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TUBULAR ARTICLES OF BIAXIALLY ORIENTED POLYMERS

5 This invention relates to tubular articles, such as cylindrical can bodies or bottles, of biaxially oriented crystallisable thermoplastic polymers. It is particularly concerned with the production of such tubular articles from tubular articles of smaller dimensions having at least one open end, such as straight open-ended tubes, tubes with one closed end, or moulded or extruded pre-forms having an approximation to the shape of the article to be produced.

10 It is known to produce tubular articles, such as bottles, of biaxially oriented crystallisable thermoplastic polymers, particularly saturated linear polyesters such as polyethylene terephthalate, by stretch-blowing heated preforms into external
15 moulds, using internal gas pressure introduced through the open end or mouth of the preform. In our copending U.K. Patent Application No. 8037137, published under No. 2089276A, we have described a method of forming biaxially oriented tubular articles which may be used as bodies for processable food containers, the method involving
20 longitudinal stretching of an open-ended tube by mechanical means, which temporarily clamp and seal both ends of a section of the tube, and radial expansion of the tube by internal fluid pressure within an external mould, at a temperature which is normally in the range from 75°C to 120°C. The external moulds employed in these prior processes
25 are costly and have a high heat capacity which makes it impossible to subject the article to a heat treatment, such as annealing, involving rapid changes in temperature, until the article has been removed from the mould, but the use of an external mould has hitherto been thought necessary to achieve accurate dimensioning of the finished article.

30 According to the present invention, a method of producing a tubular article of a biaxially oriented crystallisable thermoplastic polymer from a tubular article of smaller dimensions having at least one

open end, comprises the steps of heating the smaller tubular article to a temperature above the glass transition temperature of the polymer but below its melting temperature, stretching the tubular article axially by mechanical means, and simultaneously or
5 sequentially stretching the tubular article both axially and transversely by internal fluid pressure introduced through said open end, without external constraint.

It has surprisingly been found that this combination of mechanical
10 axial stretching with fluid pressure axial and transverse stretching, in the absence of any external constraint such as a mould, can result in the production of tubular articles of consistent wall thickness in a reproducible manner, while also achieving the good mechanical properties normally provided by biaxial orientation of the polymer.

15 Preferably the method comprises the further step of subsequently heating the stretched and biaxially oriented article to a temperature above the stretching temperature to anneal the polymer, while maintaining an internal fluid pressure. The absence of an external
20 mould or other constraint facilitates such further heating and enables it to be carried out immediately after the stretching without transferring the article to another apparatus. The method also preferably comprises the further step of cooling the annealed article before releasing the internal fluid pressure. Again the absence of
25 an external mould facilitates this operation.

During the annealing and/or cooling, the internal fluid pressure is preferably reduced below that used for the stretching of the tubular article.

30 The axial stretching by mechanical means may, for example, be effected by an external clamp and an internal plug securing one end and a chuck element engaging the other end of the tubular article, the chuck element having an inlet for fluid pressure and being

movable relative to the clamp and plug. Alternatively it may be effected by a chuck element securing said open end and having an inlet for fluid pressure, and a head mounted on a rod which is slidable through, and movable relative to, the chuck element.

Preferably the axial stretching by mechanical means is commenced before the internal fluid pressure is introduced to effect both axial and transverse stretching, so that the mechanical axial stretching is in advance of the axial stretching caused by the internal fluid pressure, e.g. by 10 to 20%.

Application of heat to the article may be continued during the stretching thereof.

In the case where the crystallisable polymer is polyethylene terephthalate, preferably the stretching temperature is in the range from 75°C to 120°C and the internal fluid pressure is provided by compressed gas at a pressure in the range from 7 to 15 bars.

The article may be heated to a temperature in the range from 110°C to 240°C to effect the annealing. The annealed article may be cooled to a temperature of less than 120°C before release of the internal fluid pressure. The reduced internal fluid pressure employed during annealing and/or cooling may be in the range from 2 to 4 bars.

The method of the invention permits the production of tubular articles, in particular open-ended tubes, at high speed and with the good mechanical properties and consistent wall thickness mentioned above. Any minor variations in diameter or departures from coaxiality can easily be corrected by heat setting the tubular article on a mandrel by the method described and claimed in our copending Application No. 8310966.

The invention also resides in apparatus for producing a tubular

article of a biaxially oriented crystallisable thermoplastic polymer from a tubular article of smaller dimensions having at least one open end, comprising means for engaging both ends of the smaller tubular article and for moving said engaging means apart so as to stretch the article axially, means for introducing fluid under pressure through the engaging means which engages the open end into the interior of the smaller tubular article so as to stretch it axially and transversely, the apparatus being characterised by the absence of any means for external constraint of the stretched tubular article.

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Specific embodiments of the invention will now be described by way of example and with reference to the accompanying drawings, in which:-

FIGURE 1 is a diagrammatic longitudinal section showing a straight open-ended tube prepared for stretching in accordance with one embodiment of the invention,

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FIGURE 2 is a similar illustration of the tube after subjection to the stretching process,

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FIGURE 3 illustrates the subsequent annealing of the stretched tube,

FIGURE 4 illustrates the subsequent cooling of the tube,

FIGURE 5 is a similar illustration to Figure 1 of a tube with a closed end, or preform, being prepared for the stretching process,

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FIGURE 6 shows the stretched tubular article and

FIGURE 7 is a graphical representation of the variation in temperature of the outside and inside of the tubular article during an embodiment of the present invention.

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Figure 1 illustrates a straight open-ended tube 10 of a crystallisable thermoplastic polymer, specifically a saturated linear polyester such as polyethylene terephthalate. At one end 11, the tube 10 is clamped between an external stationary clamp 12 and an internal plug 121 on the end of a rod 122. At its other end, the tube 10 is engaged and gripped internally and externally by a movable chuck element 13 having an inlet passage 131 for admitting compressed air to the interior of the tube 10 and being slidably mounted on the rod 122. A generally cylindrical heater 14 is arranged around the tube 10 so as to heat it to a stretching temperature above the glass transition temperature of the polymer but below its melting temperature, specifically a temperature in the range from 75°C to 120°C in the case of polyethylene terephthalate. Alternative sources of heat may be, for example, a hot gas circulated around the tube 10, infra red radiation from electric heaters surrounding the tube, or a dielectric heater. Where radiated heat is used, the tube 10 can be rotated and the heat be directed from one side only.

After the initial heating which raises the surface temperature of the tube 10 to a temperature somewhat above the selected stretching temperature, the application of heat is stopped or reduced so that the temperature throughout the thickness of the tube 10 can be equalised and brought to the selected stretching temperature.

Axial stretching of the tube 10 is then commenced by drawing the chuck element 13 away from the clamp 12 in the direction of the arrow 15. Simultaneously, or very shortly afterwards, gas under pressure, e.g. at a pressure of 7 to 15 bars, is introduced through the inlet 131 to assist the axial stretching and to effect transverse or radial stretching of the tube 10, as shown in Figure 2.

On completion of the axial and radial stretching, which effects biaxial orientation of the polymer material, the fluid pressure is

reduced, e.g. to a pressure in the range of 2 to 4 bars, and heat is applied by a heater 16 as shown in Figure 3 to raise the temperature of the stretched and oriented tube 10 to a temperature above the stretching temperature, e.g. in the range from 110°C to 240°C, to effect annealing of the polymer while still under internal fluid pressure.

Finally, while still maintaining the reduced internal pressure of 2 to 4 bars, the annealed tube 10 is cooled by means of a cooler 17 (Figure 4) e.g. a cylinder with passages 171 for a cooling fluid, or by directing a cold fluid on to its surface, to reduce its temperature sufficiently to ensure that the dimensional stability of the tube 10 will be retained when the internal pressure is removed and the tube is released from the clamp 12 and chuck element 13. The surface temperature is preferably reduced to less than 120°C before release of the internal fluid pressure and the clamp and chuck.

Figures 5 and 6 illustrate the application of the invention to the production of a bottle with a substantially hemispherical bottom from a closed ended tube or preform 20. The preform 20 is secured at its open end 21 by a chuck element 22 having a compressed air inlet 221 and heated as described above with reference to Figure 1. The heaters and cooler are omitted from Figures 5 and 6 for clarity. After equalisation of the temperature throughout the thickness of the preform 20, it is subjected to axial stretching by means of a mechanically operated push rod 23 having an enlarged head 24 and slidable axially through the chuck element 22. At the same time, or preferably after the preform has been stretched to substantially 10 to 20% its desired final length, fluid pressure is introduced through the inlet 221 into the preform 20 to expand it radially and to assist in stretching it axially to the form of the bottle 25 shown in Figure 6.

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Annealing and cooling of the bottle then follow as previously described with reference to Figures 3 and 4, using similar pressures and temperatures.

5 It is found that the surface temperature of the tube 10 or preform 20 increases during the axial and radial stretching, usually to the amount of 10 to 25°C, as a result of the exothermic nature of the stretching process. This increase in temperature can be made use of for the subsequent annealing of the article, whereas in the known
10 processes operated with an external mould it would be lost to the mould.

In a specific example of a preform 20 heated to a stretching temperature of 105°C, the heat developed during the stretching
15 process was found to increase the surface temperature of the biaxially oriented and stretched bottle 25 to about 125°C. Radiated heat subsequently directed on to the surface of the bottle 25 for annealing could either maintain the surface temperature at 125°C during the annealing period or be used to increase it to about 150°C.

20 The method can be carried out extremely rapidly so as to enable a high rate of production to be achieved. In the case of bottles 25 of approximately 350mm length and 80mm diameter, with a mean wall thickness of approximately 0.35mm, produced from preforms 20 of
25 length approximately 120mm, diameter approximately 20mm and mean wall thickness approximately 4.5mm, the stretching of the bottle to shape can be effected about 0.4 to 0.8 seconds with the push rod 23 being moved at a rate of 0.3 to 1m per second and the pressurised fluid being delivered at between 7 to 15 bars. The subsequent annealing
30 treatment may take approximately 5 to 10 seconds and cooling to the required temperature for release of the article may be take approximately 10 - 20 seconds.

The method described above will normally produce stretched and biaxially oriented articles with what are known as "natural draw ratios" as defined by thickness reduction of the polymer. The natural draw ratio in any particular case is determined by the stretching temperature, the orientation (if any) of the unstretched polymer and the polymer viscosity, and occurs at the point at which strain hardening is encountered. It is possible to increase the axial stretch ratio by the mechanical stretching means (chuck element 13 or push rod 23). The following Tables 1 and 2 illustrate the results obtained when producing bottles 25 from preforms 20 as illustrated in Figures 5 and 6, while varying the stretched length by means of the push rod 23. It will be seen that as the stretched length increases the diameter tends to decrease but the mean wall thickness remains substantially constant.

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TABLE 1Preform

5 Mean diameter = 20.5m
 Mean Wall Thickness = 4.5mm
 Stretchable Length = 121mm

Stretched Bottle - Various Axial Extensions

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Stretch Temp- erature	Stretch Blow Time	Line Blow Pres- sure	Stre- tched Length	Diameter Range	Mean Wall Thick- ness Range	Thick- ness Reduc- tion Ratio	Trans- verse Stre- tch Ratio	Axial Stre- tch Ratio
°C	Sec	Bar	mm.	mm.	mm.			
105	0.6	11	325	81.8- 81.1	0.35- 0.36	12.56	4.00	3.13
			335	81.8- 79.5	0.34- 0.35	13.04	3.94	3.31
			350	82.7- 79.6	0.33- 0.35	12.93	3.97	3.25
			370	78.0- 77.0	0.35- 0.37	12.50	3.78	3.30
			380	79.6- 78.0	0.34- 0.36	12.74	3.83	3.33
			390	77.3- 76.4	0.34- 0.36	12.89	3.75	3.44
			410	75.4- 73.2	0.32- 0.35	13.04	3.63	3.59

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TABLE 2

Preform

5 Mean diameter = 21.0mm
Mean Wall Thickness = 4.0mm
Stretchable Length = 132mm

Stretched Bottle - Various Axial Extensions

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Stretch Temp- erature °C	Stretch Blow Time Sec	Line Blow Pres- sure Bar	Stre- tched Length mm.	Diameter Range mm.	Mean Wall Thick- ness Range mm.	Thick- ness Reduc- tion Ratio	Trans- verse Stre- tch Ratio	Axial Stre- tch Ratio
15	105	0.6	11	340	84.3- 81.5	0.34- 0.37	11.33	3.95
				360	82.8- 80.2	0.33- 0.37	11.69	3.87
				385	82.7- 80.5	0.33- 0.36	11.63	3.87
				410	80.5- 79.6	0.31- 0.33	12.74	3.80
20								
25								

30 Table 3 illustrates results obtained while maintaining the stretched length substantially constant, and illustrates the consistency of the results obtained, in spite of the absence of any external constraint such as an external mould. The stretched lengths, diameter and wall thickness can in fact be controlled within narrow limits, with a diameter variation along the length of the bottle of not more than



2.5mm and a wall thickness variation of not more than 0.04mm, in the bottles referred to in the Tables above.

TABLE 3

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Preform

Mean diameter = 20.5mm

Mean Wall Thickness = 4.5mm

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Stretchable Length = 121mm

Stretched Bottle - Same Axial Extensions

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Stretch Temp- erature °C	Stretch Blow Time Sec	Line Blow Pres- sure Bar	Stre- tched Length mm.	Diameter Range mm.	Mean Wall Thick- ness Range mm.	Thick- ness Reduc- tion Ratio	Trans- verse Stre- tch Ratio	Axial Stre- tch Ratio
105	0.6	13	385	78.3- 80.2	0.33- 0.35	13.63	3.87	3.52
			380	81.1- 79.6	0.33- 0.34	13.63	3.90	3.49
			380	81.1- 78.6	0.33- 0.35	13.63	3.88	3.51
			380	80.8- 78.3	0.33- 0.34	13.63	3.87	3.52
			380	81.8- 78.9	0.32- 0.36	13.63	3.92	3.48

The results listed in Tables 1, 2 and 3 were all obtained with the same rate of mechanical axial extension, the push rod 23 being extended at 0.46 m.sec^{-1} .

- 5 In the following Table 4, results are given for experiments with varying rates of mechanical axial extension, and for stretching by blowing without mechanical extension for comparison. This Table also lists the results of tests of the modulus and yield stress of the bottle side wall material after stretching, measured in the
- 10 transverse and axial directions. For comparison, the amorphous unoriented polyethylene terephthalate material had a tensile modulus of 2200 MPa and a yield stress of 55 MPa.

- 15 It will be seen that the additional mechanical stretch in the axial direction results in a greater reduction in wall thickness than stretching by blowing alone, and the axial yield stress in particular improves with higher rates of mechanical extension.

Preform

Mean Diameter

= 21.0 mm

Mean Wall Thickness

= 4.0 mm

Stretchable Length

= 132 mm

TABLE 4Stretched Bottle

Stretch Temperature	Stretch Blow Time	Line Blow Pressure	Stretched Length	Mechanical Axial Stretch Rate	Diameter Range	Mean Wall Thickness Range	Thickness Reduction Ratio	Transverse Stretch Ratio	Axial Stretch Ratio	Transverse Tensile Modulus	Transverse Yield Stress	Axial Tensile Modulus	Axial Yield Stress
105	0.6	11	295	-	85.9-82.7	.37 -.45	9.87	4.00	2.47	3705	154	2634	83
			320	0.50	84.3-81.8	.31 -.35	12.16	3.97	3.06	3827	158	2470	91
			350	0.32	83.4-80.2	.30 -.34	12.46	3.88	3.21	4359	154	3105	101
			380	0.32	78.0-75.8	.32 -.36	11.40	3.66	3.11	3250	125	3315	116
			380	0.55	80.5-77.7	.24 -.28	15.38	3.77	4.08	3759	136	3269	112
95	0.6	11	280	-	83.4-80.2	.39 -.47	9.21	3.91	2.36	4322	161	2527	84
			340	0.32	81.2-78.6	.33 -.37	11.49	3.75	3.06	4014	154	2779	89
			340	0.50	77.2-74.0	.32 -.36	11.76	3.61	3.26	3412	124	3246	99
			330	0.32	78.6-76.0	.32 -.36	11.80	3.65	3.23	4387	148	2990	88
			385	0.55	76.6-73.2	.29 -.31	13.33	3.57	3.73	3881	136	3539	114



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CLAIMS

1. A method of producing a tubular article of a biaxially oriented crystallisable thermoplastic polymer from a tubular article of smaller dimensions having at least one open end, comprising the steps of heating the smaller tubular article to a stretching temperature above the glass transition temperature of the polymer but below its melting temperature, stretching the tubular article axially by mechanical means, and simultaneously or sequentially stretching the tubular article both axially and transversely by internal fluid pressure introduced through said open end, without external constraint.
2. A method according to Claim 1 comprising the further step of subsequently heating the stretched and biaxially oriented article to a temperature above the stretching temperature to anneal the polymer while maintaining an internal fluid pressure.
3. A method according to Claim 2 comprising the further step of cooling the annealed article before releasing the internal fluid pressure.
4. A method according to any one of the preceding Claims wherein the axial stretching by mechanical means is effected by an external clamp and an internal plug securing one end and a chuck element engaging the other end of the tubular article, the chuck element having an inlet for fluid pressure and being movable relative to the clamp and plug.
5. A method according to any one of Claims 1 to 4 wherein the axial stretching by mechanical means is effected by a chuck element securing said open end and having an inlet for fluid pressure, and a head mounted on a rod which is slidable through, and movable relative to, the chuck element.

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6. A method according to any one of the preceding Claims wherein the axial stretching by mechanical means is commenced before the internal fluid pressure is introduced to effect both axial and transverse stretching so that the mechanical axial stretching is in advance of the axial stretching caused by the internal fluid pressure..
7. A method according to any one of the preceding Claims wherein application of heat to the article is continued during the stretching thereof.
8. A method according to any one of the preceding Claims wherein the crystallisable polymer is polyethylene terephthalate, the stretching temperature is in the range from 75°C to 120°C and the internal fluid pressure is provided by compressed gas at a pressure in the range from 7 to 15 bars.
9. A method according to Claim 8 as appendant to Claim 2, wherein the article is heated to a temperature in the range from 110°C to 240°C to effect the annealing.
10. A method according to Claim 8 or 9 as appendant to Claim 3 wherein the annealed article is cooled to a temperature of less than 120°C before release of the internal fluid pressure.
11. A method according to any one of Claims 8 to 10 as appendant to Claim 4 wherein the reduced internal fluid pressure employed during annealing and/or cooling is in the range from 2 to 4 bars.
12. A method of producing a biaxially oriented tubular article of a crystallisable thermoplastic polymer, substantially as hereinbefore described with reference to the accompanying drawings.



13. Biaxially oriented tubular articles produced by a method according to any one of the preceding Claims.

5 14. Apparatus for producing a tubular article of a biaxially oriented crystallisable thermoplastic polymer from a tubular article of smaller dimensions having at least one open end, comprising means for engaging both ends of the smaller tubular article and for moving said engaging means apart so as to stretch the article axially, means for introducing fluid under pressure through the engaging means which
10 engages the open end into the interior of the smaller tubular article so as to stretch it axially and transversely, the apparatus being characterised by the absence of any means for external constraint of the stretched tubular article.

15 15. Apparatus according to Claim 14 wherein the means for engaging both ends of the smaller tubular article comprise an external clamp and an internal plug securing one end and a chuck element engaging the other end of the tubular article, the chuck element having an inlet for fluid pressure and being movable relative to the clamp and
20 plug.

16. Apparatus according to Claim 14 wherein the means for engaging both ends of the smaller tubular article comprise a chuck element securing said open end and having an inlet for fluid pressure, and a
25 head mounted on a rod which is slidable through and movable relative to, the chuck element.

17. Apparatus for producing a tubular article of a biaxially oriented crystallisable thermoplastic polymer from a tubular article
30 of smaller dimensions having at least one open end, substantially as hereinbefore described with reference to Figures 1 to 4 or Figures 5 and 6 of the accompanying drawings.
