sulfonates and the like. Other classes of surface-active agents include the fatty amines and amides, such as dodecyl amine, hexadecyl amine, stearamide, N-stearyl methylolamide, and the like. In general, it appears that those surface-active agents which have the greatest antistatic action exert the most effective action in the practice of this invention, although there appears to be no a priori reason why this should be so. Surface-active agents in general may be defined as compounds containing from 8 to 22 carbon atoms coupled to highly polar solubilizing groups such as alkali metal and ammonium carboxylate groups, sulfate half-ester groups, sulfonate groups, phosphate partial ester groups, primary, secondary, tertiary and quaternary amine groups and the like. The surface-active agent is conveniently supplied to the applicator rolls as a 0.5 to 10% (preferably 2.0%) solution in water, and a sufficient amount will be used so that the filament takes up 0.01 to 3.0% (preferably 1.0%) of surface-active agent, based on the weight of the filament.

A point which it is highly desirable to observe in the application of the surface-active agent to the filament is that it shall be withheld until after the orienting step, e. g. the cold stretching effected in advance of the drawing rolls 42. It has been found, for operational reasons, that the surface-active agents interfere with the cold-drawing behavior of the filament, causing wrapping of broken filaments on rolls 42 and slippage of the main filament band resulting in uneven orienting stretch.

With the foregoing general discussion in mind, there

are given herewith detailed examples of modes of operation of the process of this invention. All parts given are by weight.

Examples

RESIN FORMULATION

	Parts
Vinylidene chloride resin (a copolymer of 85% vinylidene chloride, 15% vinyl chloride)-----	92
Tertiary-butyl phenyl salicylate-----	5
Phenoxy propene oxide-----	2 10

SURFACE-ACTIVE AGENTS

Two-percent aqueous solutions of each of the following:

"Antistat N," an anionic phosphated alcohol mixture marketed by E. I. du Pont de Nemours and Co. 15

"Olate flakes," a relatively pure, neutral sodium oleate marketed by Armour and Co.

"Avitone A," a modified anionic sodium salt of sulfonated hydrocarbon marketed by E. I. du Pont de Nemours & Co. 20

"Polyethylene Glycol 400 Monolaurate," marketed by Kessler Chemical Co.

"Zelec DP," a "permanent" antistat having an acrylic ester resin base, marketed by E. I. du Pont de Nemours & Co. 25

"G 1300," a polyoxyethylene sorbital derivative, marketed by Atlas Powder Company.

"Profine," an anionic fatty ester based compound, marketed by Armour & Co.

"Nacconol NR," a sodium alkyl aryl sulfonate, marketed by National Aniline Division, Allied Chemical & Dye Corporation. 30

The ingredients listed under "Resin Formulation" were blended together and placed in the extruder of Figs. 1-3. A number of runs was made, using a different one of the surface-active agent solutions listed above in each of the several runs. Specifications and dimensions of the equipment, and operating conditions, were as follows: 35

EXTRUDING MACHINE 10

Make: National Rubber Machinery Co.

Size: 2 inch

Revolutions per minute of screw: 30-45 R. P. M.

Temperature of barrel: 170° C.

Temperature of extruding head: 190° C. 40

EXTRUDING DIE 16

Number of orifices 18: 250

Diameter of orifices 18: 0.0125 inch

Diameter of circle upon which the orifices are arranged: 6 inches 45

Temperature of die 16: 170° C.

Through-put of resin through each die orifice: .0012 lb. per min.

Theoretical speed of filament extruded through orifice (assuming no contraction of diameter of filament): 132 ft. per min. 50

FLOW OF COOLING AIR

Diameter of air jet orifice: 3/8 inch

Volume of air to jet: 125 cubic ft. free air per min. 60

Distance from nozzle tip to deflector plate: 3/8 inch

Approximate velocity of air flow outward past the filament at a point 3/4 inch below die: 16 ft. per sec.

QUENCHING, ORIENTATION AND TAKE-OFF

Temperature of water quenching bath 40: 15° C. 65

Distance of surface of quenching bath 40 below the die: 4 inches

Length of filament immersed in bath 40: 6 ft.

Peripheral speed of drawing rolls 42: 675 ft. per min. 70

PROPERTIES OF THE CURLY FILAMENT

Diameter of individual filament: .0018 inch

Tensile strength: 26,100 pounds per square inch

Kinks in filament per inch of length of filament: 8 75

Approximate height of kinks from one extreme to other: .125 inch

Elongation at break: 13%

The filaments in all runs relaxed immediately upon removal from the windup drums 44 and 46, and formed tight kinks and curls immediately. In comparable runs in which no surface-active material was applied, it was necessary to hang the filaments in a relaxed condition for from 1 to 24 hours in order to develop the optimum curl and kink in the filaments.

The filaments produced as above described had excellent feel, handle and loft comparable to naturally kinked fibers such as wool, alpaca and the like. The filaments were spun into yarn which was woven as the pile of an Axminster carpet, and in this application exhibited a very desirable loft and showed good resistance to ordinary wear. The filaments were likewise found highly suitable when spun into yarns for knitting into garments, and for ordinary woven fabric.

From the foregoing general discussion and detailed description of the operation of the invention, it will be evident that this invention provides a novel process and apparatus for the production of curly, kinked fine filaments of oriented crystalline resins. The process can be carried out with a minimum of skilled attendance and the apparatus required is both simple and inexpensive. The process avoids the necessity for providing a large detention space for hanging the filament to develop the curl thereof, which has heretofore been necessary in processes of the general type.

What is claimed is:

1. Process which comprises melt-extruding an array of filaments of an orientable crystalline thermoplastic resin, chilling the filaments when freshly extruded by blowing air thereupon, quenching said filaments in a cooling medium, subjecting the filaments to cold drawing in the absence of a surface-active agent, applying a surface-active agent to the filaments while still under tension, and thereafter removing tension from the filaments to permit them to relax into a crimped configuration, the surface-active agent applied to the filaments being a compound containing from 8 to 22 carbon atoms coupled to a highly polar solubilizing group selected from the group consisting of alkali metal carboxylate groups, ammonium carboxylate groups, sulfate half-ester groups, sulfonate groups, phosphate partial ester groups, primary amine groups, secondary amine groups, tertiary amine groups and quaternary amine groups. 45

2. Process which comprises melt-extruding an array of filaments of a vinylidene chloride resin, chilling the filaments when freshly extruded by blowing air thereupon, quenching said filaments in a cooling medium, subjecting the filaments to cold drawing in the absence of a surface-active agent, winding the cold-drawn filaments upon a collecting drum, applying a surface-active agent to the filaments while still under tension and wound upon said collecting drum, and stripping said filaments from said drum so as to remove tension from the filaments to permit them to relax into a crimped configuration, the surface-active agent applied to the filaments being a compound containing from 8 to 22 carbon atoms coupled to a highly polar solubilizing group selected from the group consisting of alkali metal carboxylate groups, ammonium carboxylate groups, sulfate half-ester groups, sulfonate groups, phosphate partial ester groups, primary amine groups, secondary amine groups, tertiary amine groups and quaternary amine groups. 60

3. Process which comprises melt-extruding an array of filaments of an orientable crystalline thermoplastic resin, chilling the filaments when freshly extruded by blowing air thereupon, quenching said filaments in a cooling medium, subjecting the filaments to cold drawing in the absence of a surface-active agent, winding the cold-drawn filaments upon a collecting drum, applying a 65

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phosphated alcohol surface-active agent containing from 8 to 22 carbon atoms to the filaments while still under tension and wound upon said collecting drum, and stripping said filaments from said drum so as to remove tension from the filaments to permit them to relax into a crimped configuration.

4. Process which comprises melt-extruding an array of filaments of an orientable crystalline thermoplastic resin, chilling the filaments when freshly extruded by blowing air thereupon, quenching said filaments in a cooling medium, subjecting the filaments to cold drawing in the absence of a surface-active agent, winding the cold-drawn filaments upon a collecting drum, applying an alkali metal soap containing from 8 to 22 carbon atoms to the filaments while still under tension and wound upon said collecting drum, and stripping said filaments from said drum so as to remove tension from the filaments to permit them to relax into a crimped configuration.

5. Process which comprises melt-extruding an array of filaments of a vinylidene chloride resin, chilling the filaments when freshly extruded by blowing air thereupon, quenching said filaments in a cooling medium, subjecting the filaments to cold drawing in the absence of a surface-active agent, winding the cold-drawn filaments upon a collecting drum, applying a phosphated alcohol

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surface-active agent containing from 8 to 22 carbon atoms to the filaments while still under tension and wound upon said collecting drum, and stripping said filaments from said drum so as to remove tension from the filaments to permit them to relax into a crimped configuration.

6. Process which comprises melt-extruding an array of filaments of a vinylidene chloride resin, chilling the filaments when freshly extruded by blowing air thereupon, quenching said filaments in a cooling medium, subjecting the filaments to cold drawing in the absence of a surface-active agent, winding the cold-drawn filaments upon a collecting drum, applying an alkali metal soap containing from 8 to 22 carbon atoms to the filaments while still under tension and wound upon said collecting drum, and stripping said filaments from said drum so as to remove tension from the filaments to permit them to relax into a crimped configuration.

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