

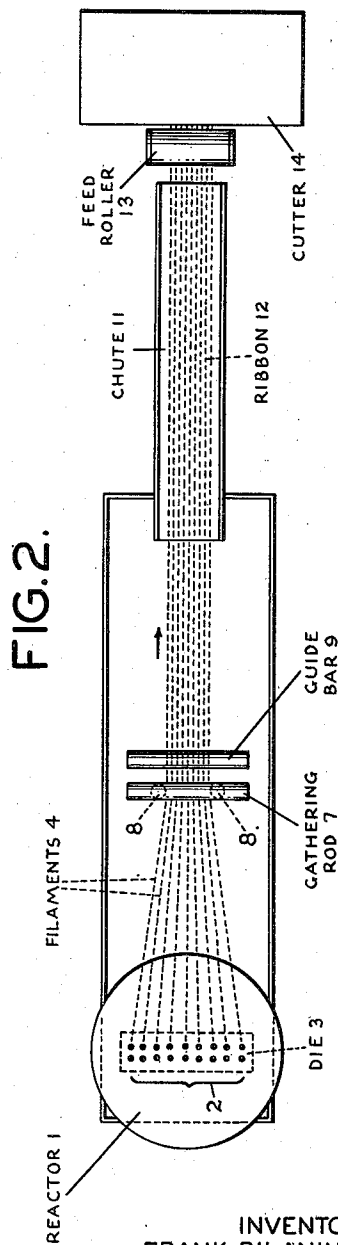
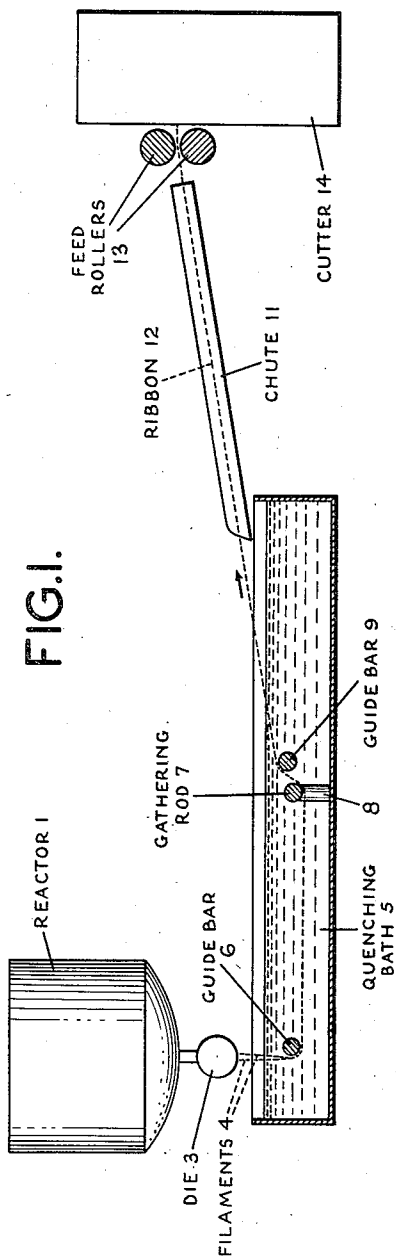
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GRANULATION OF FUSIBLE, MOLECULARLY ORIENTABLE POLYMERS

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GRANULATION OF FUSIBLE, MOLECULARLY ORIENTABLE POLYMERS

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This invention relates to conversion of a fusible, molecularly orientable polymer into granules suitable for use as molding powder, as starting material for a melt spinning process, etc. The process is applicable generally to polymers having the physical properties of being fusible in substantially pure condition, being extrudable from the melt, having a crystalline or micro-crystalline character and being molecularly orientable upon stretching.

It has been proposed to convert such polymers into chips for use in melt spinning processes by extruding a strip of polymer from the melt, cooling the strip and cutting the strip into suitably sized squares by use of a punch press. In this proposed process the strip must be kept cool enough to prevent stickiness and at the same time kept hot enough to reduce irregular breakage and shattering which result in nonuniformity and fines.

The present inventors have discovered and developed a process for granulation of fusible, molecularly orientable organic polymers which allows cutting very uniform granules over a wide temperature range without producing fines such as result from brittleness of the polymer cut by the above cited process. This new process permits high production rates and permits utilization of a high speed rotary cutter.

In the new process molten organic polymer is extruded as separate, rounded, essentially uniform, essentially nonmolecularly-oriented filaments into water or like inert liquid. Then the submerged filaments are gathered into side by side contact in one or more banks, forming a ribbon, while still at average temperature in the plastic range as defined below but not until the filament surface has cooled below the "weld" temperature, i. e. the temperature at which fusion occurs and coalescence between filaments will develop immediately at all adjacent areas.

The cooling liquid extracts heat from the exposed surfaces of the resulting ribbon of filaments so as to keep at least the major portion of these exposed surfaces below the weld temperature; but heat flow from the cores of the individual filaments brings their laterally contacting surfaces into the plastic range of temperature, i. e. in the temperature range in which measurable permanent deformation of the polymer surface will occur under the pressure being exerted laterally to gather the extruded filaments, within the time in which the filaments are held in contact before being cut.

Travel through the cooling liquid is continued at least until the ribbon has solidified into a connected structure, preferably until temperature at the points of contact of the filaments has been reduced below the plastic range. Preferably the average temperature remains above the boiling point of the cooling liquid which will then evaporate off from the finished granules.

Thereby a lateral bond is formed at points of contact between the individual filaments, having a strength which depends upon the average temperature of the filaments at the time they are brought into contact (and thus upon the quench time, between extrusion and gathering); upon

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the lateral pressure exerted in gathering the filaments; and upon the time of contact of the filaments while in the plastic range of temperature.

The resulting filament ribbon is drawn through the nip of a pair of feed rolls, and the advancing end of the ribbon is severed by a cutter preferably with a slicing action, e. g. by a high speed rotary cutter. The speed of the feed rolls should be coordinated with the rate of filament extrusion so that no tension develops sufficient to substantially molecularly orient the filaments by stretching. Unoriented filaments cut and break apart better than oriented filaments; hence orientation is avoided.

The stroke of the cutter breaks the lateral bonds between individual filaments beyond the cutting block at the same time as it cuts them transversely, thereby forming granules. Because the filaments adhere laterally at least until they pass through the feed rolls, the problem of uneven feed of individual filaments to the cutter is solved.

It is not necessary that all the filaments adhere laterally throughout the full length of the ribbon emerging from the cooling bath; it is only necessary that lateral bonds occur at such intervals that the ribbon maintains its integrity until it passes through the feed rolls. The filaments must substantially maintain their individual identity throughout their length and must be bonded weakly enough so as to form uniform granules when severed. It is a comparatively easy matter in the present process to adjust the cooling liquid temperature, the gathering force, and the contact time with the cooling liquid, before and after gathering, to produce a ribbon of adhering filaments with the characteristics above specified.

The accompanying drawing is a flow sheet illustrating the process of the invention. Figure 1 is a side view of suitable apparatus, diagrammatically illustrated. Figure 2 is a top view of the same apparatus.

In the operation of the process illustrated in the drawing molten polymer is extruded from reactor 1 through the orifices 2 in the die 3. A single row of orifices forming one bank of filaments can be used but in preferred operations in accordance with the present process two or three rows of orifices are used.

The filaments 4 enter cooling bath 5, suitably passing around guide bar 6 which is set at a point where the filament surface has been cooled below the weld temperature.

From guide bar 6 the filaments travel through the cooling bath to the gathering rod 7 which suitably has the form of a doorway or inverted U. The position of gathering rod 7 is adjustable along the length of the cooling bath, and the separation between its vertical side bars 8 is adjustable. Top and bottom pressure on the ribbon of filaments as they are gathered is adjustable by raising and lowering guide bar 9, which is associated with gathering rod 7 a short distance away in the line of movement of the filaments. It is shown as following the gathering rod, but it can also precede the gathering rod.

The filaments travel through the cooling liquid until they are solidified into a connected ribbon structure. In the drawing the path of travel through the liquid includes a chute 11 which can be moved lengthwise of the cooling bath to vary the length of travel of the filaments through the cooling liquid after the gathering point. Alternatively a guide bar beyond the gathering rod, similar to guide bar 6, can be used to fix the length of travel of the ribbon 12 of gathered filaments in the cooling liquid after the gathering point.

The solidified ribbon passes through feed rolls 13 into cutter 14, which is a conventional cutter such as a rotary cutter. The stroke of the cutter slices across the advancing end of the ribbon and cuts a strip which falls into granules under the force of the cutting stroke. Any granules which remain adhering together can easily

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be separated, when operation is properly controlled as outlined below, by a short tumbling or whirling or like operation.

The operation of the present process can very easily be controlled, since an easily adjusted variable of dominant influence is the time of contact with the bath before the gathering point, i. e. the time which can be called the quench time. Under proper operation the ends of the individual rods forming the connected ribbon drop off largely or entirely separate, as granules, when the ribbon is cut. In the event that a considerable proportion of these ends adhere together after cutting, a longer quench time is given by moving the gathering rod further from the extrusion die, while maintaining the same extrusion temperature, extrusion rate, and cooling bath temperature as before. This adjustment is effective; it is not, however, unduly delicate and can usually be left unchanged even though there may be minor variations in operating conditions.

Conversely, if the quench time is too long, individual filaments will become separated from the ribbon structure ahead of the feed rolls for the cutter; and will tend to buckle and cross over, producing irregular feed or jamming the feed rolls. This condition calls for shorter quench time, which is given by moving the gathering rod nearer to the extrusion die. With the bath water at ordinary cold water temperatures of about 10°–25° C., the quench time will usually be about 1–2 seconds.

The gathering pressure exerted laterally must be sufficient to bring the extruded filaments into a ribbon. This ribbon can consist of a single bank of filaments but preferably will be two or three banks thick. The number of banks is fixed by setting the width of the gathering rod equal to the width of ribbon which will be formed when the extruded filaments are in light side-by-side contact in the desired number of banks. As take up speeds to the cutter increase, there will be a tendency to attenuate the filaments as they issue, in fused or plastic condition, from the die, reducing their diameters and the width of the ribbon formed. This can be compensated by increasing extrusion pressures to the point where the filaments come through the die at the desired diameter and travel smoothly, without piling up below the extrusion die.

The following are specific examples illustrative of the present process, but the herein claimed invention is not to be understood as limited to the details of the examples.

Example 1.—Molten molecularly orientable polycaprolactam under carbon dioxide pressure of 17 p. s. i. gauge was extruded as round filaments of rods downward into a water bath from a horizontal cylindrical die transverse to the water bath, having along its down side a row of 50 round orifices, the holes being about $\frac{3}{32}$ inch in diameter and about $\frac{1}{4}$ inch apart. Two and three rows of holes instead of only one row can be used, giving increased production and generally better handling going through the cutter. The holes suitably number 25–75 per row. Advantageously, to prevent filaments from clinging to the die, the orifices can be given the external form of pyramids say $\frac{1}{16}$ inch high. These improvements are illustrated in Example 2 below.

The water bath tank had an overflow pipe at the near end and a feed chute at the far end from the extrusion die.

The temperature of the molten polycaprolactam was about 250° C. The surface of the water cooling bath was about $\frac{1}{2}$ inch below the die face. The temperature of the water entering the bath was about 15° C. and the temperature of the water bath was about 20° C. The first guide bar contacted by the filaments was about 8 inches below the surface of the water in the cooling bath, at which point the filament surfaces had been superficially hardened, by cooling below the weld temperature of about 215° C.

The gathering rod was located 22 inches from the first guide rod. The average temperature of the filaments

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passing under the gathering rod was about 200° C. The side bars were separated by about the sum of the filament diameters, i. e. about 3 inches. The contacting surfaces of the filaments at this temperature became heated to the plastic range of temperature. They stuck to each other at areas of mutual contact but would not stick to a good heat conductor in contact with a cooling liquid such as the liquid in the cooling bath. The above mentioned guide bar and the gathering rod and associated bars were made of metal whereby sticking thereto of the filaments was avoided.

The horizontal guide bar associated with the gathering rod was about one inch farther along the cooling bath than the gathering rod and was placed slightly higher than the top of the gathering rod whereby a flat ribbon of filaments was formed.

The ribbon of filaments passed out of the cooling bath and up a chute about 15 feet long at a speed of travel of 78 feet per minute to the feed rolls; thus the quench time, between extrusion and gathering, was about 2 seconds. All filaments ran smoothly and evenly as an integral ribbon through the feed rolls into a conventional rotary knife cutter rotating at 1080 R. P. M.

The slicing strokes of the knives upon the end of the ribbon passing over the cutting block of the cutter formed cylindrical pellets of about $\frac{1}{16}$ inch diameter and about $\frac{1}{16}$ inch long. These pellets or granules were free flowing, very uniform, and substantially free of fines. Most were completely separate. A short whirling operation separated substantially all of the granules one from another.

The lateral separation between the vertical side bars of the gathering rod was adjusted to bring all the filaments into light side-by-side contact in one ribbon. The distance between the first guide bar and the gathering rod was adjusted relative to the temperature of the cooling bath and the speed of travel of the filaments so that the emerging filament-ribbon maintained its integrity, going through the feed rollers while all filaments substantially maintained their individual identity throughout the length of the ribbon, rather than forming a corrugated sheet. The lateral adhesion of one filament to the next in the ribbon approaching the feed rolls was interrupted at random intervals but all filaments were laterally joined in one integral ribbon structure. The lateral bonds between filaments in the ribbon approaching the feed rolls could be broken by a sudden twist such as produced by the slicing action of blades in a rotary cutter set at a small vertical angle such as 1° to the transverse line across the ribbon of filaments, whereby the filaments are cut sequentially rather than simultaneously.

Example 2.—Using the apparatus described in Example 1, molten molecularly orientable polycaprolactam under carbon dioxide pressure of 35 p. s. i. gauge was extruded from a horizontal rectangular die transverse to the water bath. This die had on its lower face two rows each with 25 round orifices of pyramidal external form. The holes were $\frac{1}{4}$ inch in diameter, and $\frac{1}{4}$ inch apart.

The extruded filaments or rods, somewhat attenuated as they moved away from the die, passed downward and under a guide bar approximately 8 inches below the surface of the cooling water. At this point some filaments were moving together in 2 banks but most were spread out, more or less at random, in a single bank. The gathering rod located 16 inches from the guide bar gathered the filaments into a ribbon 5 inches in width. It cooperated with a horizontal bar placed 3 inches beyond the gathering rod and slightly higher than the top bar thereof, to bring the filaments into a 2-bank ribbon as they passed under the gathering bar and over the horizontal bar. Speed of travel of the ribbon was 150 feet per minute. The quench time, between extrusion and gathering of the filaments, was accordingly about 1 second.

The ribbon, which was like that of Example 1 except for being 2 banks thick, was fed by rolls into a conven-

tional rotary knife cutter rotating at 1300 R. P. M. This cutting followed by a short tumbling operation produced separate cylindrical granules like those of Example 1, about 0.1 inch long and 0.2 inch in diameter.

Dimensions of the apparatus and values of other variables given in the examples can be varied within reasonable ranges. For instance, the distance between the orifices can be from about $\frac{1}{32}$ inch to about $\frac{1}{2}$ inch; and the extrusion rate of the filaments can vary over a range such as 10 feet per minute up to 200 feet per minute and faster. The practical upper limitation on extrusion rate is the take-up speed of available cutters.

When polycaprolactam is the polymer which is being granulated, the extrusion temperature should be at least about 210° C. and can be as high as about 300° C. A preferred range is about 225°–275° C. The cooling water temperature can be from freezing to about 90° C. The temperature of the contacting surfaces of the polycaprolactam filaments at the gathering rod must be below the weld temperature of about 215°–225° C.

The average filament temperature must be in the plastic range in order to form a ribbon of filaments held together by weak lateral bonds while essentially preserving the individual identity of all filaments. In general this means that the average temperature of the filaments when gathered should be below but within 50° C. of the fusion temperature. An average filament temperature during gathering not above the weld temperature is desirable since such average temperature assures that the contacting filament surfaces will be generally below the weld temperature. Preferably the average filament temperature during gathering is in the range between about 190° C. and about 210° C. for polycaprolactam polymer.

The invention claimed is:

1. Process for forming granules from fusible, molecularly orientable thermoplastic organic polymer which comprises extruding from a melt of said polymer separate, rounded, essentially uniform, essentially nonmolecularly-oriented filaments; passing said filaments into an inert cooling liquid; cooling said filaments to surface temperature below the temperature at which fusion occurs and coalescence between filaments will develop immediately

at all adjacent areas but with average temperature remaining in the range in which the filaments adhere while maintaining their individual identity; then immediately gatherings said filaments into side-by-side contact in a ribbon wherein each filament substantially maintains its individual identity throughout its length; continuing to extract heat from the surface of the ribbon except the laterally contacting surfaces of the filaments, at a rate to allow the ribbon to solidify into a connected structure; then passing the ribbon under nonorienting tension to a cutter which severs the free end thereof and breaks the severed strip into separate granules at the lateral bonds between filament ends.

2. Process as defined in claim 1 wherein polycaprolactam is the polymer employed; extrusion is at a temperature in the range between about 210° C. and about 300° C.; the cooling liquid is water at about 0°–90° C.; and the filaments are gathered after a quench time of about 1–2 seconds between extrusion and gathering.

3. Process as defined in claim 2 wherein the filaments are extruded from 2 banks of holes spaced about $\frac{1}{32}$ – $\frac{1}{2}$ inch apart; the filaments are gathered into a ribbon 2 banks thick; and the average temperature of the filaments when gathered is about 200° C.

4. Process as defined in claim 2 wherein cooling liquid temperature, gathering force, and contact time with the cooling liquid are interadjusted so that the lateral adhesion of one filament to the next in the ribbon approaching the feed rolls is interrupted at random intervals but all filaments are laterally joined in one integral ribbon structure.

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