Aug. 17, 1943. P. R. AUSTIN 2,327,087

MANUFACTURE OF STAPLE FIBERS

Filed Dec. 18, 1940

Fig. 1.

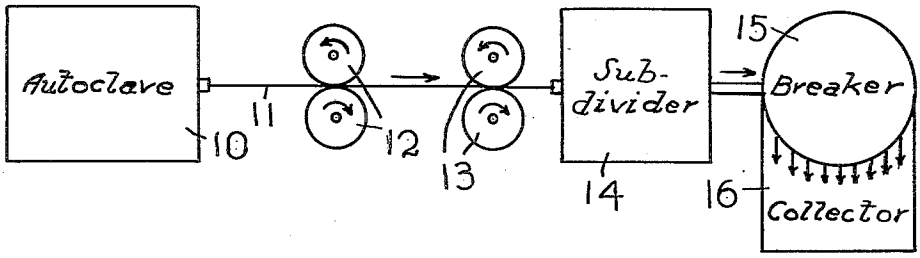


Fig. 2.

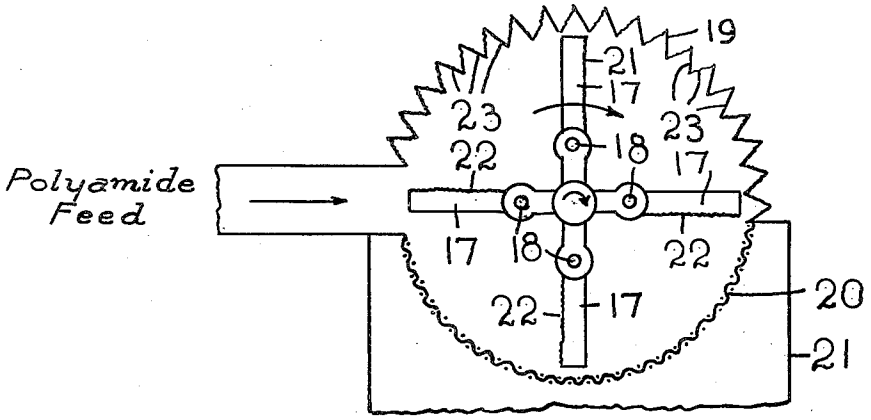
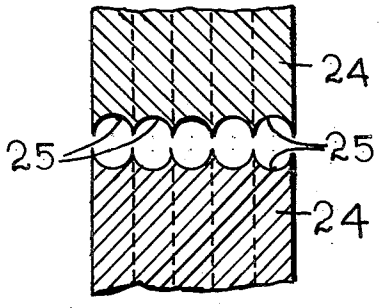


Fig. 3.



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MANUFACTURE OF STAPLE FIBERS

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7 Claims. (Cl. 18—54)

This invention relates to the manufacture of staple fibers. More particularly this invention relates to staple fibers fabricated directly from bodies of solid oriented synthetic linear polyamides of types such as hereinafter described. For convenience, the said types will be referred to at times, hereinafter, simply as polyamides.

Heretofore staple fibers of the said polyamides have been made by cutting continuous filaments into staple lengths. This is a lengthy and costly process since it involves spinning filaments through the minute holes of a spinneret, followed by cold-drawing and cutting. A further disadvantage of this process is that it gives staple having a smooth surface.

Attempts already have been made to produce staple fiber directly from films, by cutting the same into strips, but such procedure generally has yielded fibers having excessively sharp corners and excessively rectangular or square cross-sections, the said corners and cross-sections being such as to present considerable difficulties in certain weaving processes. Moreover, no economically satisfactory methods have been discovered for circumventing such difficulties. Staple fibers usually are very much improved, however, by treatments which serve to impart to their surfaces a roughened effect sufficient to cause the short fibers to cling or adhere to each other. It is noteworthy, in this connection, that such roughness generally is not advantageous in the case of continuous or long-length filaments, as distinguished from staple fibers.

This invention has as an object the production of staple fibers directly from ribbons, films or other solidified polyamide bodies. The invention also has as an object the concurrent impartation to the staple fibers of desired surface and cross-sectional characteristics. Further, the invention has as an object the manufacture of staple polyamide fibers in a continuous process, starting with the synthesis of the polyamide, or the melting of preformed polyamide. In addition, the invention has as an object the economical utilization of waste or scrap polyamide.

These objects, as well as others which will appear either expressly or impliedly hereinbelow, are accomplished according to the invention in its broadest aspects, by forming or starting with a solidified polyamide body, imparting orientation thereto preponderantly in a given direction, unless it already possesses such orientation, and then subjecting the said body to a breaking force. While the expression "breaking force" is here employed as comprehending torsional forces, i. e., forces such as are exerted when a body is wrenched by the exertion of a lateral force tending to turn one portion of it along a longitudinal axis, while another portion is held fast or turned in the opposite direction, the best results in the practice of the invention have been obtained where the breaking force has been imparted by relatively violent impact. Usually, also, the best results have been achieved when the polyamide body has been in a state of relatively free and unsupported motion at the instant of receiving the said impact. It will be noted, however, that in any case the reduction of the parent body to staple fibers is accomplished by an essentially non-cutting operation, i. e., solely or mostly by a breaking or shattering action, as distinguished from a cutting action.

Referring to the drawing accompanying the present application:

Fig. 1 is a flow sheet showing in diagrammatic form various steps to which the polyamide materials are subjected from their synthesis through the production of the staple fibers.

Fig. 2 is a somewhat schematic vertical section of a suitable mechanism for breaking an oriented polyamide sheet into staple fibers.

Fig. 3 is a side elevation in vertical section through a pair of corrugated rollers which may be employed for imparting rounded configurations to an oriented polyamide sheet passed between the rollers.

The accompanying drawing furnishes illustrative, but not limitative, examples of preferred modes of practicing the invention. Thus:

Fig. 1 shows, in flow-sheet style, the initial synthesis of polyamide in autoclave **10**, the extrusion of molten polymer therefrom, and the solidification of the same into cooled ribbon **11**, the cold-drawing of the ribbon, as between rollers **12** and **13**, the latter rollers being rotated at a sufficiently higher rate than the former to effect the desired degree of longitudinal stretching and consequent orientation of the ribbon, the transformation of the oriented ribbon, by subdivider **14**, into pieces whose size makes them appropriate for being fed into a breaking mechanism **15**, and collector means **16** for receiving the staple fiber produced in the breaking mechanism.

Fig. 2 is a schematic drawing of a type of mechanism which is especially appropriate for accomplishing the aforesaid breaking. The said mechanism is equipped with rotor bars **17** which are hinged, as at **18**, and rotated at speeds as high, e. g., as 9600 R. P. M., so as to impinge violently upon the incoming pieces of polyamide, as indi-

cated in the drawing, and to project them violently against corrugated cover plate 19, from which they drop to a retaining screen 20 whose mesh is such as to permit fibers of sufficiently small dimensions to escape to the collector means 21 but to retain for further comminution, by the rotor and/or corrugated cover plate, those fibers or pieces having excessively large dimensions. Suction means such as are familiar to those skilled in the art conveniently may be employed as an aid in facilitating the screening process.

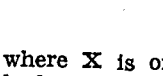
Preferably the impact surfaces 22 on the rotor bars, as well as the inner surfaces 23 or the corrugations of the cover plate, are of an abrasive nature, such as to impart minute scratches to the polyamide at the instant of impingement of the same thereupon.

Fig. 3 represents a special type of corrugated roller 24 which advantageously may be employed for the purposes served by roller 13 and/or roller 12 illustrated in Fig. 1. As already stated, it frequently is desirable for the staple fibers to have more or less round or rounded, as distinguished from excessively square or rectangular, cross-sections. The corrugations or fluted surfaces 25 of rollers such as 24 therefore are designed with minute semi-circular, or arcuate configurations—shown in greatly exaggerated size, at 25—so as to impart to the ribbon or film of polyamide drawn through them corresponding surface characteristics. Optionally, also, the surfaces of the semi-circular or arc-like corrugations or flutes may be formed of or provided with minutely divided abrasive materials in order to impart an initial roughening effect to the ribbon or film, as well also as to aid in traction. The pliability characteristics inherent in solidified polyamide make the action of the corrugated rollers uniquely effective. In consequence, the ribbon or film not only receives a high degree of orientation, but also acquires a large number of tiny longitudinal surface grooves, as it emerges from roller 13; and, when subsequently subjected to breaking forces in accordance with the invention, tends to break apart into staple fibers whose cross-sections are substantially rounded rather than excessively square or rectangular, or otherwise possessed of undesirably sharp corners.

Often it may be found desirable to effect the initial extrusion from the autoclave through a slit having minute flutes or corrugations such as those at 25. In such event the film or ribbon, as formed, will have the aforesaid longitudinal grooves or natural lines of cleavage, to supplement the tendency of the longitudinal orientation to cause the subsequent breaking-up to occur in the said direction. In such event, also, the orientation—the manner of production of which is more particularly referred to hereinafter—will most effectively be produced by cold-drawing, rather than by any compressive cold-working which might destroy the said grooves.

It is to be noted that whereas fluted rollers have been employed in the past, for the purpose of imparting grooves in aid of subsequent disintegration of sheet material, neither such procedure nor its effect, insofar as applicant is aware, has involved, either as an object or as a result, the production of filamentary or fibrous pieces having rounded cross-sections or arcuate cross-sectional peripheries.

The polyamides which are suitable for use in this invention are of the types described in U. S. P. 2,071,250, 2,071,253, 2,130,523, and 2,130,948. It may be observed, in passing, with respect to the structure of the polyamides that they contain amide groups



where X is oxygen or sulfur and where R is hydrogen or a monovalent hydrocarbon radical) as an integral part of the main chain of atoms in the polymer. It also may be observed that in these polyamides the average number of carbon atoms in the segments of the chain separating the amide groups is at least two. Accordingly, in this specification and the claims appended hereto, the expression, synthetic linear polyamide, is to be understood as applying to polyamides not only belonging to types described in the aforesaid patents, but also coming within the limitations pointed out in this paragraph. It will be apparent therefore that in the claims the expression "synthetic linear polyamide" is not intended to include polymers such as are formed by the condensation of urea and formaldehyde.

As indicated in the above-mentioned patents, the high molecular weight synthetic linear polyamides may be formed into useful films or ribbons which upon application of tensile stress are permanently stretched or "cold-drawn" into pliable, strong fibrous products which show by characteristic X-ray diffraction patterns that they are microcrystalline in structure and are oriented along their fiber axes. Orientation may also be effected by the application of compressive stress such as that which takes place in the process of cold rolling as described in Miles Ser. No. 170,470, now U. S. Patent 2,244,208, filed October 22, 1937, and assigned to the assignee hereof, or by a combination of tensile and compressive stresses such as takes place in the die drawing which is described in Brubaker Ser. No. 284,556, filed July 14, 1939, also assigned to the assignee hereof. It is desirable to orient the films while they contain a mild swelling agent therefor, e. g. water or an alcohol.

The conditioning or setting treatment which is given to the films or ribbons after their orientation is an important factor in determining their properties. Thus, subjection to dry heat treatment enhances their brittleness, which is desirable since it facilitates the subsequent breaking of the films. The heating operation advantageously may be carried out by placing the films in a heated oven or pressing them between heated surfaces.

It will be apparent that the invention rests in considerable degree upon the discovery that on being oriented preponderantly in a given direction the polyamide bodies will tend, upon being subjected to breaking or torsional forces, to separate along the lines of the said preponderant orientation. The ribbons, films, sheets, bands or rods, e. g., of the oriented polyamide preferably should be thin, e. g., 1–5 mils in thickness, in order to yield staple fibers having diameters as small, e. g., as 1 mil and lengths of as high, e. g., as several inches. It will be noted that the staple fibers thus produced vary in size, both transversely and longitudinally, thus contrasting with the uniformity in size of staple fibers normally produced by conventional processes.

In addition to the preferred procedures detailed hereinabove, for disintegrating the parent bodies of polyamide, it is possible to employ, with varying degree of success, various other processes. For instance, even grinding, pulverizing, twisting,

crushing, shredding or rubbing actions, generally, as distinguished from the preferred violent impact actions, ordinarily will yield staple fibers. Or, in place of apparatus such as depicted in Fig. 2, papers beaters, powder mills, or conventional types of swing hammermills, e. g., may be employed for delivering violent impacts to the parent bodies.

Further types of disintegrating apparatus which are more or less appropriate for the practice of the invention include conventional centrifugators wherein the parent material is centrifugated violently against peripherally located obstacles, pulverizers wherein collisions of convergent streams of pieces of the parent material are produced centrifugally, pulverizers in which such collisions are produced non-centrifugally, as by convergent streams of gas or air, and comminutors in which the initial pieces to be subjected to size-reduction are delivered to impact surfaces at speeds whose rate is greatly augmented by streams of high pressured gas or air.

It will be apparent that a horizontal arrangement of the parts of mechanism such as that depicted in Fig. 2, e. g., could be substituted for the vertical arrangement therein shown. It also will be apparent that the effectiveness of abrasive surfaces such as indicated in Fig. 2 could be supplemented by the addition of small amounts of silica dust or other very finely divided abrasive material to the atmosphere in the interior of the high speed mechanisms. Further, the efficiency of such equipment may be enhanced by designing the peripheral impact member, such as represented by the cover plate 19 in Fig. 2, in such manner that it rotates at high speed relative to the rotor bars, instead of remaining stationary, during the carrying out of the process of the invention.

An appropriate surfacing material for imparting the desired abrasive quality to the impact surfaces is finely divided tungsten carbide. Should the use of such surfacing, or the introduction of abrasive powders into the circumambient atmosphere, lead to excessive wear or heating-up of parts of the apparatus, however, there may be substituted for such expedients that of incorporating with the initial polyamide bodies, at least at their surface portions—for instance by introduction into the original solutions or melts—salts such as calcium chloride, and subsequently leaching out the same. The resulting products contain minute pores which make their surfaces especially appropriate for staple fibers.

A preferred polyamide for use in the practice of the invention is polyhexamethylene adipamide. A sample thereof, having a direct melt viscosity of 160 poises at 285° C., after extrusion into a film 4–5 mils in thickness and orientation of the same in the longitudinal direction, by cold-rolling, followed by subdivision into one inch lengths, has been found to yield a large percentage of very fine, short fibers, running as low as a mil in diameter and from several millimeters to an inch in length, when subjected to the action of a machine such as that shown in Fig. 2. The rotors of the said machine were rotated at a speed of 9600 R. P. M. It was found advantageous to employ a $\frac{3}{8}$'' screen in the first instance, and then to re-introduce into the apparatus the fragments initially screened out by it, for further disintegration, and screening through a $\frac{3}{32}$'' screen.

Other polyamides which are especially suitable for use in the practice of the invention include polyhexamethylene sebacamide, polydecamethylene adipamide and interpolymers such as that prepared from hexamethylenediammonium adipate and hexamethylenediammonium sebacate. Generally the best results are obtained with superpolyamides having a direct melt viscosity of 100–1000 poises at 285° C, and a melting point above 150° C. The invention is also applicable to modified polyamides such as polyester-amides and polyurethanes.

The invention is not limited to compositions containing polyamide alone. That is, the polyamide may be mixed with other agents such as plasticizers, resins, cellulose derivatives, pigments, dye or different polyamides. Certain of such agents are particularly valuable because of their brittleness-enhancing effect on the polyamide. Thus, substances such as p-tertiarybutylphenol formaldehyde resin or p-hydroxyldiphenyl formaldehyde resins are especially useful, in this respect, and consequently, when mixed with the polyamide in amounts ranging from 5% to 25%, give a product which is considerably more readily shattered in the direction of orientation than are polyamide bodies in the pure state.

A further method for increasing the brittleness of the parent polyamide material consists in subjecting the same to prolonged heat-treatment. It has been discovered that heating tends to weaken the lateral bonds of longitudinally oriented polyamide, and therefore to increase the ease with which it may be split into fragments. For instance, an oriented polyhexamethylene adipamide film which had been heated for 20 hours at 150° C. and as a result had attained a bone dry state, was found to have become sufficiently brittle to be shattered into staple fibers merely by rubbing or frictioning. In this connection, it is to be observed that the staple fibers can be formed at high temperatures, e. g., up to within 15–20° C. of the melting point of the polyamide composition, thus making it not only unnecessary but to some extent disadvantageous to conduct the operations at room temperatures.

Often the embrittling effect of heat may be augmented by steaming the stretched polyamide under tension. Or, such effect may be augmented, or independently brought about, by incorporating in the polyamide agents such as inorganic salts, which cause fissures or voids, since the polyamides will tend to be shattered more readily by reason of the existence of such fissures or voids.

The invention is not limited to parent polyamide bodies which have been formed by solidification from melt. Thus, films or ribbons which have been produced by solvent casting processes, likewise become eligible for use, upon being oriented in a given direction.

Staple fibers prepared in accordance with the invention have utility for many purposes. Typical applications for them are threads, woven fabrics, pile fabrics, upholstery stuffing, rope, and electrical insulation. They also may be molded into useful articles either alone or in conjunction with a resin or unoriented polyamide.

Among the outstanding advantages of the staple fibers of the invention is their capacity for being washed, scoured, boiled, or subjected to other ordinary heat treatments, without losing their shape, without undergoing serious shrinkage, and without reverting to an unoriented state.

Although the high speed impact process exemplified by, and in connection with, Fig. 2 uses less power and causes less heating-up of the equipment than ordinary grinding processes, e. g.,

it nevertheless is an advantageous feature of the invention that any need for neutralizing such heating is avoided or minimized by reason of the fact that the breaking-up or shattering of the polyamide bodies can be effected at temperatures approaching their melting point. Since the polyamides characteristically have relatively high melting points, this advantage becomes especially significant. The susceptibility of the polyamides to be ground at elevated temperatures is unique among high molecular weight organic polymeric materials, since, as is well known, the softening of thermoplastic materials of such types, produced by heat generated during their grinding, customarily necessitates resort to expensive cooling apparatus, in order to make possible successful carrying on of such grinding.

Since many apparently widely different embodiments of this invention may be made without departing from the spirit 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. A process for the production of staple fibers of varying cross-sectional areas from synthetic linear polyamide which comprises forming a sheet of said polyamide, imparting orientation thereto preponderantly in a given direction and subjecting said oriented polyamide sheet to violent impact while said sheet is in a relatively free and unsupported state thereby breaking said sheet along the lines of said preponderant orientation into staple fibers having rough surfaces and varying cross-sectional diameters.

2. The process as set forth in claim 1 wherein the synthetic linear polyamide is polyhexamethylene adipamide.

3. The process as set forth in claim 1 wherein the synthetic linear polyamide has a melt viscosity of 100–1000 poises at 285° C. and a melting point above 150° C.

4. A process for the production of staple fibers of varying cross-sectional areas from synthetic linear polyamide which comprises forming a sheet of said polyamide, imparting orientation thereto preponderantly in a given direction and subjecting said oriented polyamide sheet to abrasion and violent impact while said sheet is in motion in a relatively free and unsupported state thereby scratching and breaking said sheet along the lines of said preponderant orientation into staple fibers having rough surfaces and varying cross-sectional diameters.

5. A process for the production of staple fibers of varying cross-sectional areas from synthetic linear polyamide which comprises forming a sheet of said polyamide, enhancing the brittleness of said sheet by incorporating in said polyamide from which said sheet is formed a phenol-formaldehyde resin, imparting orientation to said sheet preponderantly in a given direction and subjecting said oriented polyamide sheet to violent impact while said sheet is in a relatively free and unsupported state thereby breaking said sheet along the lines of said preponderant orientation into staple fibers having rough surfaces and varying cross-sectional diameters.

6. A process for the production of staple fibers of varying cross-sectional areas from synthetic linear polyamide which comprises forming a sheet of said polyamide, imparting orientation thereto preponderantly in a given direction, heating said oriented sheet until its brittleness has been enhanced, and subjecting said embrittled oriented polyamide sheet to violent impact while said sheet is in a relatively free and unsupported state thereby breaking said sheet along the lines of said preponderant orientation into staple fibers having rough surfaces and varying cross-sectional diameters.

7. In the process of manufacturing longitudinally oriented staple fibers of material comprising synthetic linear polyamide, the said fibers having at least partially arcuate cross-sectional peripheries, from sheet material comprising the said polyamide, the steps which consist of passing the sheet material between pressure rollers having minute oppositely disposed arcuate flutes, thus forming correspondingly arcuate grooves in the said sheet material, simultaneously stretching the said sheet material, thus imparting longitudinal orientation thereto, and thereafter shattering the said sheet material by subjecting it to violent impact.

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