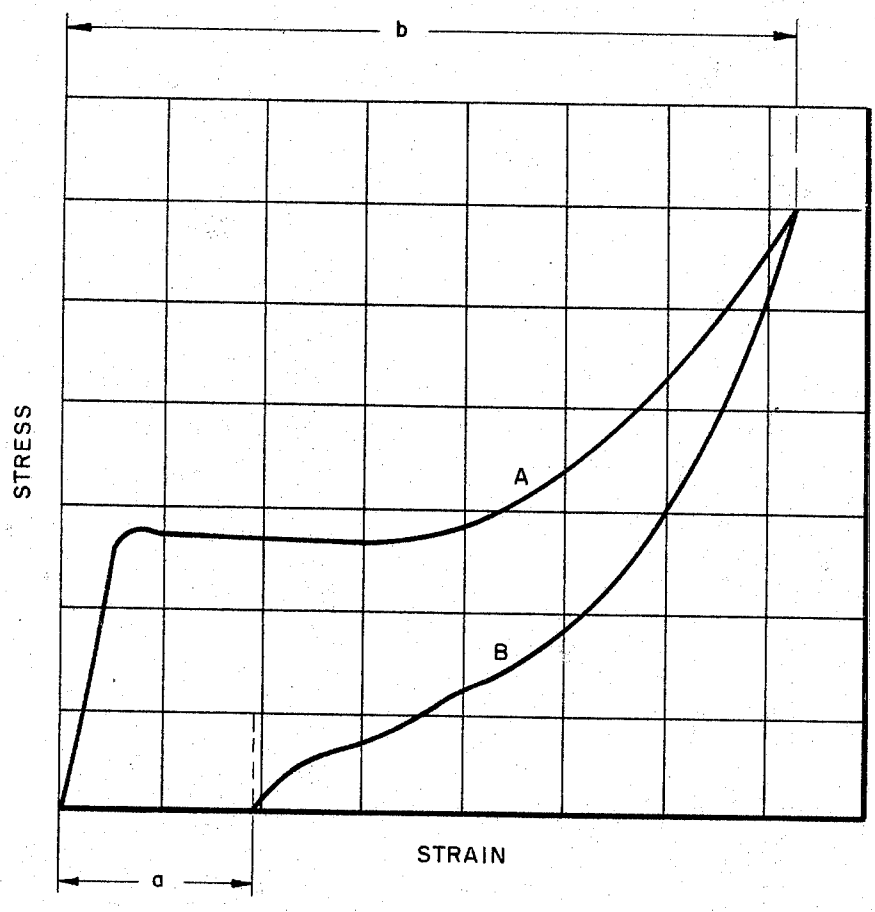
June 6, 1967 A. BOLTNIEW 3,323,190

ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963 8 Sheets-Sheet 1



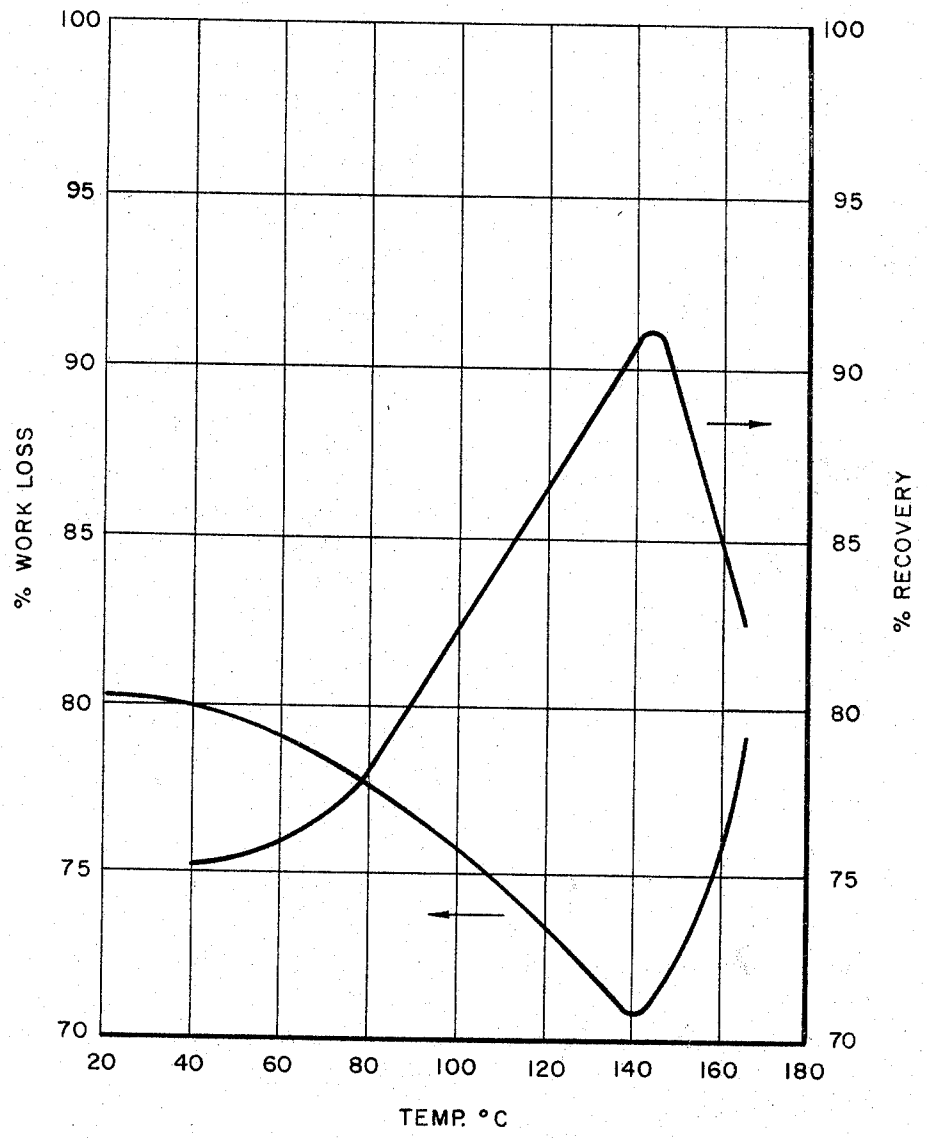
TYPICAL ELONGATION - RECOVERY CURVE

FIG. 1

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963 8 Sheets-Sheet 2



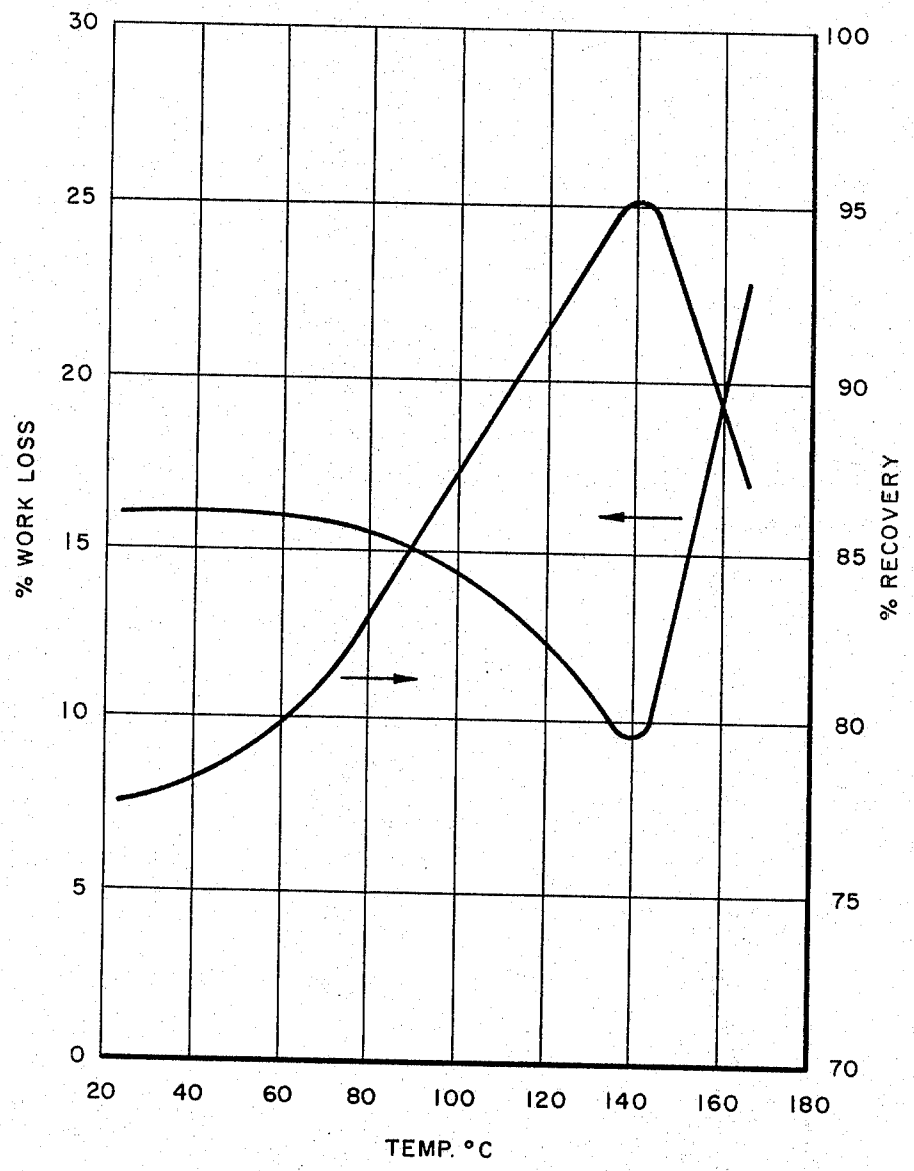
% RECOVERY & WORK LOSS AFTER A SINGLE 75% ELONGATION VS HEAT TREATMENT TEMPERATURE (UNDRAWN YARN)

FIG. 2

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963



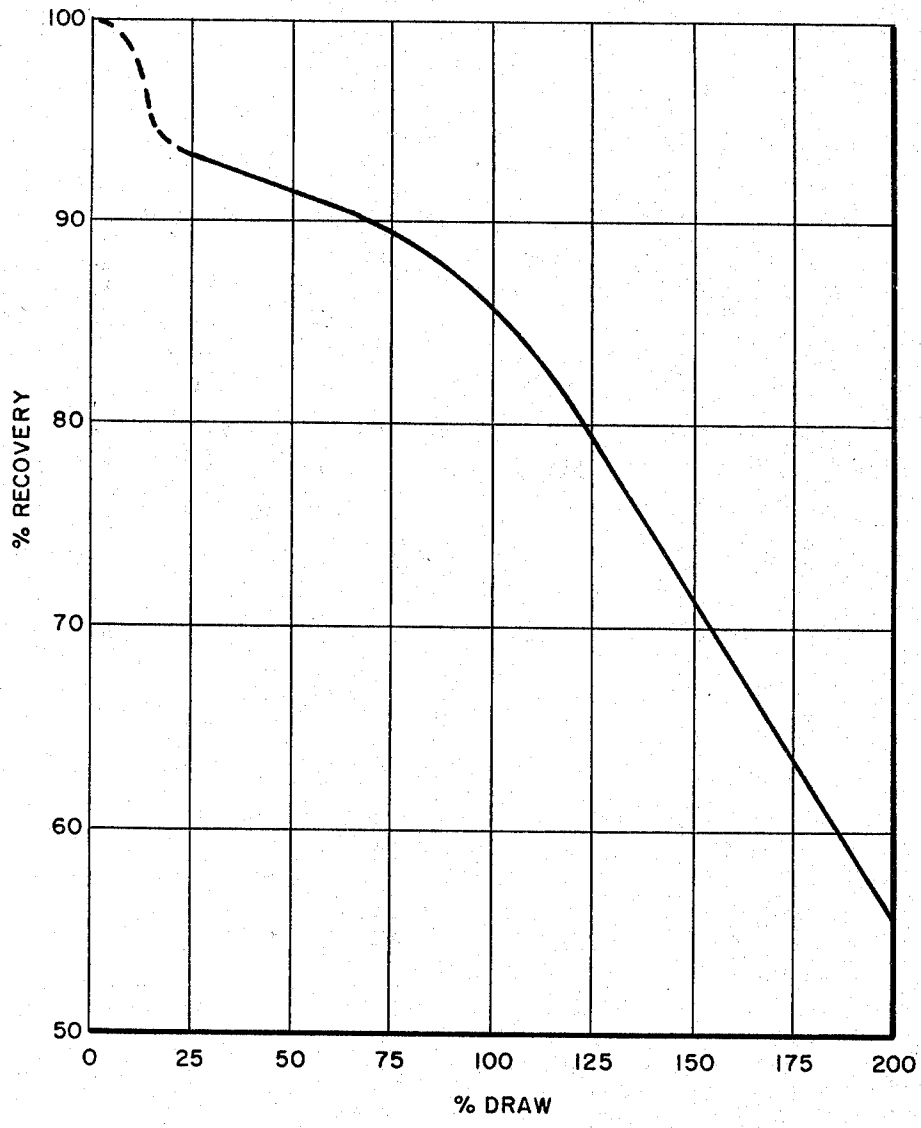
% RECOVERY & WORK LOSS AFTER 10 CYCLES 0-50% ELONGATION VS HEAT TREATMENT TEMPERATURE

FIG. 3

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963 8 Sheets-Sheet 4



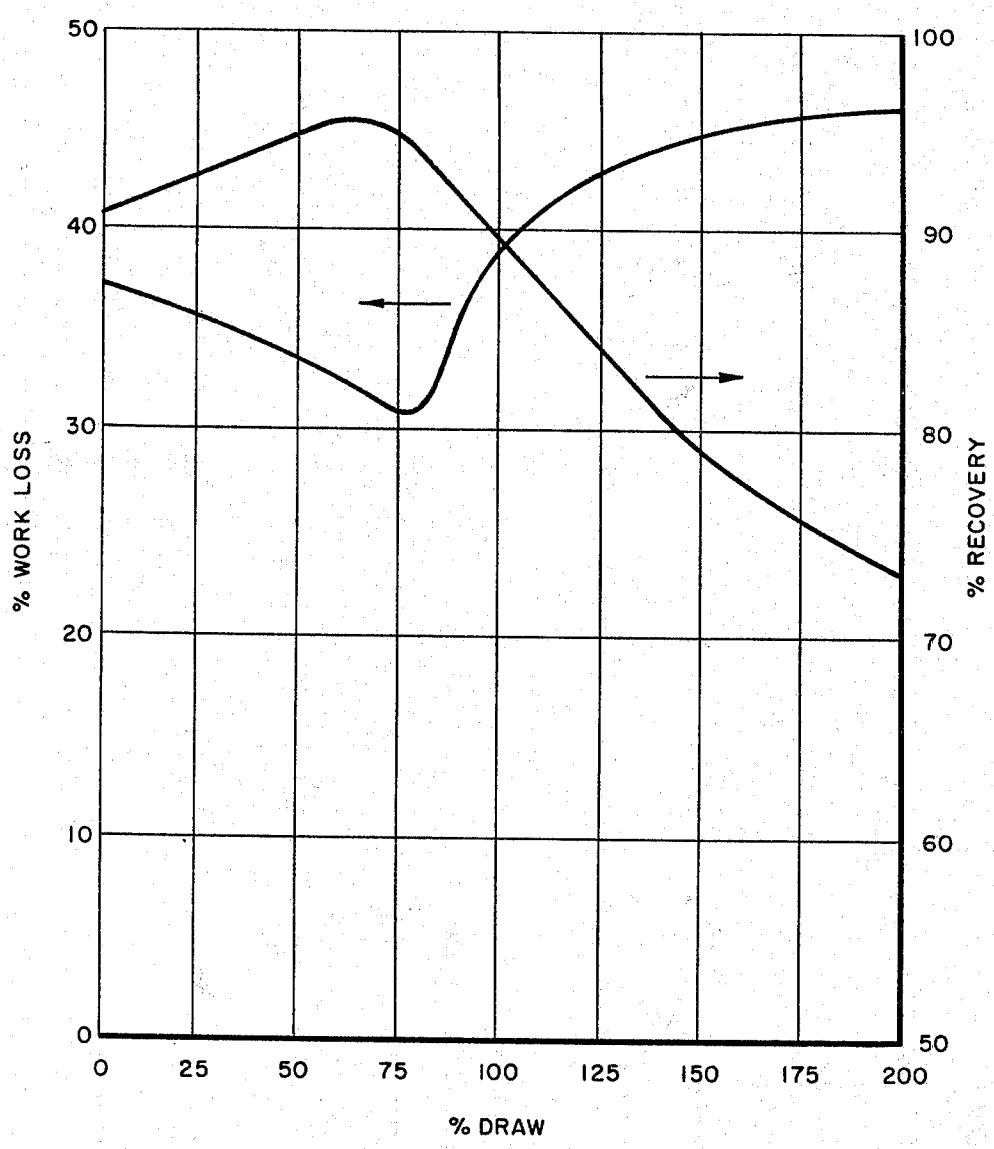
% RECOVERY FROM SINGLE ELONGATION AFTER HEAT TREATMENT AT 140°C FOR 10 MINUTES

FIG. 4

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963 8 Sheets-Sheet 5



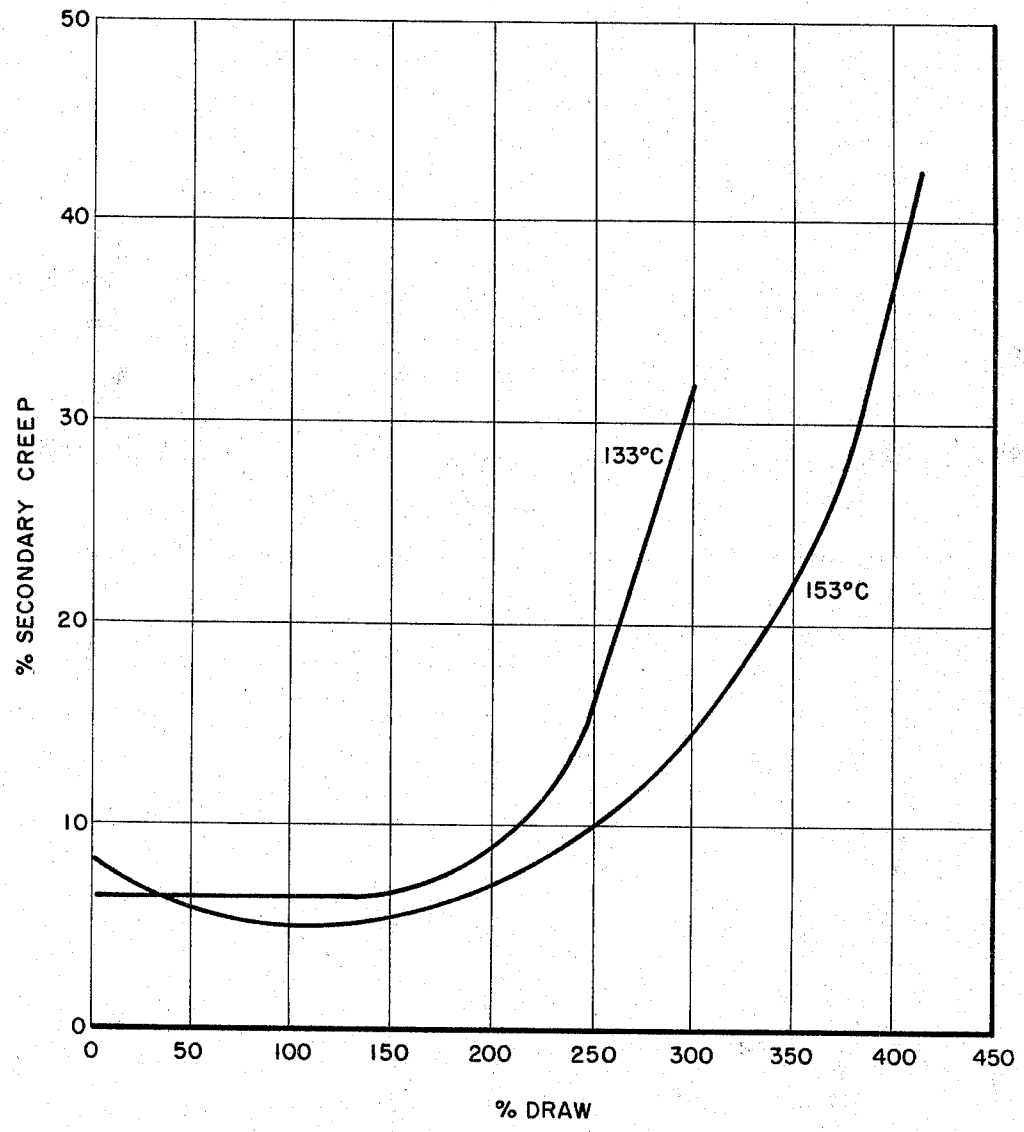
% RECOVERY & WORK LOSS FROM 10 CYCLES 0-50 % ELONGATION VS % DRAW AFTER HEAT TREATMENT AT 140° C

FIG. 5

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963 8 Sheets-Sheet 6



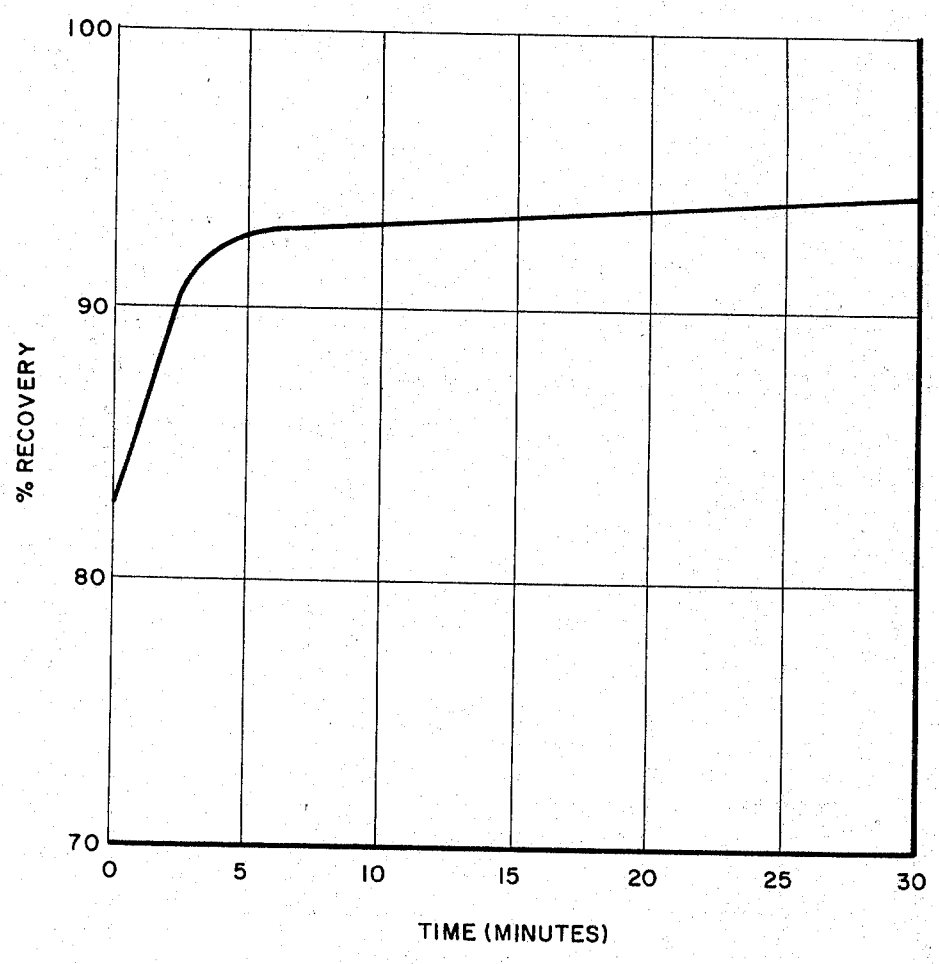
% SECONDARY CREEP AFTER 10 CYCLES 0-50% ELONGATION VS % DRAW PRIOR TO HEAT TREATMENT AT 133°C & 153°C (ALL YARNS DRAWN 75% AFTER HEAT TREATMENT & BEFORE CYCLING)

FIG. 6

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Filed June 12, 1963 8 Sheets-Sheet 7



% RECOVERY VS TIME OF HEAT TREATMENT AT 140°C

FIG. 7

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ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

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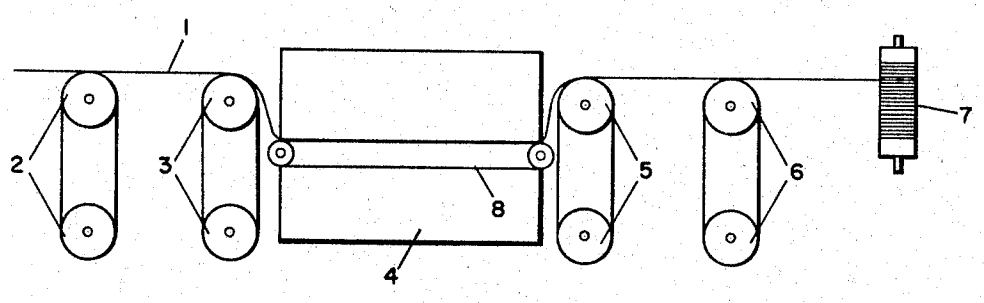


FIG. 8

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United States Patent Office 3,323,190 Patented June 6, 1967

3,323,190

ELASTIC POLYPROPYLENE YARN AND PROCESS FOR ITS PREPARATION

Alexander Boltniew, Covington, Va., assignor to Hercules Incorporated, a corporation of Delaware

Filed June 12, 1963, Ser. No. 287,326
3 Claims. (Cl. 28—72)

This invention relates to yarns of polyropylene having a high degree of elasticity and to processes for the production thereof.

So-called "stretch yarns" are much in demand in the textile field at the present time. Such yarns are finding increasing application in the clothing industry in general in the manufacture of a wide variety of snug fitting garments. They are particularly adaptable to the manufacture of items such as hosiery, corsets, gloves, stretch pants and the like, designed to fit a range of sizes.

A number of stretch yarns of synthetic polymer are known to the trade. Many stretch yarns are based upon the plastic memory of the synthetic polymer from which they are made. The basis of manufacture of such stretch yarns is that the synthetic yarn is held in a crimped or folded condition by some means or other while at an elevated temperature so that upon cooling it remains crimped. If yarn that has been so crimped is pulled, the crimps straighten out, and the yarn extends in length. When the extending force is removed, the crimps reform, and the yarn contracts in length. Other types of synthetic stretch yarns depend upon the insertion of a high twist at some stage of their production. Still other types of stretch yarns are helically crimped. The common feature of all such yarns, however, is that stretchability is a result of the physical structure of the yarn rather than inherent elasticity of the synthetic polymer.

There are also known to the art stretch yarns which are truly elastic, i.e., they possess the ability to be stretched repeatedly and regain substantially their original length after removal of the elongating force due solely to the inherent elasticity of the material from which they are made. An example of elastic yarn is so-called spandex, which is made from a rubbery polyurethane.

It was not to be expected that a truly elastic yarn could be made from crystalline polypropylene because this polymer has very little inherent elasticity. This is shown by the fact that polypropylene yarn as it is normally produced has no significant amount of elasticity. It was therefore most surprising to find, in accordance with this invention, that it is possible to produce a polypropylene yarn that has the ability, due solely to the elasticity of the polymer, to be stretched repeatedly to at least 50% elongation and then recover substantially its original length. The present invention relates to such a yarn and to the methods by which it can be produced. More particularly, the invention relates to polypropylene yarn which is characterized by being capable, due solely to the elasticity of the polymer, of recovering to at least 90% of its original length after repeated cycling between 0 and 50% elongation. The invention further relates to a method of producing such a yarn which comprises subjecting polypropylene yarn which has been drawn not more than about 150% to heat treatment between 135 and 150° C. for from 3 to 30 minutes and thereafter drawing the heat treated yarn 40 to 80%.

The attached drawings, FIGS. 1 to 7 are graphical presentations of data which will be used to define the invention. More particularly, FIG. 1 is a typical elongation-recovery curve for undrawn polypropylene yarn. FIG. 2 is a graph showing the effect of heat treatment temperature on undrawn polypropylene yarn with respect to percent recovery and percent work loss after a single cycle draw of 75%. FIG. 3 is a graph showing percent

recovery and percent work loss following ten cycles of 0–50% extension on polypropylene yarn that has been heat treated at various temperatures before drawing and then drawn 75% after the heat treatment. FIG. 4 is a graph depicting percent recovery from single elongation cycles varying between 25 and 200%. FIG. 5 shows the effect of varying the amount of preliminary draw on the elastic properties after cycling between 0 and 50% elongation. FIG. 6 is a graph showing the effect on elasticity of drawing the yarn prior to heat treatment. FIG. 7 shows the effect of varying heat treatment time on elastic properties. FIG. 8 is a schematic diagram of apparatus suitable for treating yarn according to this invention. These drawings will be discussed more fully below with reference to the examples and definitions to which they are pertinent.

A familiar criterion for measuring the elastic properties of a yarn is its ability to return to its original length when it is relaxed after being subjected to cyclic, or repeated, stresses. This property is referred to as "elastic recovery" or more simply as "recovery." It is expressed as a percentage of recovery of the original length. It should be obvious that higher values of recovery indicate higher elasticility. The amount of the original length which is not recovered when the yarn is relaxed after repeated stresses is expressed as percentage of permanent set or secondary creep. Secondary creep is equal to 100 minus percent recovery.

Another criterion for measuring elasticity is the percent work loss. This parameter is determined by measuring the area under the elongation and relaxation curves of yarns subjected to an elongating stress. It is expressed as the ratio of the area between the elongation and relaxation curves to the total area under the elongation curve.

In FIG. 1 is shown a typical elongation-recovery curve. Referring to this curve, percent recovery is defined as

$$\frac{b-a}{b}\times 100$$

where b=distance elongated and a=distance which sample failed to relax.

Percent work loss is defined as

$$\frac{S_A-S_B}{S_A}\times 100$$

where S_A=area under elongation curve A, and S_B=area under relaxation curve B.

In the usual method of production of multifilament synthetic yarns, molten polymer is formed into filaments by extruding through a spinneret and subjecting the polymer in the molten state to a high degree of elongation to effect a reduction in denier. This denier reduction, or draw down, is usually on the order of about 30 to 150. After the draw down, the polymer is stretched between differentially driven rolls at a temperature below its melting point. Stretching at a temperature below the melting point of the polymer—referred to simply as "drawing"—results in a high degree of molecular orientation and improved physical properties of the yarn. The stretch oriented yarn is then ready for use as is or it can be subjected to further operations, such as bulking, twisting, stapling, or other textile finishing operations prior to use. An undrawn yarn within the contemplation of the present invention is one which has not been drawn.

The elastic yarns of the invention are prepared by a novel process involving a unique heat treatment of polypropylene yarn that has been drawn not more than a prescribed maximum followed by drawing of the heat-treated yarn to a relatively small degree. The invention has not yet been explained theoretically in terms of the changes that take place in the physical structure of the polymer but presumably changes of some sort do occur.

Two embodiments of the process by which the elastic yarns of the invention can be made have been defined. In one embodiment yarn which has been drawn not more than about 2.5×(150%) is heat treated in the relaxed state to a temperature of about 135 to 155° C. for a time of at least about 3 minutes, following which the heat treated yarn is drawn about 1.40 to 180×(40 to 80%). In the second embodiment, the yarn which is substantially undrawn is given a heat treatment by wrapping it several times around a roll heated to a temperature from about 145 to 155° C. and is then drawn about

1.20 to 1.30×(20 to 30%)

by pulling from the hot roll onto another heated roll (about 145 to 155°C.) where it is also wrapped several times around the roll. In the latter embodiment several wraps on each roll appear to be necessary to give sufficient residence time on the rolls to bring the yarn to approximately the same temperature as the rolls.

In the embodiment of the invention depicted in FIG. 8, yarn **1** is fed around first feed rolls **2** to first draw rolls **3** onto moving belt **8** and through heat treating oven **4**. The yarn is advanced through the oven by second feed rolls **5**, then to second draw rolls **6** and collected on bobbin **7**.

The initial drawing is effected in known manner by first draw rolls **3** at a rate up to about 2.5 times the rate of first feed rolls **2**. The drawn yarn is then deposited on moving belt **8**, advanced through oven **4** and drawn off by second feed rolls **5**. The relationship between the speed of the first and second draw rolls and that of the moving belt is adjusted such that there is no tension on the yarn as it passes through the oven and the quantity of yarn thereon remains constant. The drawing after the heat treatment is then accomplished in the span between second feed rolls **5** and second draw rolls **6**.

It should be understood that the equipment arrangement depicted is not critical and forms no limiting part of the invention. Only the process steps are critical and these can be carried out with any suitable equipment in either a continuous process as depicted or in a stepwise operation wherein the yarn is collected between the steps.

Following are examples of the invention.

Examples 1 to 3 were performed on a 5700 denier, 210 filament yarn. This yarn is a composite of three individual yarns produced by extruding molten polypropylene at a temperature of about 265° C. through a 70 hole spinneret, drawing down the molten yarn by about 83 to 1, and plying the three yarns to form a single yarn. Heat treatment was effected by placing 8 to 10 yards skeins of the yarn completely relaxed on a metal grating covered with cotton gauze and placing the metal grating into a forced draft oven. The draw in each case, whether effected before or after the heat treatment, was performed at a rate of 60% per minute.

Example 1

Samples of undrawn polypropylene yarn were heat treated for 10 minutes at a range of temperatures varying from room temperature up to 165° C. After the heat treatment, the samples were removed from the oven, and allowed to cool to room temperature. Each of these samples was subjected to a 75% draw at room temperature at the rate of 60% per minute. From this cycle percent recovery and percent work loss were determined. The data thus derived are presented graphically in FIG. 2, in which the curve shows a sharp peak at a heat treatment temperature of about 140–145° C.

The same yarn samples were permitted to relax fully and then were subjected to ten 0–50% extension cycles. The percent recovery for the ten cycles was found to reach a maximum of about 95% in the yarn which had been heat treated at 140–145° C. Correspondingly, the work loss reached a minimum at this point. Recovery and work loss data for the ten cycles are presented graphically

in FIG. 3. In contrast to the 95% recovery exhibited by the 140° C. treated yarn, undrawn polypropylene yarn which had been processed similarly but not heat treated showed only 79.0% recovery (or 21% secondary creep) after 75% draw and ten 0–50% elongation cycles.

Example 2

As indicated in Example 1, it was not unexpected that some degree of permanent set would be induced by the 75% preliminary draw following heat treatment. In order to determine the effect of the amount of this preliminary draw on ultimate elastic properties, substantially undrawn yarns were heat treated at 140° C. as in Example 1. Different samples of yarn were drawn at room temperature to elongations between 25 and 200%. FIG. 4 shows the percent recovery from a single elongation for various degrees of elongation. As is shown, between 25 and 75% draw, the per cent recovery shows very little change, then decreases very rapidly at higher draws. However, as FIG. 5 will indicate, the amount of this draw is a significant factor in creating elastic properties in the yarn.

After each draw, the resulting slack was taken out of the yarn and the sample was cycled ten times at room temperature between 0 and 50% elongation using an Instron tensile tester. At the end of the cycling, work loss and per cent recovery were measured. The results of these measurements are depicted graphically in FIG. 5. From FIGS. 4 and 5, it can be seen that the greatest elasticity (i.e., maximum recovery and minimum work loss after ten cycles of 50% elongation) is developed by the samples which have been subjected to about 60–75% draw after heat treatment.

Summarizing the data in Examples 1 and 2, it can be said that the optimum conditions for developing elasticity in undrawn yarn are 140–145° C. heat treatment for 10 minutes, followed by about a 75% draw at room temperature. Yarn thus treated is capable of recovering about 95% of its original length after repeated cycling to 50% elongation.

Example 3

Though application of the invention to undrawn yarns is the preferred embodiment, the invention is also effective in imparting elasticity to partially drawn yarns. By partially drawn yarns is meant yarns which have been subjected to a cold draw prior to the heat treatment, the amount of the draw being less than the maximum to which the same polypropylene yarns can be drawn without breakage of fibers.

In this example the yarn samples were drawn at room temperature in increments of 50% from 0 to 400% at a rate of 60% per minute prior to heat treatment. Samples of yarn at each degree of draw were treated at 133° C. and 153° C. for 10 minutes using the method outlined in Example 1, subjected to 75% draw at room temperature and 10 cycles of 0–50% elongation. The designated treatment temperatures were selected as reasonable limits of satisfactory heat treatment. Experimental values for percent recovery after 10 elongation cycles are depicted in FIG. 6. It can be seen that optimum elasticity of the yarn is developed when the draw before heat treatment is less than 150%. In each case, that is 133° C. and 153° C. tratment, elasticity deteriorates rapidly as the draw exceeds 150%.

Considering FIG. 6, it can be seen that, for yarn drawn prior to heat treatment, higher heat treatment temperature produces better elastic properties. Though recovery properties are quite similar in the best range (up to 150%), the 153° curve reaches a higher value for percent recovery and also decreases less rapidly as the draw is increased above 150%. Also pertinent to this conclusion is the observation that highly drawn samples treated at 133° C. could not be given the desired 75% draw after heat treatment. This relatively low break point and elastic limit indicate a less elastic condition at higher draw.

Example 4

In this example, samples of undrawn 3600/210 polypropylene yarn were heat treated by use of heated feed and draw rolls rather than by placing them relaxed in an oven. The yarn was drawn around a feed roll heated to 150° C. at a rate of 140–150 meters per minute, with about sixty (60) wraps of yarn on the roll. From the feed roll, the yarn was drawn at a rate of 52.5 meters per minute (1.25× draw) to a draw roll heated at 150° C., making 15 wraps around the draw roll.

The yarn thus produced was subjected to 10 elongation cycles of 0–50%. The elasticity of the resulting yarn gave 6.5–8.5% secondary creep or 91.5–93.5% recovery.

The heat treatment time in the first three examples has been fixed at 10 minutes. Experience has shown that this is not a limitation on the process. This time can be varied through rather wide limits, from about 3 to 30 minutes with substantially the same results. The permissible wide variation in treatment time is shown graphically in FIG. 7 which shows the very small effect on percent recovery after 10 cycles of 0–50% elongation.

Other methods of performing the process of the invention will occur to those skilled in the synthetic fiber art. For instance, the heat treatment in the relaxed state can be conducted by conveying the yarn on a continuous belt through the oven or heated treating chamber. In the heat treatment in the non-relaxed state using rolls, the heating medium may be a hot inert gas or a hot solvent bath rather than heated rolls.

The term "polypropylene" is used herein to designate the crystalline polypropylene of commerce, often called isotactic polypropylene.

What I claim and desire to protect by Letters Patent is:

1. A process for producing elastic polypropylene yarn exhibiting a recovery of at least 90% after being subjected to a minimum of ten 0 to 50% elongation cycles in each

of which the yarn is elongated at least 50% of its unstretched length which comprises subjecting polypropylene yarn which has been drawn not more than about 150% to heat treatment between 135 and 150° C. for from 3 to 30 minutes and thereafter drawing the heat-treated yarn 40 to 80%.

2. The process of claim 1 wherein the yarn is in the relaxed state during heat treatment.

3. The process of claim 1 wherein the initial yarn is undrawn.

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