Drawing of Nylon-6 by the Novel Incremental Drawing Process

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Synopsis

Multifilament nylon-6 fibers are drawn by the novel incremental drawing process as well as by the conventional drawing process. In this process the fibers are stretched in 36 stages along the surface of two corotating cones fitted on the incremental drawing machine. Fibers are obtained from each stage, and from their diameter measurements it is shown that they are stretched in a predicted manner. Mechanical properties, as measured by Instron and by a sonic modulus tester, show higher tenacity and modulus values for the incremental process than for the conventional one at equivalent draw ratios. Structural properties are analyzed by density measurements, wide angle X-ray diffraction and birefringence. These showed higher crystallinity and higher crystalline as well as amorphous orientation factors for the incremental process at equivalent draw ratios. The mechanical property results are explained on the basis of structural development during drawing. It has been shown that the incremental drawing process is a suitable technique for obtaining superior properties in fibers and has commercial potential.

INTRODUCTION

The use of polymeric materials for producing synthetic fibers holds a very important place in today's man-made fiber industry. The process of producing a fiber from a polymer is well established and discussed in detail in the literature.[1-4] Conventionally it involves a spinning step which produces continuous filaments of uniform cross section which are of low strength and high extensibility. The spun filaments are then drawn to align the molecular structure along the filament direction to attain high strength and low extensibility which are essential fiber properties. The drawing step thus becomes an important one for achieving and controlling the desired properties in the fiber. The conventional drawing process (CDP) involves the stretching of the spun fibers between two sets of rollers with the take-up roller rotating at a considerably higher speed than the feed roller. The draw ratio is simply the ratio of surface speeds of these two rollers. This process results in impulsive drawing, which occurs over a relatively short span of time and length of fiber, and is generally accompanied by the formation of a neck. This is a one step process and the tensions that result in the fiber set an upper limit to the draw ratio and strength possible.

An alternate route to drawing of fibers involves stretching in several successive steps in which a small amount of stretching occurs in each step.

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Journal of Applied Polymer Science, Vol. 31, 441–455 (1986)
CCC 0021-8995/86/020441-15$04.00

In such a process invented by Sussman,[5] the fibers are drawn incrementally and is referred to as the incremental drawing process (IDP). The design details of this novel process are described elsewhere,[5-7] and only a brief description is given in this paper. The major thrust of this paper is to study the structure and properties of commercial nylon-6 fibers by the IDP. The same fiber was also drawn by the conventional process, and the results for the two drawing processes are compared.

INCREMENTAL DRAWING PROCESS

The incremental drawing process (IDP) is a novel process in which spun synthetic fibers are stretched in several successive steps to orient the molecular structure in an incremental manner. The larger the number of steps, the smaller is the magnitude of each increment. This is physically achieved by drawing the fiber between two bodies, one or both of which have a continuously increasing diameter. Figure 1 illustrates such a process using two identical cones with steps. The fiber is fed to the lower diameter end and is made to travel along the surface towards the higher diameter end. As it moves up a step, it experiences a higher surface velocity resulting in its drawing at every step. The steps are designed to give a microterraced surface such that the fiber nests in each

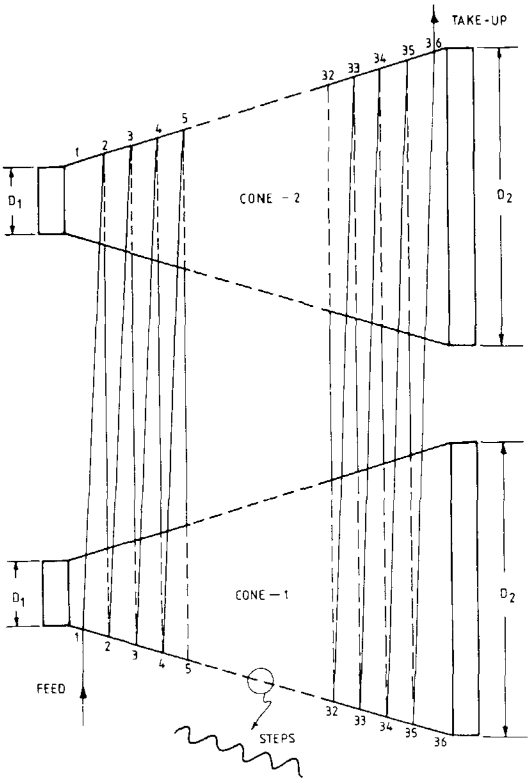

Fig. 1. Illustration of incremental drawing process using cones.

step and does not slip down to the lower diameter. A schematic diagram of the complete assembly of the incremental drawing machine is shown in Figure 2, and a photograph of the set up that was constructed is shown in Figure 3. It is designed so that the cones could be taken out and replaced with new ones if desired. The cones used for this work consisted of 36 steps, to give a maximum draw ratio of 4.11.

The fiber to be drawn is fed at the lowest diameter (or an intermediate diameter) of the cone, threaded through each of the steps, and finally taken up at the highest diameter. In this manner the fiber follows a helical path as it moves along the cones both of which rotate at the same speed. As a consequence of passing successively from lower to higher diameters, the surface speed increases at each step, resulting in incremental acceleration and incremental drawing brought about through the frictional contact with the drawing surface. The draw ratio at each step is simply the ratio of the step diameter it is on to the immediately lower diameter. The overall draw ratio is the ratio of diameter at the take-up end to the diameter at the feed end. Varying draw ratios can be attained by changing either the step at which the fiber is fed or the step at which it is taken up. In our setup the take-up was fixed as this is linked with the take-up mechanism, and thus the feed step was varied to obtain desired draw ratios. The draw ratio is unaffected by the drawing speeds. The present set-up has no arrangement for heating; thus all drawing is done at room temperature.

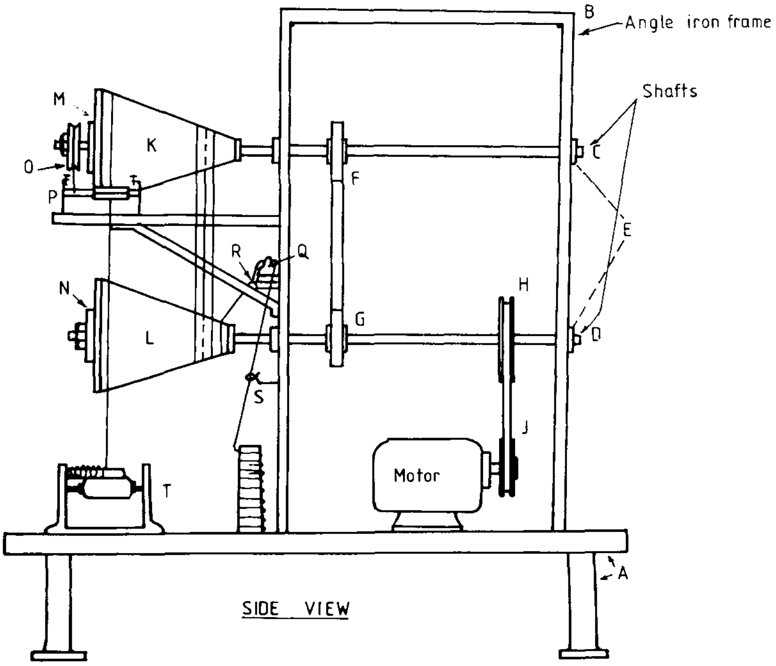

Fig. 2. Schematic diagram of the incremental drawing machine: (A) table; (B) frame; (C, D) shafts; (E) bearings; (F, G, H, J) pulleys; (K, L) cones; (M, N) washers; (O) washer cum pulley; (P, R) tensiomers; (R, S) guides; (T) winding unit.

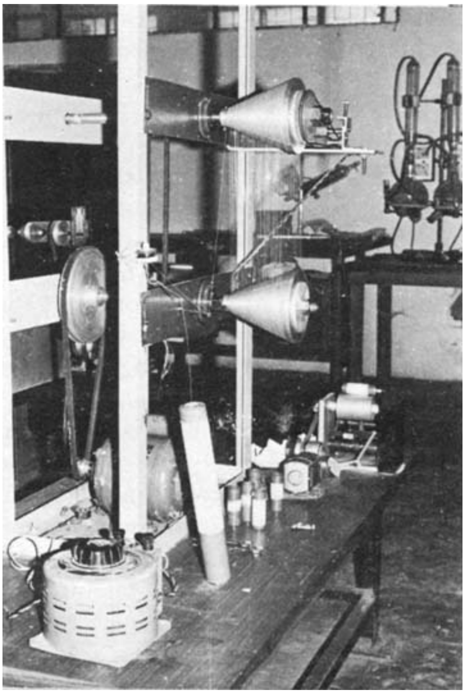

Fig. 3. Photograph of the incremental drawing machine.

EXPERIMENTAL

Sample Preparation

Nylon-6 was chosen for this study as it can be drawn easily at room temperature. Multifilament as-spun nylon-6 (22 filaments) yarn with a denier of 370 was obtained from Modipon Ltd., Modinagar, and was drawn within 3 days of spinning. The polymer used for making these fibers had a number average molecular weight of 18,400 and an intrinsic viscosity of 0.71 in 85% formic acid.

Multifilament yarn was drawn both by the IDP as well as the CDP. For the conventional process five different draw ratios were used in range of 1–4. For the IDP, the maximum attainable draw ratio of 4.11 with the present set of cones was used. The machine was run at a constant speed, and, when steady state was attained, the machine was suddenly stopped, which is hereby referred to as "freezing" of the system. Under these conditions the fibers at each step have different draw ratio. These were carefully cut and used to follow the changes in properties and structure as a function of draw ratio. This represents a unique method of studying the process of molecular orientation that accompanies the drawing of fibers. Drawing and freezing under identical conditions was done several times to obtain a sufficient amount of fibers at each step. Three methods were used for threading the fiber on the incremental drawing machine with the following sequences between the two cones (refer to Fig. 1):

(1) Feed—step 1, cone 1—step 2, cone 2—step 2, cone 1—step 3, cone

2–step i, cone 1—step $i+1$, cone 2—step $i+1$, cone 1—last step, cone 2—take-up.

By this procedure drawing takes place only when fiber goes from cone 1 to cone 2 while some relaxation of structure may take place when going from cone 2 to cone 1. The fiber is drawn by the maximum number of step in a draw–relax–draw sequence and is referred to as IDP-1.

(2) Feed — step 1, cone 1 — step 2, cone 2 — step 3, cone 1 – step i, cone 1— step $i+1$, cone 2—step $i+2$, cone 1—last step, cone 2—take-up.

In this procedure the number of steps remains the same as in IDP-1, but the fiber is drawn every time it goes from one cone to the other. This draw–draw–draw sequence is referred to as IDP-2.

(3) Feed—step 1, cone 1—step 3, cone 2,—step 5, cone 1–step 1, cone 1,—step $i+2$, cone 2—step $i+4$, cone 1,—last step, cone 2—take-up.

This procedure is similar to that of IDP-2 except that one step is skipped every time the fiber changes cones and thus the total number of steps are reduced to half. This is referred to as IDP-3.

Determination of Draw Ratio and Diameter

For determining the draw ratio, the denier of the fiber was first obtained at all the steps. A fixed length of 25 cm was marked on the fibers at each step when they were still under drawing tension with the machine stopped. This length was then cut and weighed for at least 10 samples at each step. The denier under tension is then calculated. For determining the denier under relaxed conditions, the cut fibers were subjected to a pretension of 5 gm and the distance between the original marks of 25 cm (under tension) determined. This was also done for at least 10 samples, and the denier under relaxed conditions calculated. The draw ratio for both the cases was then determined. The diameter of the fibers can be theoretically predicted at each step by simple mass balance. The mass flow rate (w) at any step is given as

$$w = \text{density} \times (\text{cross-sectional area of the fiber at any step}) \times (\text{surface velocity at that step})$$

$$= \rho_i \times (\pi d_{fi}^2/4) \times (\pi N D_{ci})$$

where ρ_i = density at ith step, d_{fi} = diameter of the fiber at ith step, D_{ci} = diameter of the cone at ith step, and N = rotational speed. w is known from the feed rate, ρ_i can be measured at each step, D_{ci} and N are known quantities; thus d_{fi} can be easily determined for all the steps. Experimentally, the diameters of all the filaments were measured on a Projectina microscope after the fiber was allowed to relax. At least 30 samples were taken for determination of each value. Knowing the percent shrinkage, the diameter under tension can also be calculated.

Mechanical Properties

Load elongation curves were obtained for all fiber samples using an Instron Universal Testing Machine at 25°C with the gauge length as 5 cm and a crosshead speed of 10 cm/min. Twenty-five test specimens were taken for each sample, and results averaged. The experimental results were used to determine stress–strain curves which were in turn used to determine tenacity, initial modulus, and elongation at break.

Sonic Modulus

The sonic modulus values for the fibers were measured using a Dynamic Modulus Tester PPM 5. This is a useful measurement as sonic modulus is an indirect measure of amorphous orientation in the fiber samples.[8]

Crystallinity

Degree of crystallinity was determined by measuring density values in a density gradient column. The amorphous and crystalline densities were taken to be 1.080 g/cc and 1.235 g/cc, respectively.[9]

Birefringence and Orientation

The birefringence (Δn) values were determined by the retardation method using a compensator fitted to a polarizing microscope. Herman's crystalline orientation function (f_c) was determined by wide angle X-ray diffraction.

Birefringence of an oriented crystalline polymer is given as

$$\Delta n = X_c f_c \Delta n_c^0 + (1-X_c) f_a \Delta n_a^0$$

where X_c is the degree of crystallinity and f_c and f_a are the crystalline and amorphous orientation functions, respectively. Δn_c^0 and Δn_a^0 are the intrinsic birefringence values for perfectly oriented cyrstals and amorphous polymer. In the above equation Δn, X_c, f_c are determined experimentally while Δn_c^0 and Δn_a^0 are available from the literature, and thus f_a can be easily calculated.

RESULTS

In this section results are mainly presented for IDP-1 or IDP-2 arrangements and compared with those obtained with CDP. Some comparisons between IDP-1, IDP-2, and IDP-3 arrangements are also made. Figure 4 shows the variation of filament diameter along the steps with IDP-2 arrangement as obtained theoretically with density correction along with experimentally obtained values both under tension as well as under relaxed conditions. The excellent agreement between the predicted and experimental values clearly demonstrates the successful operation of the incremental drawing process. The draw ratios obtained for IDP-2 under tension and relaxed states are presented in Table I. Here again it can be seen that the draw ratio values under tension matches very closely to the theoretical values and as expected go down a bit when the fiber is relaxed. The value of draw ratio under tension obtained at the highest step was 4.1 which is equal to the maximum possible for the cones used in the present setup. Higher draw ratios would be possible with cones that are designed to give a higher draw ratio. Work is currently in progress to make cones to give draw ratios as high as 6*X*. The results for IDP-1 and IDP-3 are similar in

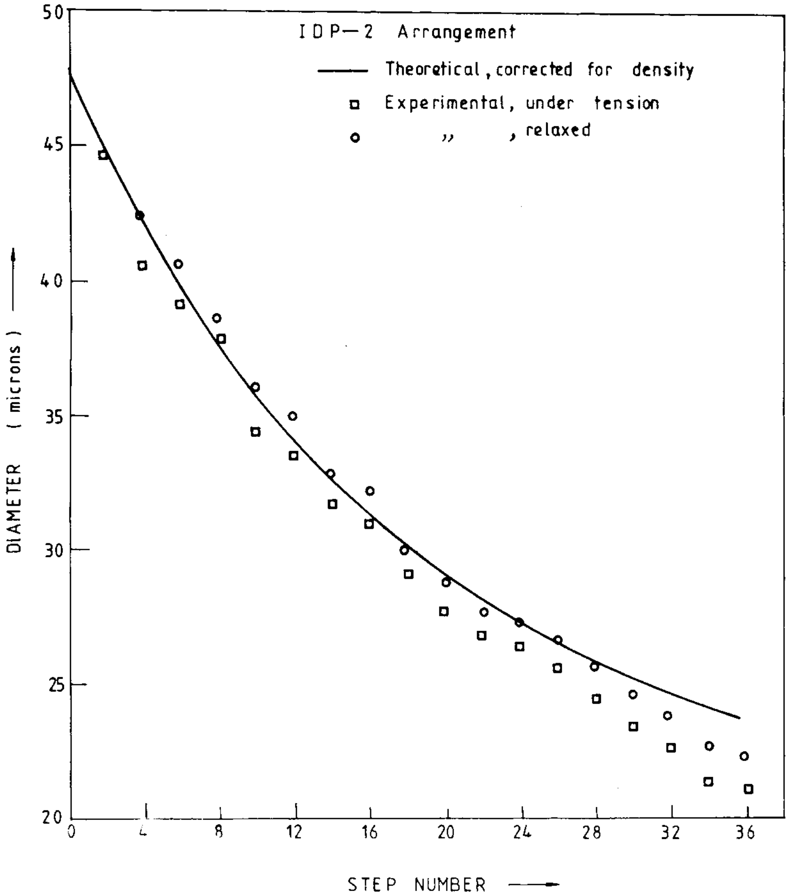

Fig. 4. Plot of fiber diameter as a function of step number for IDP-2 arrangement.

TABLE I
Variation of Draw Ratio with Step Number for IDP-2

Step no.	Draw ratio Theoretical	Under tension	Relaxed
Spun	1.00	1.00	1.00
4	1.27	1.22	1.12
8	1.62	1.62	1.53
12	1.98	1.89	1.75
16	2.33	2.30	2.18
20	2.69	2.69	2.59
24	3.04	2.99	2.80
28	3.40	3.50	3.29
32	3.76	3.90	3.55
36	4.11	4.09	3.63

nature. The draw ratios used for the CDP were 1.24, 1.97, 3.33, and 3.89, which were chosen to be in the range of draw ratios obtained in IDP.

The experimentally obtained values for tenacity, initial modulus, and elongation at break are presented in Figures 5, 6, and 7, respectively. Tenacity and initial modulus curves follow similar trends in that they increase with draw ratio and have higher values for the IDP than for CDP. Furthermore, at high draw ratios the IDP-1 arrangement shows highest values followed by IDP-2 and IDP-3. Another interesting feature in the plots is that the values for IDP show an increasing trend at the highest draw ratio while that for CDP appear to be levelling off. Hence when higher draw ratios could be obtained, the differences between IDP and CDP would be even higher. The elongation-at-break (Fig. 7) shows higher values for IDP

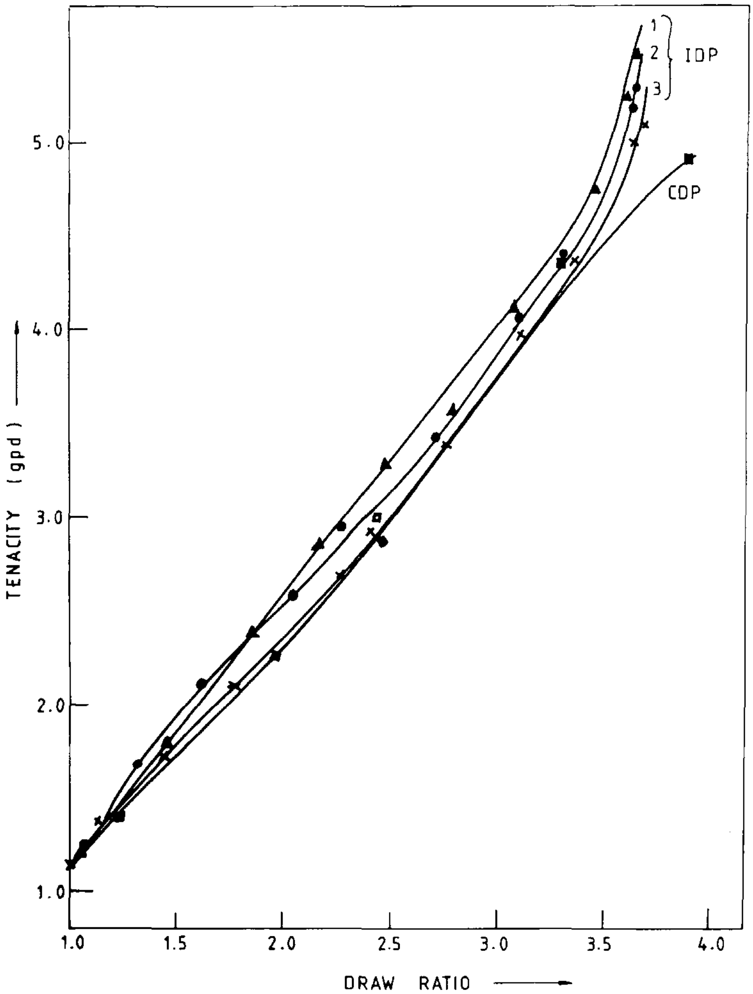

Fig. 5. Plot of tenacity versus step number as a function of draw ratio for IDP and CDP. The values for tenacity and modulus can be converted from grams per denier (gpd) to SI units (newtons per tex) by the relationship: 1 newton/tex = 11.3 gpd.

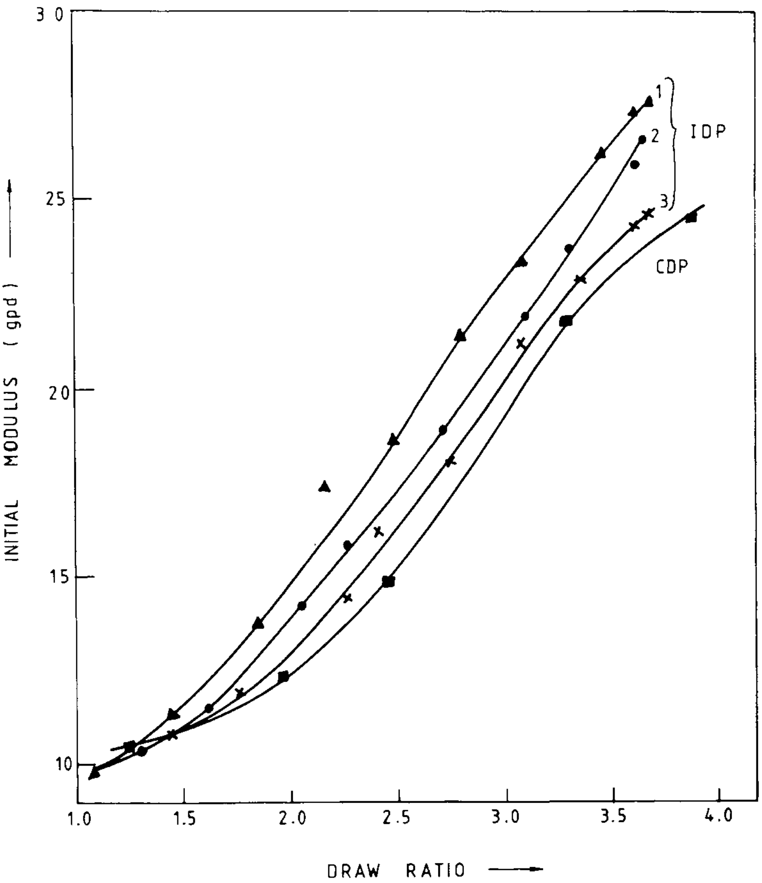

Fig. 6. Plot of initial modulus as a function of draw ratio for IDP and CDP. The values for tenacity and modulus can be converted from grams per denier (gpd) to SI units (newtons per tex) by the relationship: 1 newton/tex = 11.3 gpd.

than for CDP which is contrary to expectations as will be discussed later. Figure 8 shows the variation of sonic modulus with draw ratio for IDP-1, IDP-2, IDP-3, and CDP. As seen earlier, here again the IDP samples show higher values than CDP at equivalent draw ratios, and IDP-1 shows the highest values followed by IDP-2 and IDP-3 at high draw ratios. At lower draw ratios IDP-2 shows higher tenacity and sonic modulus than IDP-1 with a crossover at a draw ratio of about 2.0 while the other trends are the same as before.

The changes in degree of crystallinity and birefringence as a function of draw ratio are shown in Figures 9 and 10, respectively. For IDP the crystallinity rises rapidly at first then slowly and finally increasing rapidly again, whereas for CDP it rises slowly at first then rapidly and slowing down again at higher draw ratios. Birefringence shows uniform increase with draw ratio for all cases. For both crystallinity and birefringence, the IDP samples show higher values than CDP at equivalent draw ratios. IDP-1 shows highest values followed by IDP-2 and IDP-3 at higher draw ratios

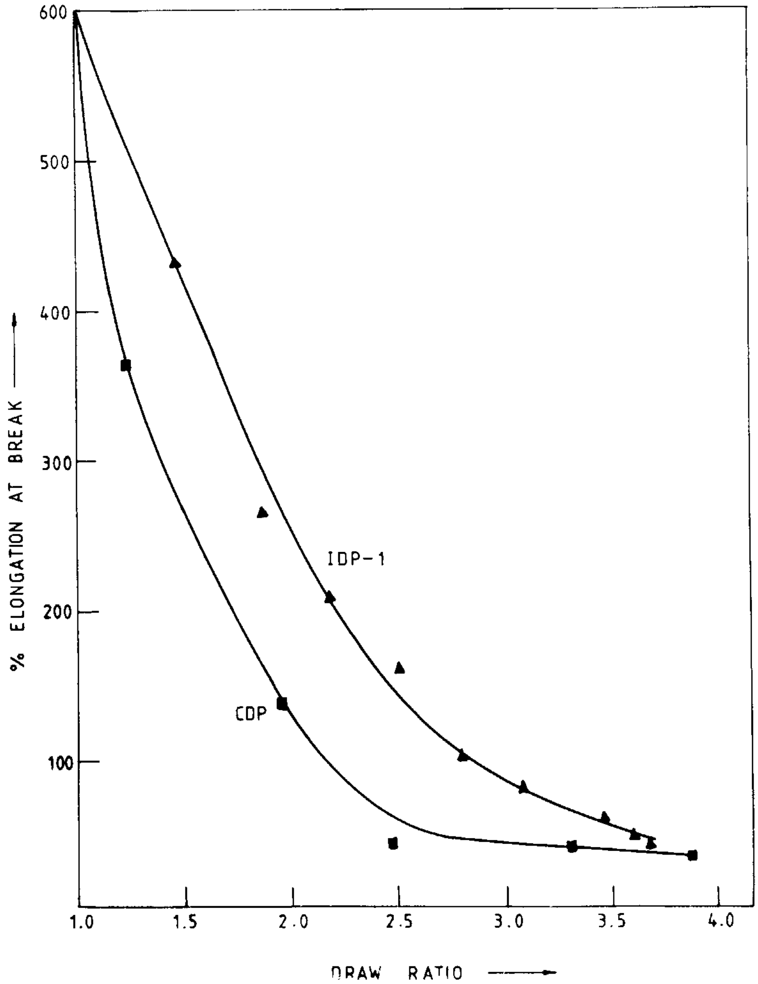

Fig. 7. Plot of elongation-at break as a function of draw ratio for IDP and CDP.

while IDP-2 has higher values than IDP-1 at low draw ratios with a crossover at a value of about 2.0.

Values for Herman's crystalline orientation factor (f_c) were determined only for IDP-2 and CDP samples and are plotted as a function of draw ratio in Figure 11. As described earlier, values for amorphous orientation factor (f_a) were calculated and are plotted in Figure 11. It can be seen that both f_c and f_a have higher values for IDP than CDP samples. The differences in f_a values are greater than those in f_c at high draw ratios.

DISCUSSION

It has been shown that drawing by the novel incremental process produces fibers of higher tenacity and modulus (Figs. 5 and 6) when compared to the conventional process at equivalent draw ratios. These observations are in agreement with the crystallinity and birefringence results (Figs. 8 and 10).

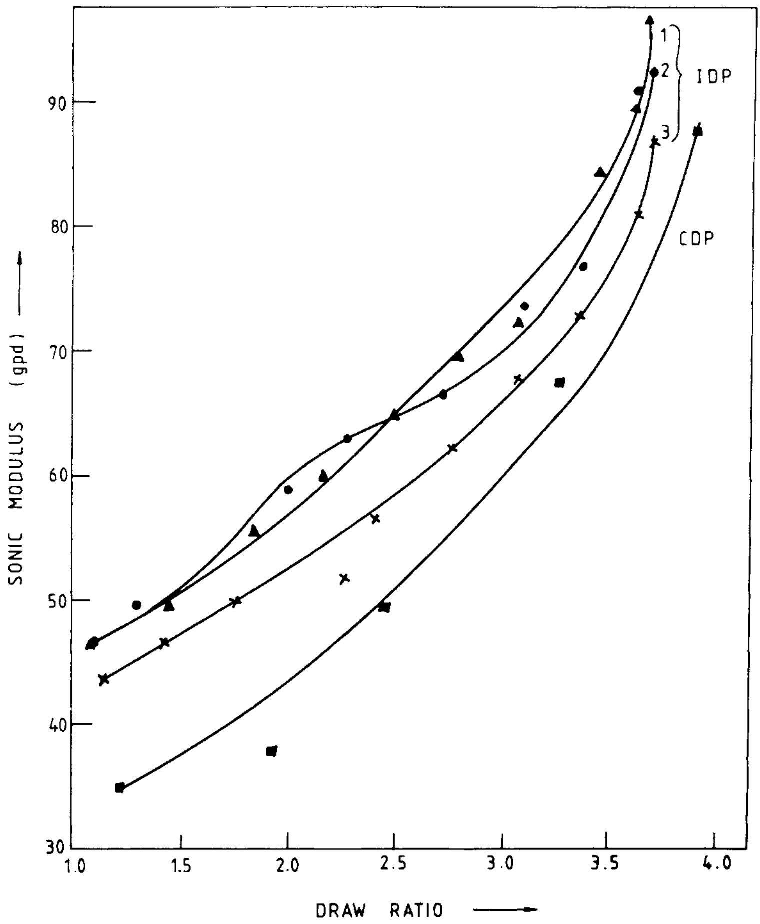

Fig. 8. Plot of sonic modulus as a function of draw ratio for IDP and CDP. The values for tenacity and modulus can be converted from grams per denier (gpd) to SI units (newtons per tex) by the relationship: 1 newton/tex = 11.3 gpd.

The higher tenacity and modulus values for IDP can thus be attributed to a better crystalline order and higher orientation. At equivalent drawing speed, the fiber spends a much greater time for drawing in IDP than in CDP. For example, at 60 m/min the drawing residence time is 50 s for IDP-1, 30 s for IDP-2, and 21 s for IDP-3 and only a fraction of a second for CDP. Thus in the case of IDP the fibers are drawn in small stages, which result in much more gradual changes in molecular structure over the drawing period as compared to CDP at equivalent draw ratios and drawing speeds.

It is well known that on drawing crystalline polymers the initially existing spherulitic structure changes to a microfibrillar structure.[10-14] In the case of CDP the abrupt changes at the neck do not allow the polymer chains sufficient time to unfold and flow past each other. This would thus result in lesser orientation and weaker links between the fibrils. On the other hand, in IDP the polymer chains get sufficient time between the stages to

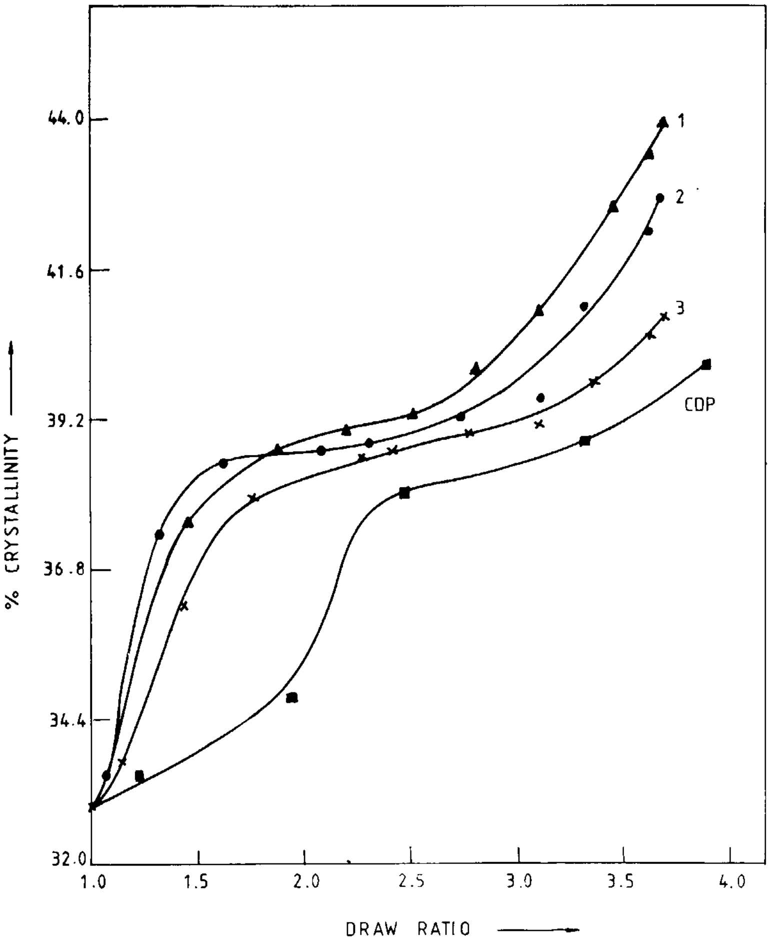

Fig. 9. Plot of degree of crystallinity as a function of draw ratio for IDP and CDP.

align themselves better and thus result in higher overall orientation as seen in the birefringence results (Fig. 10). The contributions to this come both from crystalline as well as amorphous regions. Figure 11 shows that the orientation factors from both these regions are higher for IDP than for CDP. It is generally accepted that the interfibrillar region is amorphous and an improved order in this region would improve the fiber strength. Furthermore, it is known that sonic modulus of an oriented film or a fiber is related to the orientation of the structure and is an indirect measure of amorphous orientation.[8] The sonic modulus results (Fig. 8) also show high values for IDP than CDP and are supportive of birefringence as well as orientation factor results.

An interesting observation was that the IDP samples show higher elongation at break than CDP samples. This is probably because in CDP not only are the molecules less oriented but they are also highly entangled, which results in their breakage at lower elongation values. In IDP samples the amorphous regions are more oriented and probably less entangled, thus allowing mobility of chains in the amorphous regions, resulting in higher

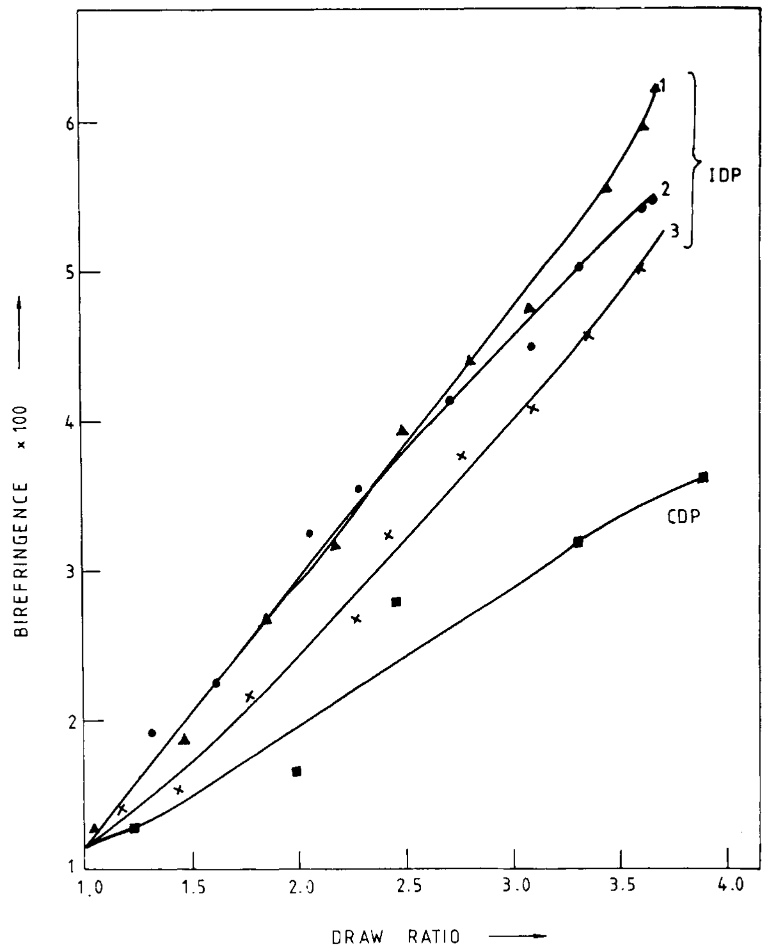

Fig. 10. Plot of birefringence as a function of draw ratio for IDP and CDP.

orientation rather than breakage. Breakage in a fiber generally would occur due to failure in the amorphous regions as these are the weak links in the morphology. The fiber cannot be stronger than the weakest link in the structure. Thus in the IDP samples the amorphous regions have higher orientation and order providing stronger links between the crystalline fibrils than in CDP samples. This becomes a governing factor for the observed higher mechanical properties for IDP fibers than for CDP fibers.

The differences between IDP-1 and IDP-2 are primarily due to the draw–relax–draw sequence in the former and draw–draw–draw sequence in the latter. In IDP-1 the fiber at the earlier steps would relax and give slightly lower values for mechanical properties, crystallinity, and birefringence than in IDP-2. However, at latter steps the draw–relax sequences results in a more uniform structure thus giving higher property value for IDP-1. IDP-3 shows lower property values than both IDP-1 and IDP-2 primarily because of lesser number of steps. In a limiting case as the number of steps are reduced the IDP would approach results obtained for CDP.

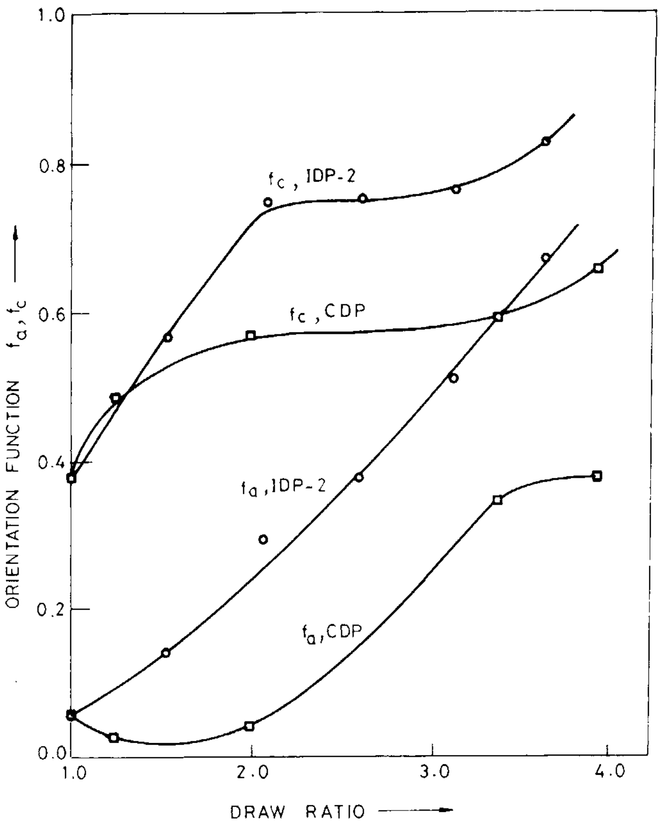

Fig. 11. Plot of crystalline and amorphous orientation functions as a function of draw ratio for IDP-2 and CDP.

Currently work is under progress to investigate the full potential of this new method of drawing. Future work consists of determining the maximum attainable draw ratio, effect of drawing rate, drawing at elevated temperature and drawing to produce ultradrawn fibers[14,15] by the IDP.

CONCLUSIONS

1. The drawing of nylon-6 by the novel IDP has been demonstrated, and it has been shown that IDP produces superior fiber properties than CDP.
2. IDP offers an excellent method for studying the development of structure in a fiber being drawn, as samples are available for analysis from each incremental step.
3. The structural studies show a greater degree of orientation, especially in the amorphous regions, for IDP which in turn results in higher mechanical properties. Results have been explained in terms of a higher drawing residence time without sacrifice in drawing speeds.
4. The IDP shows excellent possibilities for commercial exploitation.

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Received May 25, 1984
Accepted April 12, 1985