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NYLON PELLETING PROCESS

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FIG. 2.

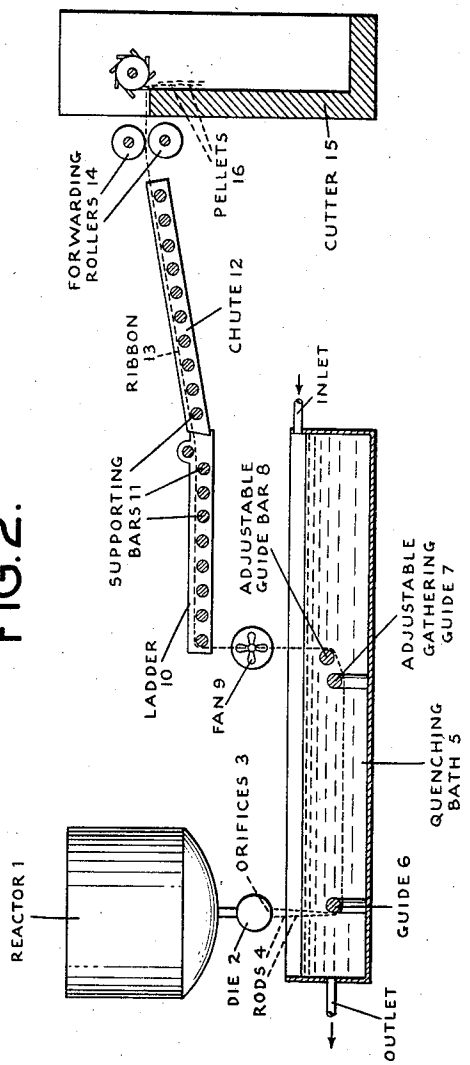
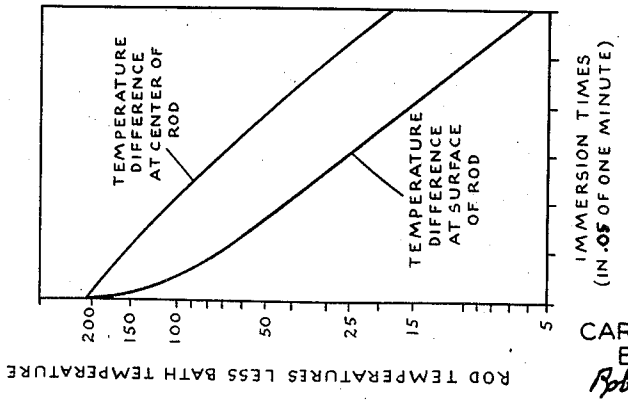


FIG. 1.



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NYLON PELLETING PROCESS

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2 Claims. (Cl. 18—47.5)

This invention relates to pelleting nylons of the types useful for spinning and/or molding, i. e. the fusible, filament-forming, molecularly orientable types of nylons. Filament-forming, molecularly orientable epsilon caprolactam polymer is exemplary of the materials to which the invention applies.

In accordance with this invention, I extrude molten filament-forming molecularly orientable nylon at a temperature in the melt of at least about the flow temperature as defined below into a water quenching bath, in the form of a plurality of separate, parallel, generally circular rods of average diameter between about 0.02 and about 0.2 inch; then when the rods have cooled to average temperature in the range not above about the flow temperature less 15° C. and not below about 110° C. I withdraw the rods still in parallel arrangement from the quench bath and guide the rods toward a forwarding device such as a pair of pinch rolls and contact the rods with a gaseous drying medium as they move toward the forwarding device. At a point in the travel of the rods through the quench bath and up to the forwarding device I gather the parallel rods into side-by-side contact by use of a guide, when their average surface temperature is below the tacky point at which they adhere to the guide and while they remain sufficiently hot to adhere together along lines where mutual contact is maintained forming a ribbon wherein the individual identity of the rods is preserved and which when cool is separable into constituent rods by a sharp twist.

I pass the ribbon thus formed, after it is dry on the surface, through the forwarding device under tension insufficient to molecularly orient the nylon and at surface ribbon temperature of at least about 40° C. Then I shear off in transverse succession the ends of the rods as the ribbon advances, so that the ribbon is twisted by the effect of the cutting stroke and adhesions between the ends of the rods in the ribbon are broken at the same time that the rod ends are being sheared off. The average temperature of the resulting freshly cut pellets, in accordance with my invention, is not above about 110° C. By my process exceptionally uniform, generally cylindrical pellets are formed with few or no adhesions between pellets; and any adhesions occurring are so weak as to be easily broken in a breaker.

Important phases of my process are:

(1) establishing the side-by-side lines of adhesion to make a ribbon;

(2) passing the water quenched ribbon at average temperature initially at least about 110° C. through a gaseous drying medium;

(3) maintaining surface temperature of the ribbon entering the forwarding device of at least about 40° C.; and

(4) cutting the ribbon at average ribbon temperature not above about 110° C.

Establishing side-by-side adhesion

Side-by-side lines of adhesion are established in my process by bringing generally circular rods of diameter in

the range between about 0.02 and 0.2 inch, average over all the rods, into side-by-side contact after cooling to surface temperatures as described below and while still hot in the interior. The broad objective is to cool the surface of the rods at least to a temperature below the tacky point so that the rod surfaces are solid and can contact metal guides without sticking thereto and can, when in side-by-side contact, maintain their individual identity. By gathering the rods together into a ribbon of one or more layers of parallel rods in side-by-side contact while the rods are hot, and continuing to cool the surface of the rods from the temperature of gathering, the rods became sufficiently softened by interior heat at their points of contact, but not elsewhere, to develop adhesion at the lines of mutual contact while maintaining their identity.

The maximum surface temperature suitable for the gathering step is usually about 15° C. below the flow temperature of the particular nylon being processed. Flow temperatures can be determined by the method of the American Society for Testing Materials designated ASTM Method D–569–48. For the particular polycaprolactam nylon used in the example below the flow temperature is about 215° C. and the maximum suitable surface temperature when the gathering step of my process is performed is about 200° C. Lower surface temperatures than the above maximum are permissible at the moment of gathering in accordance with my process but the rods must retain sufficient heat in the interior so that when the cooling effect at the surface is reduced by the mutual contact of the rods at the lines of mutual contact, the nylon will be reheated sufficiently at these lines by heat from the interior to develop adhesion along these lines of mutual contact.

The surface and average temperautres of the rods at the gathering point during actual operation are not readily accessible to direct measurement, but correct adjustment of the gathering point in my process is easily obtainable by varying the distance of the gathering guide from the point of entry of the rods into the quench bath while keeping the other process variables constant. At a proper point of adjustment of the gathering guide the ribbon approaching the forwarding device is an integral ribbon of mutually adhering parallel rods, the individual rods of which are readily separable by a sharp twist of the ribbon. The point of proper adjustment is not delicately sensitive to the other conditions and can usually be left unchanged for many hours of running time.

The surface temperature of the rods and the heat flow from the interior to the surface of the rods will depend, among other things, on diameter of the rods, temperature of the quench bath, and time in the quench bath. Quench bath temperatures can be in a wide range such as 10° C.–90° C., quench times generally being longer the higher the quench bath temperature. Preferred surface temperatures at the gathering point have been found to be about 175° C. as calculated from measurement of bath temperatures at that point and time in the bath at that point with the aid of the curve shown in Fig. 1 of the drawings and discussed in more detail below.

The rods can be gathered into a ribbon with a single layer; a special advantage of my process is that the ribbon can be gathered in two or more layers by merely adjusting the width of the gathering guide. A multiple layer ribbon may be more economical to run depending upon the design of the cutter.

The gathering point can be either in the quench bath or in the line of travel of the rods in contact with gaseous drying medium so long as the necessary temperature conditions above described are met. Using extrusion speeds in the ordinary range of about 100 ft.–200 ft. per minute and with bath temperatures of about 40°–60° C. the gathering point will ordinarily be set at about 1 ft.–

10 ft. from the point of entry of the rods into the quenching bath, immersion time in the bath being about 1–5 seconds.

Drying period

A period of contact of gaseous drying medium with the rods initially at average temperature of at least about 110° C. is of importance in my process. Thus the average temperature of the rods when they emerge from the quench bath should be at least about 110° C., but not so high that the nylon becomes discolored as it may do if maintained at temperatures such as 200° C. in air.

Fig. 1 of the drawings herein shows two curves approximating the relation between quench bath temperature, time in the quench bath, surface temperature and center tempearture of rods of epsilon caprolactam polymer of 0.1 inch diameter. From the curve it can be seen, for example, that using a quench bath at relatively elevated temperature such as 90° C. the maximum permissible time in the bath will be about 15 seconds and will be shorter for lower bath temperatures, other things being equal, e. g. about 10 seconds for 60° C. bath temperature.

The travel of the ribbon as above described at temperatures of at least about 110° C. in contact with gaseous drying medium appears to allow relieving stresses in the ribbon which otherwise tend to warp it by the time it reaches the forwarding device. The relief of stresses is effected in my process by softening action of elevated temperatures above about 110° C. coupled with presence of hot water and steam in the rods, derived from the quenching bath.

The time of travel of the rods after leaving the quench bath should be sufficiently long, and the drying should be sufficiently gradual, so that stresses are allowed to be relieved by the above influences. The minimum time is about 2 seconds.

The rods approaching the pelletizer should be surface dried. The level of overall drying reached at the pelletizer can be from about equilibrium moisture content with the ambient air at pelletizing temperature down to a low value such as about 0.5% moisture by weight. Preferred values are in the range from about 0.5–2.0% moisture by weight. Drying to low moisture is advantageous when the pelleted product is to be used without further washing, e. g. when a pelleted polycaprolactam containing several percent monomer by weight is desired.

Air is usually convenient as the gaseous drying medium of my process but other drying media in gas phase can be used, e. g. carbon dioxide, nitrogen, superheated steam, etc. Preferably the drying gas is blown across the rods near the point where the rods emerge from the water bath to blow off drops of water. The rods are suitably brought out of the water bath at a steep angle to permit draining of water therefrom.

The rods should travel substantially free of orienting tension although it is permissible to attenuate them by pulling while still in a highly plastic condition near the die. The tension can be controlled by interadjusting the extrusion speed and the take-up speed.

Minimum temperature during drying

I have found that the surface temperature of the rods when they complete the drying step of my process should be not below about 40° C. When lower temperatures are reached before the forwarding device undue thermal stresses are introduced in the rods, which tend to cause uneven feed thereof through the forwarding device. The surface temperatures of the rods at any point of the drying step can be measured with sufficient accuracy by measuring the temperature of a suitably designed bar over which the rods travel, making correction for heat losses through the bar.

Ribbon temperature during cutting

I have found that a nylon ribbon of lightly adhering rods as formed in my process can be cleanly cut into pellets when its average temperature is not above about 110° C., preferably about 90° C. down to about 50° C. When average temperatures higher than the above are maintained through the cutting stage, many of the cut pellets will have short extensions or tails at the cut edge, pulled out before the cut was completed. These tails break off and form fines which are undesirable in subsequent operations. Especially preferred temperatures for cutting epsilon caprolactam nylon are about 55°–60° C. The average ribbon temperatures at the time of cutting as given herein are the temperatures measured in the interior of a pile of freshly cut pellets, so that the actual average temperature of the ribbon at the moment it is cut is probably a degree or two higher than herein stated.

The accompanying Fig. 2 is a diagrammatical flow sheet illustrating the process of my invention. Referring to Fig. 2, my process can be described as follows: A melt of polycaprolactam in reactor **1** at a temperature of about 260° C. is forced by pressure of inert gas into die **2** and extruded through two lines of orifices **3** in the form of rods **4** which enter quenching bath **5** and pass under guide **6**. The rods then travel through the water of the bath to adjustable gathering guide **7** and thence under adjustable guide bar **8** which is set to determine the point of emergence of the rods from the water bath. Near this point of emergence a fan **9** blows air across the moving ribbon to blow off drops of water. The ribbon travels to ladder **10** and over the supporting bars **11** of the ladder which prevent the ribbon from sagging and give access to drying gas, e. g. air. A chute **12** carries the resulting surface dry ribbon **13** to forwarding rollers **14** which supply the ribbon to a rotary cutter **15**. The cutter shears off the advancing ends of the rods in succession thus twisting the ribbon and breaking the adhesions between the rods whereby cylindrical pellets **16** are formed. Compressive forces, too, separate the rods.

A specific example of the best mode of carrying out my invention contemplated by me is as follows:

Using apparatus of the general type indicated in Fig. 2 of the drawings, 70 rods, averaging 0.1 in. in diameter were extruded at 156 ft. per minute into a water quench bath maintained at 50° C. The total travel distance from the entry point of the rods in the quench bath to the forwarding rolls of the pelletizer was about 50 ft. 2 ft. 3 in. from the die in the quench bath the rods were passed under a yoke with the curvature set to keep the rods separate. 3 ft. 6 in. from the die in the quench bath the 70 rods were again passed under a yoke set to keep the rods separate. 4 ft. 7 in. from the die the separate rods were passed under a straight stainless bar then brought out of the quench water onto a yoke 7 ft. 8 in. from the die. The curvature of this yoke was set to allow the separate rods to be fused into a ribbon. The average temperature of the emerging rods was about 150° C. calculated as a logarithmic average from the curves of Fig. 1, and the surface temperature was about 120° C. as calculated from Fig. 1.

Air was directed on the top and bottom of the polymer ribbon to remove excess surface water.

The temperature of the polymer pellets at the pelletizer was 55°–62° C. A clean pellet cut was obtained, with fusing of adjacent pellets at a minimum. Adhesions between pellets observed at the pelletizer were separated very easily. Samples of pellets which had gone through the breaker revealed no fused pellets. The pellets were uniform, free of fines, and showed no discoloration from the exposure of the ribbon to air.

The duration of the run was 5 hrs. 40 min. Continuous operation was maintained throughout.

In tests using extrusion rates of about 130 ft. per minute and bath temperature of about 40° C., the rods were brought out of the bath in ribbon form after about 3–4 ft. of travel; they were gathered into a ribbon in the bath about 1 ft. ahead of the point of emergence from

the bath. These variations in the mode of operation of the above example produced essentially the same results as those of the above example.

Bath temperatures cited herein are the temperatures measured at the end of the bath below the extrusion die. The bath temperatures may be about 5° C. lower at the point where the water enters, using a countercurrent flow of water as indicated in the drawing Fig. 2.

Advantages of my process include its requiring only very simple, readily available equipment; its relieving stresses in the ribbon fed to the pelletizer whereby light adhesion between rods suffices to maintain an integral ribbon; the consequent production of uniform, easily separable pellets; the freedom of the pellets from fines; and high production rates achieved in continuous operation with little or no attention from an operator.

While my process has been particularly discussed and illustrated above with reference to epsilon caprolactam polymer nylon, the process is similarly applicable to the other fusible, filament-forming, molecularly orientable nylons such as hexamethylene adipamide polymer; hexamethylene sebacamide polymer; interpolymers of hexamethylene diamine, adipic acid, sebacic acid, and/or caprolactam; etc.

I claim:

1. Process for pelleting nylon which comprises extruding molten, filament-forming, molecularly orientable nylon, at a temperature in the melt of at least about the flow temperature, into a water quench bath in the form of a plurality of separate, parallel, generally circular rods of average diameter about 0.02 to about 0.2 inch; withdrawing the rods from said bath when cooled to average temperature not above about the flow temperature less 15° C. and not below about 110° C.; guiding said withdrawn rods toward a forwarding device while contacting the rods with a gaseous drying medium; gathering said rods into side-by-side contact by use of a guide at a point in their travel through the quench bath and up to the forwarding device when their average surface temperature is below the tacky point at which they adhere to the guide and while they remain sufficiently hot to adhere together along lines where mutual contact is maintained forming a ribbon in which the individual identity of the rods is preserved and which when cool is separable into constituent rods by a sharp twist; after the ribbon thus formed is dry on the surface, passing it through the forwarding device under substantially non-orienting tension and at surface temperature of at least about 40° C.; and shearing off in transverse succession the ends of the rods, as the ribbon advances beyond the forwarding device, at average temperature of the resulting pellets not above about 110° C., simultaneously breaking adhesions between the ends of the rods, as they are sheared off, by twisting effect of the cutting stroke on the advancing ribbon; thereby producing generally cylindrical pellets.

2. Process as defined in claim 1, wherein the nylon pelleted is epsilon caprolactam polymer; the quench bath temperature is in the range between 40° C. and 60° C.; the extrusion rate is in the range between 100 ft. per minute and 200 ft. per minute; the total travel of the rods is about 50 ft.; the gathering point is between about 1 ft. and about 10 ft. from the point of entrance of the rods into the quench bath; the time of travel of the rods in the quench bath is not more than 10 seconds; the time of travel of the rods in contact with gaseous drying medium is at least about 2 seconds; the drying is to moisture content not above about 2% by weight; and the cutting is at average temperatures in the range between about 50° C. and about 90° C. as measured in a pile of freshly cut pellets.

References Cited in the file of this patent

UNITED STATES PATENTS

2,464,746	Gering	Mar. 15, 1949
2,746,086	Vickers	May 22, 1956

FOREIGN PATENTS

733,720	Great Britain	July 20, 1955

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,859,479 November 11, 1958

Carl R. Eckardt

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the grant, lines 2 and 3, for "assignor to Allied Chemical & Dye Corporation, of New York, N. Y., a corporation of New York," read -- assignor to Allied Chemical Corporation, a corporation of New York, --; line 12, for "Allied Chemical & Dye Corporation, its successors" read -- Allied Chemical Corporation, its successors --; in the heading to the printed specification, lines 3 to 5, for "assignor to Allied Chemical & Dye Corporation, New York, N. Y., a corporation of New York" read -- assignor to Allied Chemical Corporation, a corporation of New York --; column 2, line 12, for "became" read -- become --; line 34, for "temperatures" read -- temperatures; column 3, line 16, for "tempearture" read -- temperature --.

Signed and sealed this 31st day of March 1959.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents