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The effect of processing parameters on the mechanical properties of auxetic polymeric fibers

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Abstract Auxetic polymeric fibers have been produced using a melt-spinning technique. The effect of the processing parameters on the fibers has been examined. It was found that the auxetic effect occurs over a very tight temperature window with screw speed, take-off speed and die geometry affecting homogeneity and auxeticity. This is an important finding as it provides a method of producing more homogeneous auxetic fibers with tailored values of Poisson's ratio.

Introduction

In recent years there has been an increased general interest in a group of materials, the properties of which have been described as auxetic [1]. An auxetic material is one in which the Poisson's ratio, ν, is negative, i.e. an applied tensile strain in the longitudinal direction results in a positive strain in the transverse direction. It has been found that a number of naturally occurring materials are auxetic in behavior, for example, a range of crystalline structures including iron pyrites [2, 3] and some forms of skin [4] and cancellous bone [5] structures, though it should be noted that Poisson's ratios for bone are usually reported as positive [6]. However, it is also possible to engineer auxetic behavior and this has been shown to be possible for a number of synthetic materials. These have included polyurethane and metallic foams [7–12], composite laminates [13] and a range of microporous polymers namely polytetrafluoroethylene (PTFE) [14], ultrahigh molecular weight polyethylene (UHMWPE) [15], nylon [16], polypropylene (PP) [17], and more recently polyester [18].

Considering engineered auxetic materials, an initial mechanism for the negative Poisson's ratio behavior was produced for the foam structures developed [19, 20]. Within these foam systems, a conventional cell structure may be described using a two-dimensional analogue of a hexagonal honeycomb as shown in Fig. 1(a). In this case, the strain applied in one direction would result in a reduction in the orthogonal dimension as a result of hinging of the ribs. Analysis of this strain/strain data would indicate a positive Poisson's ratio. Following a compression/thermal treatment process, a re-entrant structure is produced having a two-dimensional analogue of a bow-tie honeycomb as shown in Fig. 1(b). Here an applied strain in one direction would initially result in an increase in the orthogonal dimension as a result of hinging of the ribs. Here, analysis of the strain/strain data would indicate a negative Poisson's ratio. Considering the polymers, it was found that, for larger cross-section cylinders, the auxetic effect is due to the complex microstructure [14–17]. This is essentially a network structure, which has been described as a system of nodules interconnected by fibrils and the mechanism for this effect is well defined [21, 22]. In essence, the re-entrant structure of the foams had been reproduced by the nodule fibril structure [17], the schematic structure for which is shown in Fig. 2 and a similar deformation mechanism was noted. In this case, it is the fibrils which are hinging, though, rather than the cell ribs as is seen in the foams. This hinging of the fibrils in turn causes the nodes to translate, producing the auxetic effect. It was also noted

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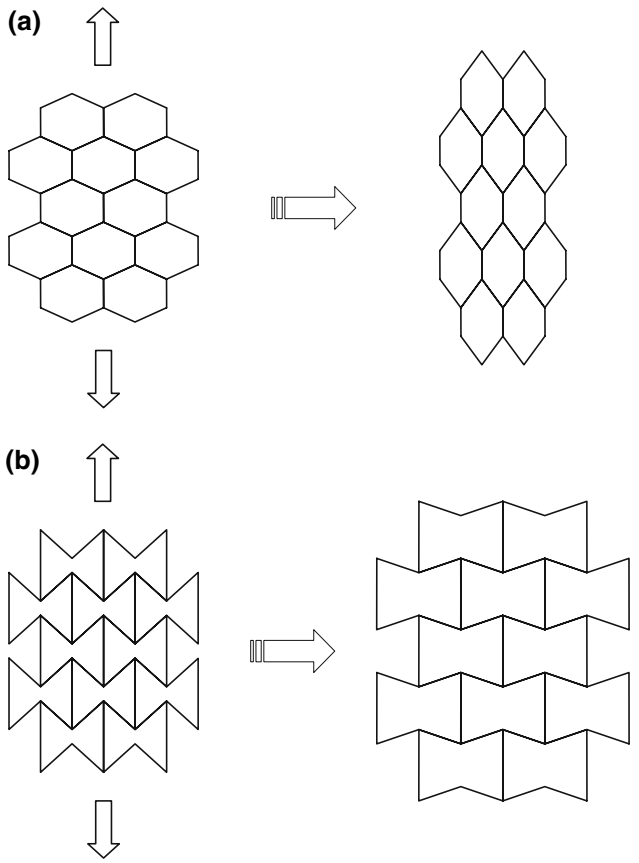

Fig. 1 Two-dimensional schematic diagram of (**a**) conventional (hexagonal) and (**b**) re-entrant (bow-tie) honeycomb models

that there was a variation in the number and size of the fibrils formed during the production of the cylinders for the various polymers, in particular, the degree of fibrillation for PP was much lower than that for PTFE or UHMWPE [17].

In 2001, auxetic fibers were fabricated using a partial melt spinning technique to possess auxetic properties [18, 23, 24]. Polypropylene, polyester and nylon fibers have been produced. Once again, the mechanisms for auxetic behavior are based on the microstructure of the fibers and may be due to a nodule-fibril model based on that discussed earlier [25] but modified to account for the low porosity of these fibers. Figure 3 shows the microstructure and there is evidence of a particulate/granular nature as well as low porosity. This is consistent with the rough morphology of the starting powder and the compaction and partial melting of the particles during the fiber extrusion process. Hence, alternative models based on an interlocked structure [26] have been developed and work is currently underway to assess both the nodule-fibril and interlocked particle models to gain a fundamental understanding of the processing-structure property relationships.

A further consequence of a material possessing a negative ν is that many of the mechanical properties are enhanced. Among these is the resistance to fiber pullout [12], whereby the fiber embedded in a matrix effectively locks into place. The production of auxetic fibers has enabled this concept to be experimentally verified and it has been shown that an auxetic fiber is up to three times more difficult to extract from a matrix than an equivalent conventional fiber [27]. The same concept was also used to develop a press fit fastener [28].

As with all auxetic materials, the processing parameters are crucial in determining if the auxetic effect is produced and to what extent. Detailed studies have been carried out previously on auxetic UHMWPE cylinders and these have shown that the processing temperature, extrusion rate and die geometry need precise definition and control [29–31].

This paper sets out to study variations in processing temperature, screw speed, take off speed and die geometry

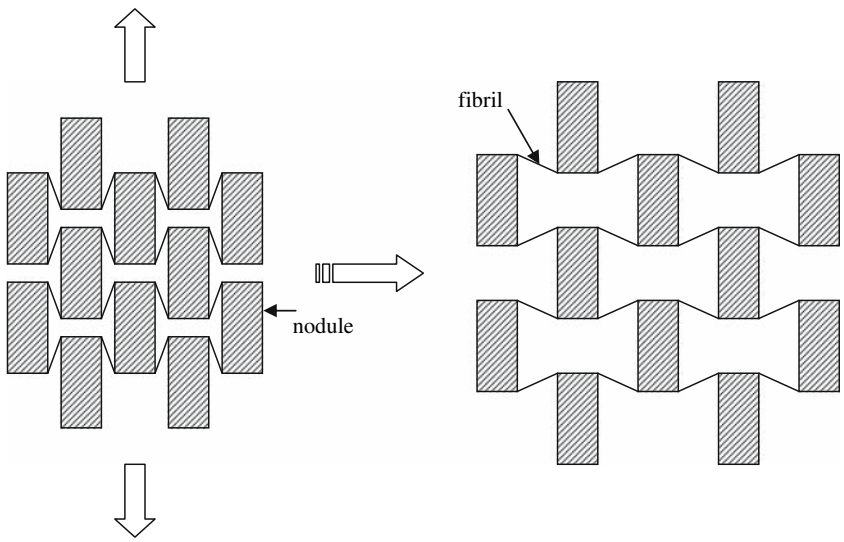

Fig. 2 Two-dimensional schematic diagram of nodule-fibril model for auxetic behavior in polymeric cylinders

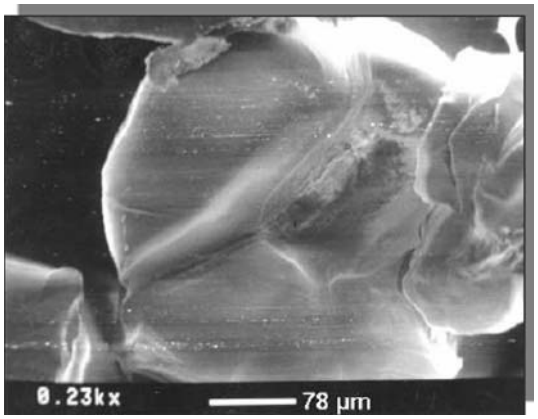

Fig. 3 Micrograph of a typical auxetic fiber

around the defined conditions for producing auxetic polypropylene (PP) fibers, i.e. a processing temperature of 159 °C, screw speed of 1.047 rad s^{-1} (resulting in a throughput of 6 g/min), take off speed of 2 m/min and a spinneret geometry with 40 holes, each of diameter 0.55 mm. These conditions have resulted in fibers with $v = -0.7$ [23]. In addition, the effect of the processing temperature on the auxeticity of nylon and polyester fibers was also investigated. It was found that the auxetic effect occurs over a very tight temperature window with screw speed, take-off speed and die geometry affecting homogeneity and auxeticity. This is an important finding as it provides a method of producing more homogeneous auxetic fibers with tailored values of Poisson's ratio.

Experimental methods

Definition of the processing window

In all three cases considered here, the processing window for the starting powders was defined prior to melt extrusion by performing melt flow index (MFI) and differential scanning calorimetry (DSC) investigations. DSC was used to analyse the polymer powder thermal behavior, in particular the onset of melting. A Polymer Laboratory Thermal Science Heat Flux DSC was used. A small amount of the powder (around 5 mg) was placed in an aluminium pan and heated at 10 °C mm^{-1} with flowing nitrogen at 10 ml/mm maintained at 0.345–0.483 bar pressure. The graphs produced were analysed to obtain the processing window, in this case defined by the onset of melting as surface melting only is required.

Melt flow index was used to investigate the rheology of the polymers, i.e. to confirm that the polymers were able to pass through the extruder at the temperatures suggested by the DSC and be capable of forming a fiber. The MFI system used was a Davenport Serial No. 613. Tests were carried out in accordance with the British Standard [32]. A close fitting piston (0.325 kg) was placed on top of the polymer in the cylinder and left for 4 min at the temperature defined by the DSC. A known weight of 2.16 kg was placed on top of the piston. The amount of polymer forced through the orifice was used to determine the MFI.

Extrusion of the fibers

Extrusion was carried out using an Emerson and Renwick Labline Mark II model with a screw of diameter 25.4 mm, 3:1 compression ratio and with five temperature zones each having individual thermostatic controls, shown schematically in Fig. 4. In order to ascertain if the temperature settings were accurate, external thermocouples were used to monitor each of the individual zones. Any slight variation (±1 °C) was corrected by resetting the thermostatic controls and allowing the extruder to come to temperature. The fibers extruded during these adjustment periods were not used in the analysis since they were produced under fluctuating conditions. Powder is fed into a hopper and is transported through the extruder by the screw. In conventional melt extrusion, the polymer is fully molten. In this process, however, as has been noted partial or surface melting is required.

For the polypropylene fibers, Coathylene PB 0580 powder, produced by Plast-Labor S.A. and supplied by Univar plc, Croydon, UK, was used. Taking the processing parameters already defined for the production of auxetic PP fibers, one variable at a time was examined keeping the other conditions constant. Processing temperatures of 157, 159 and 163 °C, screw speeds of 1.047 and 1.571 rad s^{-1}, take off speeds of 0 (i.e. the draw rollers as shown in Fig. 4 turned off), 2 and 4 m/min and die diameters of 0.55 and 1 mm were used to produce fibers.

The polyester fibers were produced from poly(trimethyleneterephthalate) (with a trade name which will be used in this paper of 3 GT) granules supplied by DuPont, Kinston, North Carolina, USA. The granules were oven dried for 24 h at 108 °C to remove moisture and then subjected to in-house cryogenic grinding to reduce them to powder. MFI measurements revealed that a screw speed of 0.525 rad s^{-1} was required to extrude successfully a fiber, with a take-off speed of 5 m/min and a die diameter of 0.55 mm. From the DSC results, processing temperatures of 215, 220, 225 and 230 °C were used.

For the nylon fibers, powder designated Nylon-R supplied by Nylon Colours, Aylesbury, UK, was used. The powder was oven dried for 2 days under low vacuum at 80 °C and nitrogen gas was injected into the powder during the course of the extrusion to avoid hydrolysis. MFI measurements revealed that a screw speed of 1.05 rad s^{-1}

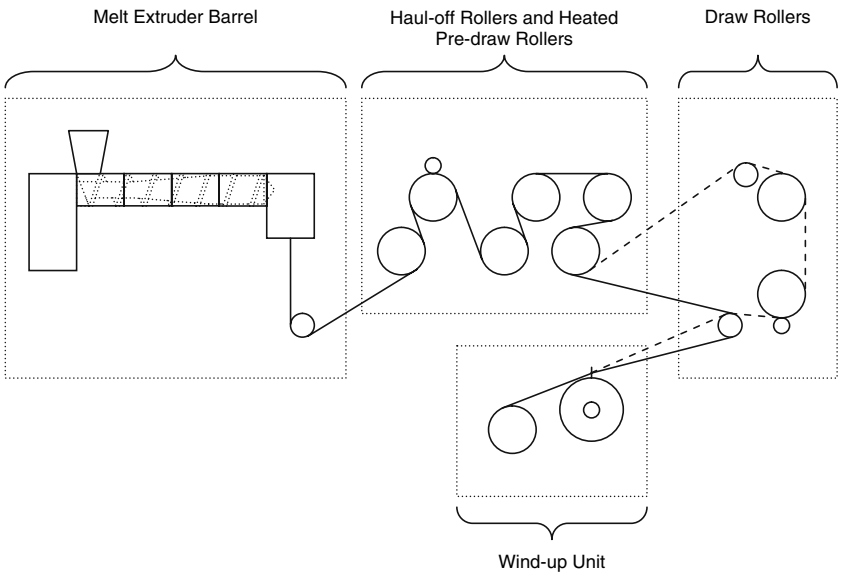

Fig. 4 Schematic of the extruder

was required to successfully extrude a fiber, with a take-off speed of 2 m/min and a die diameter of 0.55 mm. From the DSC results, processing temperatures of 190, 193, 195, 198 and 200 °C were used.

Characterisation of the fibers

The mechanical properties of the extruded fibers were measured using a Messphysik ME 46 video extensometer in combination with a Deben micro tensile tester (see Fig. 5). Video extensometry provides a non-contact method of real time strain measurement. The videoextensometer [18, 33] consists of a computer software package that employs a special algorithm based on the evaluation of the gray contrast between the specimen surface and the targets, in this case fiducial markers. The video based camera (model number 1362CA CCD-CAM) is focussed on the test specimen upon which contrasting markers are attached. The image is digitised and analysed by a computer-operated video processor where the resulting gray scale value (0–255) of each of the 640 × 480 pixels per image is stored in a buffer frame. The buffer data not only help in producing a gray scale diagram for each horizontal scanned/evaluation line and vertical column of pixels but also help in detecting the targets and following them during the test. Scanned lines of the evaluation of the length measurements are interpolated and enhance the system's resolution. When the contrast produced by the target passes from one pixel to another it crosses a gap which produces a slight disturbance to the calculated position. This simultaneous crossing of

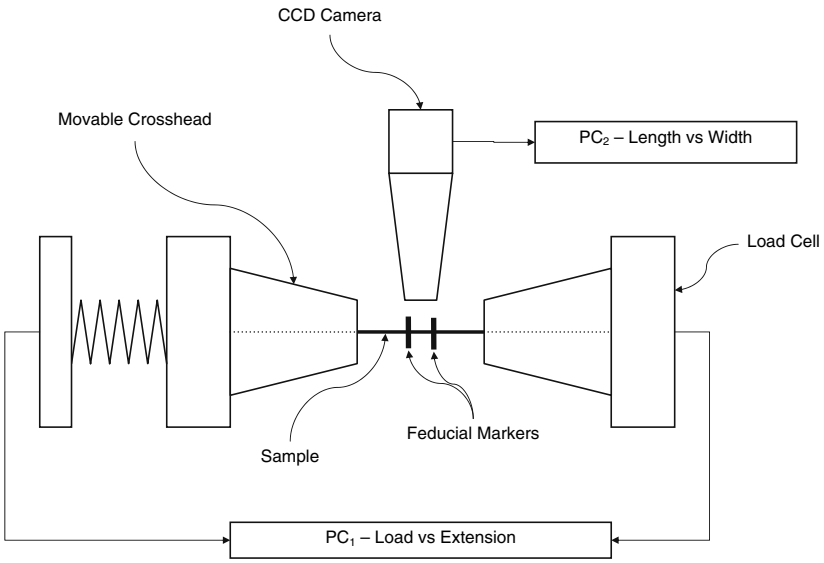

Fig. 5 Schematic of the videoextensometer

gaps causes a wave-like modulation to the strain signal. To eliminate this wave-like modulation the attached targets are slightly inclined (typically up to 5°), thus allowing the target edges to always be exposed to different pixel sections. A mean value of all the horizontal scan lines is determined, resulting in better measurement accuracy, resolution and stability for the length dimensions. The same procedure is performed for the vertical scan lines to evaluate the width variations simultaneously. Fiducial markers were placed on the fiber 1 mm apart. Fiber analysis was carried out by subjecting the fibers to five extension/relaxation cycles at 0.1 mm/min cross head speed up to a maximum of 2% longitudinal strain. Data from the third cycle were used for analysis to produce the Poisson's ratio and Young's modulus. A data sampling rate of 100 data points per second was employed.

Results and discussion

Polypropylene fibers

Figure 6 shows the raw length and width data, plotted against testing time, for a fiber processed with a flat temperature profile of 163 °C. The five testing cycles are shown and it is clearly evident that the width consistently decreases as the fiber is stretched along its length, and increases as the fiber length relaxes to its original dimension. Hence Fig. 6 is consistent with non-auxetic (positive Poisson's ratio) deformation. Figure 7 shows the width and length variations for a fiber processed with a flat temperature profile of 159 °C. In this case the width and length variations are seen to be in phase (i.e. the width increases as the length increases in response to stretching along the length of the fiber). Figure 7 is, therefore, consistent with auxetic behavior. It should be noted that there is a stair-step

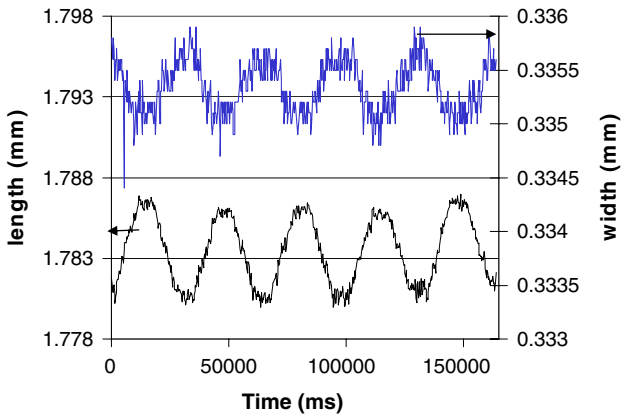

Fig. 6 Length and width variations against time for a polypropylene fiber processed with a flat temperature profile of 163 °C

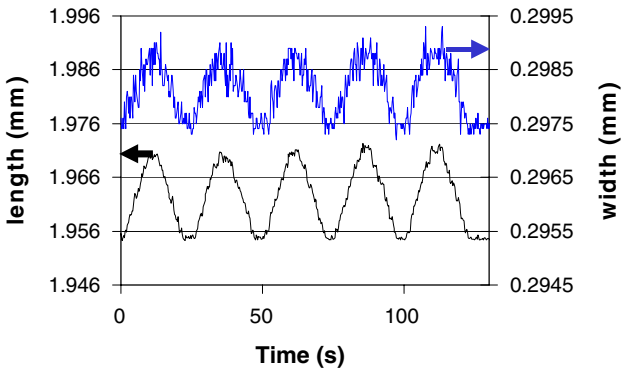

Fig. 7 Length and width variations against time for a polypropylene fiber processed with a flat temperature profile of 159 °C

effect in the fiber width data (see Figs. 6, 7). Each step corresponds to a value of 0.1 μm, which represents the resolution of the experimental set up employed in this work [18].

The relevant properties of fibers processed with a flat temperature profile of 159 °C along the five zones of the extruder barrel, with screw and take-off speeds of 1.05 rad s^{-1} and 2 m/min, respectively, through a die diameter of 0.55 mm are shown in Table 1. The % auxeticity was calculated by dividing the number of negative values of Poisson's ratio by the total number of values of Poisson's ratio for all transverse locations of several fibers processed under these conditions. This then indicates that ~20% of the length of fibers processed under these conditions is auxetic. In order to investigate the effect of processing conditions each parameter was varied in turn, while maintaining all other parameters at the values corresponding to the defined conditions.

Effect of processing temperature

Figure 8 shows the effect of varying barrel temperature on the Poisson's ratio. It is clear that varying the barrel temperature by only a few °C leads to a loss of auxetic behavior. For example, at 157 and 163 °C, there is no auxetic behavior observed. At the optimum temperature of 159 °C, Poisson's ratio values as high as +5 and as low as –2.5 were observed. As seen in Table 2, the fiber diameter

Table 1 Measured properties of PP fibers processed with a flat temperature profile of 159 °C and screw and take-off speeds of 1.05 rpm and 2 m/min, respectively, through a die diameter of 0.55 mm

% Auxeticity	19.30
Thickness (μm)	340
E (GPa)	1.5

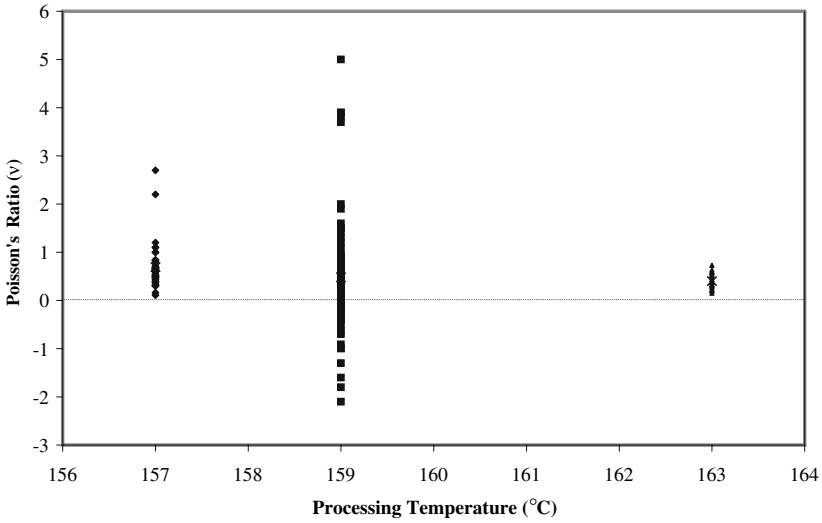

Fig. 8 Graph of Poisson's ratio against processing temperature for the polypropylene fibers

Table 2 The effect of varying the processing temperature on polypropylene fiber mechanical properties

Processing temperature (°C)	Young's modulusus (GPa)	Fiber diameter (μm)	% Auxeticity
157	1.2	200	0
159	1.5	340	19.3
163	3.0	340	0

is largely unaffected by increasing the temperature from 159 to 163 °C, but it does decrease significantly to 200 μm as the temperature decreases to 157 °C. The fiber Young's modulus increases with increasing temperature from 1.2 GPa at 157 °C to 3 GPa at 163 °C. This is consistent with full melting of the polymer powder, as indicated by the conventional nature at 163 °C of the fiber Poisson's ratio.

Effect of take-off speed

The effect of varying the take-off speed is shown in Fig. 9. Varying the take-off speed from 0 (i.e. draw rollers not operating) to 4 m/min still results in auxetic character. Indeed, the range of Poisson's ratio values is almost identical at 0 and 4 m/min, i.e. between approximately +2 and –1.5. At the optimum take-off speed of 2 m/min, there is a larger range of values, i.e. between v = +4.7 and –2.4. The percentage auxeticity is also relatively constant, varying from 19.6% at 0 m/min, to 19.3% at 2 m/min, to 18% at 4 m/min. However, varying the take-off speed leads to a decrease in both the Young's modulus and the fiber diameter. The Young's modulus decreases from 3 GPa at 0 m/min, to 1.5 GPa at 2 m/min and finally to 0.15 GPa at 4 m/min, while the fiber diameter decreases from 600 μm at 0 m/min, to 340 μm at 2 m/min and then

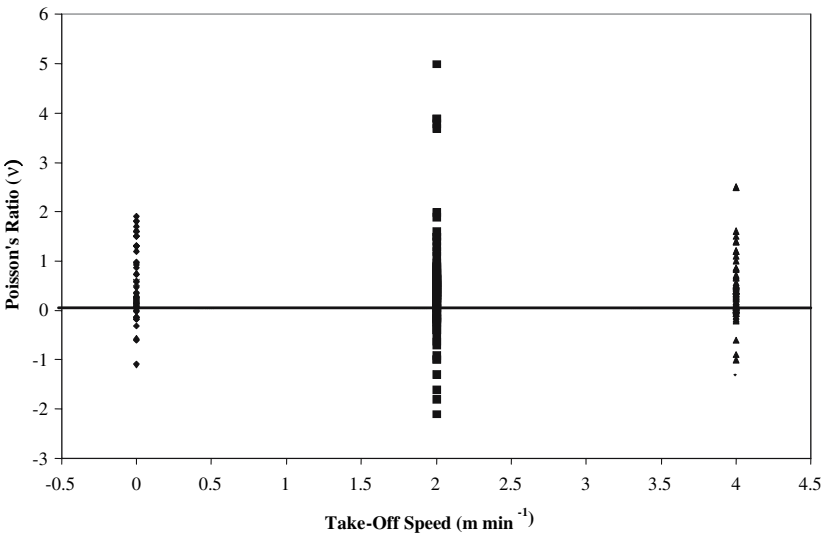

Fig. 9 Graph of Poisson's ratio against take-off speed for the polypropylene fibers

to 170 μm at 4 m/min (see Table 3). Both of these might be expected due to slight drawing out of the fiber structure as take-off speed is increased. Hence take-off speed could potentially be a useful parameter for producing fibers of equivalent auxeticity but different Young's modulus/fiber diameter values according to the requirements of specific applications.

Effect of screw speed

Figure 10 shows the effect of varying screw speed on the fiber Poisson's ratio. Increasing the screw speed leads to a decrease in % auxeticity from 19.3% to 14%. The Young's modulus and fiber diameter also decrease from 1.5 to 1.4 GPa and 340 to 255 μm respectively (see Table 4). It is noteworthy, however, that increasing the screw speed also leads to an increase in the range of negative Poisson's ratio values measured. The decrease in % auxeticity and increase in large negative values of Poisson's ratio present are both consistent with an internal fiber microstructure adopting a geometry close to the transition geometry from auxetic to non-auxetic behavior. The decrease in % auxeticity is also consistent with the increased screw speed not allowing sufficient time for sintering to occur within the extruder barrel. It is known that sintering time is important in producing auxetic functionality in auxetic microporous cylinders [30].

Effect of die diameter

Figure 11 shows the effect of varying die diameter from 0.55 mm (40-hole spinneret plate) to 1 mm (monofilament spinneret plate). In this case comparison is made between two fiber samples produced with a take-off speed of zero, all other parameters are as per the optimum process conditions.

From Fig. 11 it is clear that increasing the die diameter is accompanied by a narrowing of the range of Poisson's ratio values. In addition, the % auxeticity is increased from 19.3% to approximately 26% and the Young's modulus is reduced from 3 to 0.75 GPa. The fiber diameter remains effectively unchanged (see Table 5). In other words the fiber now displays a greater amount of auxetic character of increased uniformity along its length. This then offers significant potential in activities geared toward developing more consistent auxetic fibers, and supports the view that an increase in the die diameter-to-powder particle size ratio is required to achieve improved homogeneity in the structure and mechanical properties of the fiber.

The investigations above have shown that the processing temperature is the most important parameter when engineering auxeticity into a polypropylene fiber.

Table 3 The effect of varying the take-off speed on polypropylene fiber mechanical properties

Take-off speed (m/min)	Young's modulus (GPa)	Fiber diameter (μm)	% Auxeticity
0	3.0	600	19.6
2	1.5	340	19.3
4	0.15	170	18.0

Table 4 The effect of varying the screw speed on polypropylene fiber mechanical properties

Screw speed (rad s^{-1})	Young's modulus (GPa)	Fiber diameter (μm)	% Auxeticity
1.047	1.5	340	19.3
1.571	1.4	255	14.0

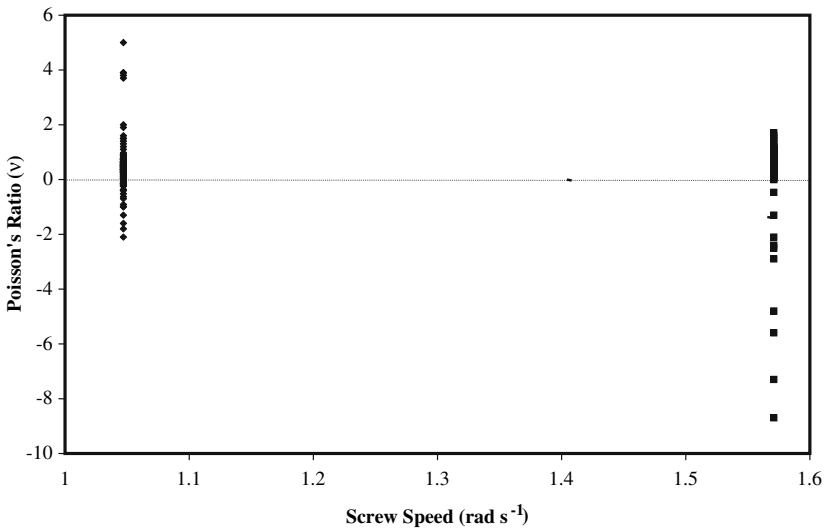

Fig. 10 Graph of Poisson's ratio against screw speed for the polypropylene fibers

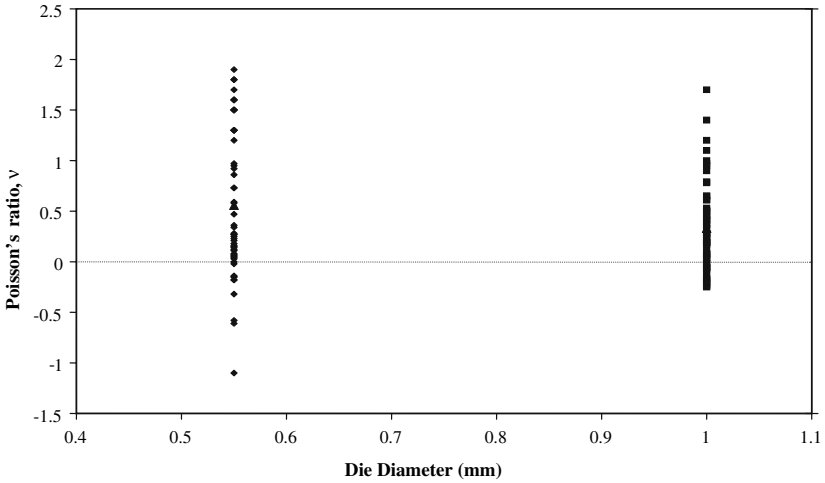

Fig. 11 Graph of Poisson's ratio against die diameter for the polypropylene fibers

Table 5 The effect of varying the die diameter on polypropylene fiber mechanical properties

Die diameter (mm)	Young's modulus (GPa)	Fiber diameter (μm)	% Auxeticity
0.55	3.0	340	19.3
1.00	0.75	340	26.0

Consequently, this key parameter was investigated for polyester and nylon fibers.

Polyester fibers

Figure 12 shows the variation of Poisson's ratio with processing temperature for the polyester fibers. It can clearly be seen that the optimum temperature for producing auxetic behavior is 225 °C. This study confirms that auxetic behavior is found over a very tight temperature window. At 230 °C, for example, all auxetic character is lost. Similarly, although some auxetic character is observed at both 215 and 218 °C, the Poisson's ratio values are predominately 0 (at 215 °C) or positive (at 220 °C). All values of v are negative at 225 °C.

Nylon fibers

Figure 13 shows the variation of Poisson's ratio with processing temperature for the nylon fibers. A range of Poisson's ratio values without any auxetic character were observed at 190, 193 and 200 °C. Auxetic behavior was observed at both 195 and 198 °C, with the higher % auxeticity being observed at 195 °C. So, once again, this points to a very tight processing temperature window.

In all cases investigated, it is the barrel temperature which has the dominant role in processing of auxetic fibers by partial melt extrusion to produce the appropriate connected microstructure for auxetic behavior. A possible explanation based on the interlocked particle model of the microstructure is given in Fig. 14. Figure 14(a) shows a schematic of the microstructural assembly at temperature,

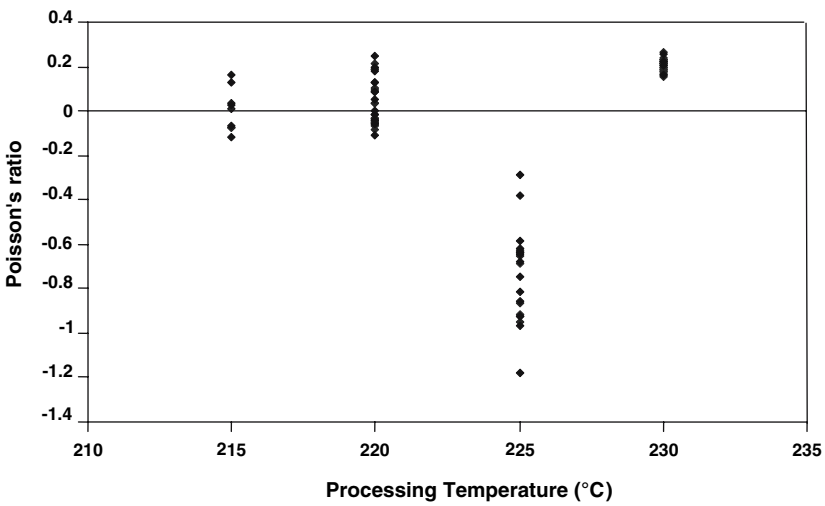

Fig. 12 Graph of Poisson's ratio against processing temperature for the polyester fibers

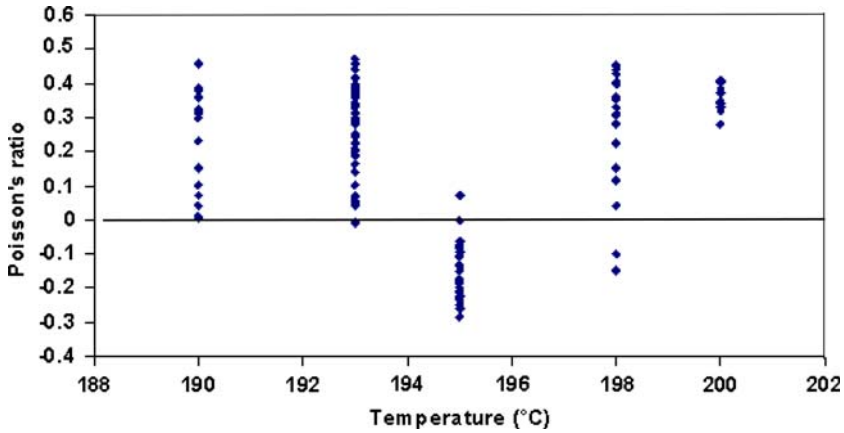

Fig. 13 Graph of Poisson's ratio against processing temperature for the nylon fibers

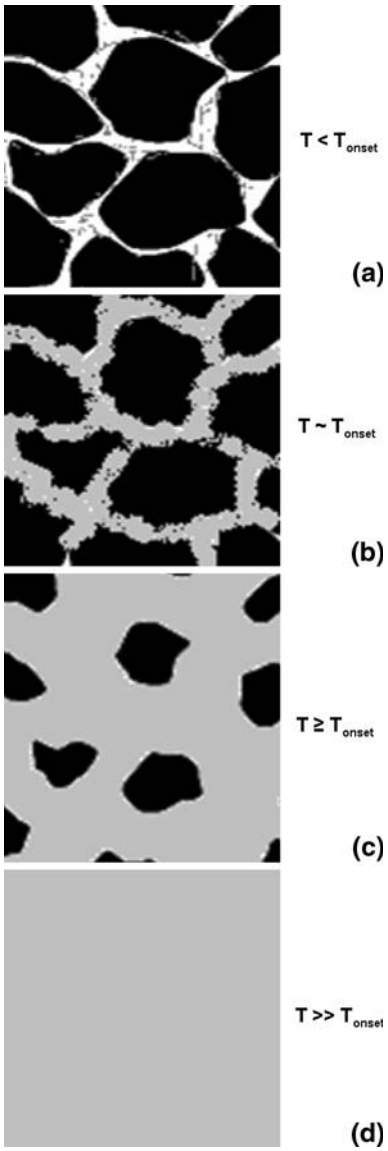

Fig. 14 Schematic proposing the effect of processing temperature in forming appropriate connected network structure

$T < T_{onset}$. It can be seen that the particles are not melted and therefore the formation of a connected surface melted network is not possible. Figure 14(b) shows the network assembly of the fibers at temperature $T \sim T_{onset}$. It can be seen that the particles undergo surface melting resulting in the formation of a network of connected interlocking rough particles that may be the causal mechanism for auxetic behavior in this system. Figure 14(c) shows the network assembly at $T \geq T_{onset}$. It can be seen that the particles undergo a considerable amount of melting, leading to resolidification of the molten regions. The same effect could also be obtained by increasing the processing time at low screw speeds. In these cases, a positive Poisson's ratio is produced. Figure 14(d) shows the network assembly at $T >> T_{onset}$. It can be seen that the particles fully melt and thus a positive Poisson's ratio is expected. An analogous situation also occurs when the second of the possible microstructures and thus mechanisms is considered. In the nodule-fibril case, partial surface melting only is required but not to "glue" loosely the particles together, rather to promote fibril formation [34].

In addition to this, the differential ratio of surface-melted thickness to particle diameter is another factor that affects the formation of the appropriate connected network structure. Large particles undergo partial melting whereas the small particles fully melt. Moreover, the amount of surface melted polymer region at the fiber surface smeared on exiting the die to fill cavities between the outer surface of the edge particles can also affect the network structure for auxetic behavior since the larger the melted polymer region, the lesser the formation of the network structure for auxetic behavior.

Conclusions

From this investigation, the key processing parameter is confirmed to be extrusion temperature which has an

extremely narrow window for all three polymers considered. From the detailed investigation into polypropylene fibers, take-off speed only slightly modifies the % auxeticity of the fiber, indicating that the slight drawing associated with the take-off speed does not adversely effect auxetic functionality for the take-off speeds investigated. This is not true for the fiber diameter and modulus which are both significantly reduced as the take-off speed is increased, thus enabling the possibility of tailoring the Poisson's ratio and Young's modulus/diameter combinations to specific values. The screw speed is also important in imparting auxetic functionality and the range of auxetic values attainable. It is likely that it is necessary to ensure that the screw speed is slow enough to allow sufficient time for sintering, although the effects of screw speed on compaction and shear are also likely to be required before the dependency on this parameter is fully understood. The die diameter appears to offer the most likely route to success in producing more consistent fibers, since it has been found that increasing the die diameter produces an increase in the % auxeticity and a narrower distribution of auxetic values along the length of the fiber.

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