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(54) **Polypropylene fibres**

(57) The invention relates to fibres from heterophasic propylene copolymers having at least two  $T_g$  peaks. The fibres are characterised by excellent softness.

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**Description**

**[0001]** The present invention relates to novel fibres comprising propylene copolymers. More particularly, the invention relates to fibres comprising heterophasic propylene copolymers, especially heterophasic propylene copolymers containing a propylene random copolymer and an ethylene rubber copolymer.

**Background of the Invention**

**[0002]** Today, polypropylene is widely used in many fibre and fabric applications. However, it is generally deficient in applications that require high softness. Such applications include nonwoven fabrics for disposable garments or diapers and also for furniture applications.

For soft-end use fibre and fabric applications random copolymers have come into use since they can be processed into fibres and fabrics that exhibit improved softness and drape characteristics compared to fibres and fabrics made from propylene homopolymers.

**[0003]** Increasing the ethylene content of the random propylene copolymers will generally also improve the softness of fibres and fabrics made from these random propylene copolymers. At higher ethylene contents, however, the produced fibres take longer to solidify with the result that they tend to stick together forming coarser fibres before solidification occurs. A fabric from such fibres is more rigid and less soft. Although this could be alleviated by lowering the production speed, to allow more time for the fibres to solidify before they come into contact, it generally becomes uneconomical to process random propylene copolymers having an ethylene content greater than 3.5 wt%.

US 6,218,010 discloses soft fibres from propylene copolymers having from 5-8 wt% of ethylene. The propylene copolymers are described as being an "alloy" from a random propylene copolymer having an ethylene content of 1-5 wt% and a bipolymer having an ethylene content of 10-30 wt%. The propylene copolymer alloy is further described as having a single  $T_g$  peak, this being an indication as to the miscibility of the random copolymer and the bipolymer. The miscibility of the random copolymer and the bipolymer is disclosed to be a prerequisite for spinnability.

**[0004]** It is therefore the object of the invention to provide fibres from propylene copolymers which fibres shall be characterised by excellent softness. The fibres shall be producible in an economical process, i.e. with high production speeds and without causing processing problems.

**[0005]** Surprisingly, the above object is achieved by a fibre comprising a heterophasic propylene copolymer containing

a) 80-99 wt% of a matrix phase comprising a propylene random copolymer with 0.2-15 wt% of ethylene and/or at least one  $C_4-C_8$   $\alpha$ -olefin and

b) 1-20 wt% of a disperse phase comprising an ethylene rubber copolymer with from 20 - 80 wt% ethylene and from 80 - 20 wt% of at least one  $C_3-C_8$   $\alpha$ -olefin,

wherein the heterophasic copolymer has at least two  $T_g$  peaks.

**[0006]** Contrary to what is disclosed in US 6,218,010, the fibres of the present invention are characterised by a heterophasic propylene copolymer having at least two discernible  $T_g$  peaks. Nevertheless, as will be further shown below, these heterophasic propylene copolymers are perfectly spinnable with high uptake speeds and the produced fibres are characterised by excellent softness.

**[0007]** Glass transition temperatures  $T_g$  are determined by Dynamic Mechanical Thermal Analysis (DMTA).

**[0008]** For the ethylene rubber copolymer the ethylene content may range from 20 - 80 wt% preferably from 20 - 50 wt%, Accordingly, the  $C_3-C_8$   $\alpha$ -olefin content may range from 80 - 20 wt%, preferably from 80 - 50 wt%, According to a preferred embodiment the ethylene rubber copolymer is an ethylene propylene rubber (EPR).

EPR's are more cost-effective than ethylene rubbers with higher  $\alpha$ -olefins and they can either be synthesised in the second step of a two-step process, where the first step synthesises the matrix polymer or they can be mixed with the matrix polymer in a separate melt blending step.

According to a further preferred embodiment the heterophasic propylene copolymer contains 2 - 15 wt%, preferably 5 - 12 wt% of the ethylene rubber copolymer. These concentration ranges for the ethylene rubber are preferred, because fibres from heterophasic propylene copolymer with the above amounts of ethylene rubber copolymer offer the best compromise as to spinnability and mechanical properties, which both in general decrease with higher rubber contents, and softness, which generally increases with higher rubber content.

According to a still further embodiment the heterophasic propylene copolymer has an MFR of from 0.1 - 50 g/10 min, preferably 2.5 - 30 g/10 min.

**[0009]** While in general a wide range of MFR's, e.g. from 0.1 to 500 g/10 min, is suitable for the fibres of the invention, the above mentioned ranges are preferred for producing very soft fibres with an economical process.

According to a still further embodiment the heterophasic propylene copolymer has an MFR of from 200 - 2000 g/10 min. The production of melt blown nonwoven fabrics of this invention requires MFR's in the range of from 200 - 2000 g/10 min.

[0010] Heterophasic propylene copolymers having the desired MFR's may be obtained by vis-breaking the low-MFR polymer with e.g. peroxides, or they may be available directly from the polymerisation process by suitable choice of conditions.

[0011] It is preferred for the fibres of the invention to comprise a heterophasic propylene copolymer having an overall ethylene content of from 1.0 - 15.0 wt%.

[0012] The heterophasic propylene copolymers of which the fibres of the invention are comprised preferably show two  $T_g$  temperatures. The first of these glass transition temperatures preferably is in the range of from -15 to +5 °C, more preferably around 0 °C. This first  $T_g$  temperature usually is attributed to the matrix phase and is influenced by the comonomer content of the matrix phase. In the case of ethylene as comonomer it is lower with higher ethylene contents. The second of these glass transition temperatures preferably is in the range of from -35 to -65 °C, more preferably from -40 to -60 °C and most preferably around -50 °C. The second  $T_g$  usually is attributed to the rubber copolymer of the disperse phase and it is influenced by its molecular weight and its ethylene content.

[0013] Diagrams showing the results of DMTA measurements for two heterophasic propylene copolymers which were used for producing fibres according to the invention are shown in Fig. 3 and Fig. 4.

[0014] A particularly preferred embodiment refers to a fibre comprising a heterophasic propylene copolymer which contains 80 - 95 wt% of a matrix phase comprising a propylene random copolymer with from 1.0 - 15.0 wt% of ethylene and 5 - 20 wt% of a disperse phase comprising an ethylene propylene rubber with from 20 - 40 wt% of ethylene and 80-60 wt% of propylene, the heterophasic propylene copolymer having an overall ethylene content of from 4.0 - 12.0 wt% and two distinct  $T_g$  peaks.

Fibres made from these heterophasic propylene copolymer are characterised by superior softness, and contrary to the disclosure of US 6,218,010, the heterophasic propylene copolymer has two  $T_g$  peaks.

[0015] The invention also refers to articles comprising fibres according to the invention. Such fabric articles include, but are not limited to: nonwoven fabrics for hygiene applications such as diapers, medical gowns and masks; woven fabrics for upholstery and clothing; ropes, twines, carpets. The heterophasic propylene copolymers may be used to produce fibres of the following types; continuous fibre, bulked continuous fibre, staple fibre, monofilament fibre, stretch tape, strapping; and nonwoven fabrics which are spunbonded, meltblown, or produced from staple fibre.

[0016] The heterophasic propylene copolymers may be produced by multistage process polymerisation of propylene and ethylene and/or an  $\alpha$ -olefin such as bulk polymerisation, gas phase polymerisation, slurry polymerisation, solution polymerisation or combinations thereof using conventional catalysts. Those processes are well known to one skilled in the art.

A preferred process is a combination of bulk slurry loop reactor(s) and gas phase reactor(s). The matrix polymer can be made either in loop reactors or in a combination of loop and gas phase reactor.

The polymer produced in this way is transferred into another reactor and the disperse phase is polymerised. Preferably this polymerisation step is done in a gas phase polymerisation.

A suitable catalyst for the polymerisation of the heterophasic copolymer is any stereospecific catalyst for propylene polymerisation which is capable of polymerising and copolymerising propylene and comonomers at a temperature of 40 to 110 °C and at a pressure from 10 to 100 bar. Ziegler Natta catalysts as well as metallocene catalysts are suitable catalysts.

One skilled in the art is aware of the various possibilities to produce such heterophasic systems and will simply find out a suitable procedure to produce suitable heterophasic polymer systems which are used in the present invention.

The heterophasic propylene copolymer may also be produced by mixing and melt blending a propylene random copolymer with an ethylene rubber copolymer.

### CR Process

[0017] A heterophasic propylene copolymer prepared as explained above, may be subjected to a controlled rheology (CR) process well known in the art, whereby the copolymer is visbroken into a resin having a narrower molecular weight distribution and lower average molecular weight in order to facilitate fibre spinning. The molecular weight (MW) of the visbroken heterophasic copolymer determines the level of melt viscosity and the ultimate desirable physical properties of the fibre. The MFR of the visbroken copolymer as determined by the MFR test (ISO 1133) may vary within a wide range from fractional to about 2000 g/10 minutes. The CR process is preferably carried out by using organic peroxides, such as 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane). The compounds used in the CR process are added to the polymer and the polymer is, for example, visbroken during the extrusion step.

The CR process may also convert the polymer granules to pellets for easier feeding into the fibre spinning extruder. Additives such as stabilizers, pigments, fillers, antioxidants, ultraviolet screening agents, nucleating agents, certain

processing oils and the like may optionally be added; however, this should not be considered a limitation of the present invention.

### Fibre Spinning Process

**[0018]** The heterophasic copolymer is then drawn to a fine diameter fibre by one of several well known in the art modifications of the basic melt-extrusion fibre process. This process consists of the steps of (1) continuously feeding the heterophasic copolymer to a melting screw extruder; (2) At the end of the screw, a spinning pump meters the melted polymer through a filter to a spinneret where the melted polymer is extruded under pressure through capillaries, typically at a rate of about 0.3-1.0 grams per hole per minute; the capillaries, depending upon the desired fibre product, may vary widely in number, size and shape; (3) solidifying the fibres by transferring the heat to a surrounding medium; and (4) winding of the solidified fibres onto packages. Further processing typically includes orienting the fibres by drawing them to many times their original length. Also, a variety of thermal and texturing treatments well known in the art may be employed, depending on the desired final properties of the fibre. Embodiments of the present invention fibres can be drawn into fine diameter fibres at generally high drawdown speed, without the individual fibres sticking together below the crystallization point.

### Spunbond Fabrics

**[0019]** A particular embodiment of the present invention involves the use of the heterophasic copolymers for spunbonded fabrics. The spunbonding process is one which is well known in the art of fabric production. Generally, continuous fibres are extruded, laid on an endless belt, and then bonded to each other, and often times to a second layer such as a melt blown layer, often by a heated calander roll, or addition of a binder, or by a mechanical bonding system (entanglement) using needles or hydro jets.

A typical spunbond process consists of a continuous filament extrusion, followed by drawing, web formation by the use of some type of ejector, and bonding of the web. First, the heterophasic copolymer is visbroken using peroxide into a resin having a narrower molecular weight distribution and about 25 MFR. During this step the polymer granules are converted into pellets. The pelletised 25 MFR heterophasic copolymer resin is then fed into an extruder. In the extruder, the pellets simultaneously are melted and forced through the system by a heating melting screw. At the end of the screw, a spinning pump meters the melted polymer through a filter to a spinneret where the melted polymer is extruded under pressure through capillaries, at a rate of 0.3-1.0 grams per hole per minute. The spinneret contains up to 6000 capillaries per metre of die width, measuring 0.4-0.6 mm in diameter. The polymer is melted at about 30 °C - 120 °C above its melting point to achieve sufficiently low melt viscosity for extrusion. The fibres exiting the spinneret are quenched and drawn into fine fibres measuring 10 - 40 microns in diameter by cold air jets, reaching filament speeds of up to 5000 metres per minute. The solidified fibre is laid randomly on a moving belt to form a random netlike structure known in the art as web. After web formation the web is bonded to achieve its final strength using a heated textile calander known in the art as thermobond calander. The calander consists of two heated steel rolls; one roll is plain and the other bears a pattern of raised points. The web is conveyed to the calander wherein a fabric is formed by pressing the web between the rolls at a bonding temperature of about 130 °C - 150 °C.

### Measurement methods

#### Crystallisation temperature $T_c$

**[0020]** Crystallisation temperatures are determined by DSC measurement according to ISO 3146 at a cooling rate of 10 K/min after a first heating to 200 °C.

#### Shear thinning index SHI (0/50)

**[0021]** The shear thinning index SHI is calculated from the flow curve  $\eta(\sigma)$  at 200 °C - which can be determined with a capillary rheometer according to ISO 11443 or calculated from the complex shear viscosity determined with a plate-plate rheometer according to ISO 6271-10 using the "Cox-Merz rule" relating the shear viscosity to the dynamic viscosity as described in W.P. Cox & E.H. Merz, J. Polym. Sci. 28(1958) 619-623. The SHI (0/50) is defined as the ratio between the zero shear viscosity ( $\eta_0$ ) and the viscosity at a stress ( $\sigma$ ) value of 50000 Pa.

**[0022]** Generally, the shear thinning index is proportional to the broadness of the molecular weight distribution (MWD) of the polymer.

At very high values it can reflect the bimodality of the molecular weight distribution (i.e. the MWD curve shows two maxima or one maximum and a pronounced shoulder). For reasons of processability and homogeneity the higher

molecular weight tail will be limited in practice.

## MFR

5 [0023] The melt flow rates were measured with a load of 2.16 kg at 230 °C. The melt flow rate is that quantity of polymer in grams which the test apparatus standardised to ISO 1133 extrudes within 10 minutes at a temperature of 230 °C under a load of 2.16 kg.

## Comonomer contents

10 [0024] Ethylene content in propylene polymer was measured by Fourier transmission infrared spectroscopy (FTIR). A thin film of the sample (thickness approximately 250 µm) was prepared by hot-pressing. The area of -CH<sub>2</sub>- absorption peak (800 - 650 cm<sup>-1</sup>) was measured with Perkin Elmer FTIR 1600 - spectrometer. The method was calibrated by ethylene content data measured by <sup>13</sup>C NMR.

## Xylene Solubles (XS)

15 [0025] For the determination of the xylene solubles fraction, 2.0 g of polymer are dissolved in 250 ml p-xylene at 135 °C under agitation. After 30 ± 2 min the solution is allowed to cool for 15 min at ambient temperature and then to settle for 30 min at 25 ± 0.5 °C. The solution is filtered with filter paper into two 100 ml flasks.

20 [0026] The solution from the first 100 ml flask is evaporated in nitrogen flow and the residue is dried under vacuum at 90 °C until constant weight is achieved. The xylene soluble fraction is calculated using the following equation:

$$25 \quad XS\% = (100 - m_1 \cdot v_0) / (m_0 \cdot v_1)$$

wherein

$m_0$  = initial polymer amount [g],

$m_1$  = weight of residue [g],

30  $v_0$  = initial volume [ml],

$v_1$  = volume of analysed sample [ml].

XS largely correlates to the rubber content of the heterophasic polymer.

## Dynamic Mechanical Thermal Analysis (DMTA)

35 [0027] The tests are carried out in accordance with ISO 6721-2 on specimens of 60x10x1 mm cut from compression moulded plaques. In a measurement at 1 Hz (free oscillation) a temperature range of at least -100 to +150 °C is covered, using a heating rate of 1 °C/min. The storage modulus  $G'$  and the tangent of the loss angle  $\tan(\delta)$  are the primary results of the tests; from  $\tan(\delta)$  the temperatures - peak position and peak broadness - of the various mobility transitions, such as the glass transition temperature  $T_g$ , in the systems, which can be attributed to the phases present, can be determined.

## Tenacity, Elongation

45 [0028] The mechanical properties of the fibres were determined on a Textechno Statimat M. according to ISO 5079. The gauge length used has been 100 mm for fibres and 200 mm for nonwoven, the speed was 100 m/min. The tensile test method which was used for nonwoven was Edana 20.2-89.

## Example 1 (invention, propylene/ethylene random heterophasic copolymer)

50 [0029] A continuous multistage process was used. The process comprised a prepolymerisation step, a loop reactor and a fluidized bed gas phase reactor.

[0030] The catalyst used was highly active, stereospecific transesterified MgCl<sub>2</sub>-supported Ziegler-Natta catalyst prepared according to US 5,234,879 at a titanisation temperature of 135 °C. The catalyst was contacted with a co-catalyst (triethylaluminium, TEAL), and an external donor (di-cyclopentyl dimethoxysilane) with the Al/Ti ratio of 200 and an Al/Donor ratio of 10, to yield a catalyst system.

55 [0031] The catalyst system and propylene were fed into the prepolymerisation reactor which was operated at ca. 30 °C and ca. 30 bar. The prepolymerised catalyst was used in the subsequent polymerisation reactors.

[0032] Propylene, ethylene and hydrogen and the prepolymerised catalyst were fed into the loop reactor which was

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operated as bulk reactor at a temperature of ca. 70 °C and a pressure of ca. 30 bar.

Then, the polymer slurry stream was fed from the loop reactor into the gas phase reactor which was operated at a temperature of ca. 70 °C and a pressure of ca. 20 bar. More propylene, ethylene and hydrogen were fed into the gas phase reactor to control the desired properties of the final polymer.

### Example 3 (comparative, random copolymer)

**[0033]** A continuous process was used. The process comprised a prepolymerisation step and a loop reactor.

**[0034]** The catalyst used was highly active, stereospecific transesterified MgCl<sub>2</sub>-supported Ziegler-Natta catalyst prepared according to US 5,234,879 at a titanisation temperature of 135 °C. The catalyst was contacted with a co-catalyst (triethylaluminium, TEAL), and an external donor (di-cyclopentyl dimethoxysilane) with the Al/Ti ratio of 200 and an Al/Donor ratio of 10, to yield a catalyst system.

**[0035]** The catalyst system and propylene were fed into the prepolymerisation reactor which was operated at ca. 30 C and ca. 30 bar. The prepolymerised catalyst was used in the subsequent polymerisation reactors.

**[0036]** Propylene, ethylene and hydrogen and the prepolymerised catalyst were fed into the loop reactor which was operated as bulk reactor at a temperature of ca. 70 °C and a pressure of ca. 30 bar. The product was degassed before being fed to an extruder for pelletisation.

Table 1

Polymer		1	2	3*	4	5
polymer split Loop Reactor	[wt%]	100	80	92	100	100
polymer split Gas Phase	[wt%]	0	20	8	0	0
MFR Loop	[g/10 min]	25	28	1,3	-	-
MFR end	[g/10 min]	16,5	17,9	1,5	26	25
XS Loop	[wt%]	7,1	3,6	4,5	3	3,1
XS end		7,1	25,1	11,9	3	3,1
C2 Loop		3,2	1,5	2,0	-	-
C2 end	[wt%]	3,2	6,9	4,5	-	-
C3 (EPR)		-	71	70	-	-
Eta 0		1827	1607	660	1060	692
SHI (0/50)		9,6	7,6	3,5	8,6	3,6
PI		4,43	3,64	2,46	3,8	2,45
Tcr	[°C]	106,5	110,9	107,2	113	113
Tm	[°C]	144,4	151,2	150,6	161,8	161,8
T <sub>g</sub> (2nd)	[°C]		-40	-55		
T <sub>g</sub> (1st)	[°C]		0	0		
Spinning speed at 235 °C	[m/min]	2000	2500	3500	2900	3800

MFR Loop: Melt Flow Rate of the matrix phase of the heterophasic copolymer  
 XS Loop: xylene soluble fraction of matrix phase of the heterophasic copolymer  
 MFR end: Melt Flow Rate of the heterophasic copolymer  
 XS end: xylene cold soluble fraction of the heterophasic copolymer  
 C2 total: total ethylene content of the heterophasic copolymer  
 C3/EPR: propylene content of the ethylene propylene rubber  
 Eta 0: zero shear viscosity  
 PI: polydispersity index  
 \* Polymer 3 is a controlled rheology material with final MFR 25 g/10 min

### Spinning tests

**[0037]** Spinning trials have been performed on a Fourné long spin pilot line. The spinneret used has 52 holes, each having a diameter of 0.5 mm. The throughput per hole has been kept constant at 0.3 g/hole·min.

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At a melt temperature of 235°C the take-up speed has been increased stepwise until fibre break. The data of the maximum take-up speeds are shown in table 1. Fibre samples have been taken and mechanical properties have been tested. The results can be seen in Fig. 1 and 2.

### 5 Spunbonded trials

[0038] Polymer 3 has been evaluated on a Reicofil 3.1 pilot line. It was run on a single beam at a melt temperature of 257 °C at the die and a throughput of 180 kg/m/h. Spinning stability was good and at a cabin pressure of 6916 Pa the filament titer was 1.6 denier. The mechanical properties obtained on a 17 g/m<sup>2</sup> web are listed in Table 2 below.

Table 2

MD tensile strength (N/5cm)	32.75
CD tensile strength (N/5cm)	22.08
MD elongation (%)	67.57
CD elongation (%)	72.88

### Softness

[0039] The samples used for determining softness were yarns produced at 1000 m/min and 235 °C from the spinability test. 600 m of yarn are wound on a reel, doubled (i.e. folded) and the ends secured between 3 cardboard sheets. Evaluation of softness was by black box panel. Seven persons tested each fibre material and marked their tactile impressions on a scale from 1-5. "1" means that the fibre has about the same softness as a fibre from a propylene homopolymer produced under the same conditions. "5" means that the fibre is very much softer than a fibre from a propylene homopolymer produced under the same conditions.

The results are shown in Table 3.

Table 3

Polymer-no.	1	21	31	4
	softness relative to homopolymer			
persons				
tester no. 1	1	5	5	1
tester no. 2	1	5	5	1
tester no. 3	1	5	5	1
tester no. 4	1	5	5	1
tester no. 5	1	5	5	1
tester no. 6	1	5	5	1
tester no. 7	1	5	5	1

[0040] All seven testers have defined the samples from polymer 2 and polymer 3 were very much softer than either homopolymer or random copolymer. Compared to the other materials it is softer by an order of magnitude.

### Claims

1. A fibre comprising a heterophasic propylene copolymer containing

a) 80-99 wt% of a matrix phase comprising a propylene random copolymer with 0.2-15 wt% of ethylene and/or at least one C<sub>4</sub>-C<sub>8</sub>  $\alpha$ -olefin and

b) 1-20 wt% of a disperse phase comprising an ethylene rubber copolymer with from 20 - 80 wt% ethylene and from 80 - 20 wt% of at least one C<sub>3</sub>-C<sub>8</sub>  $\alpha$ -olefin,

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wherein the heterophasic copolymer has at least two  $T_g$  peaks.

- 5
2. A fibre according to claim 1, **characterised in that** the ethylene rubber copolymer is an ethylene propylene rubber (EPR).
- 10
3. A fibre according to any one of claims 1 or 2, **characterised in that** the heterophasic propylene random copolymer contains 2 - 15 wt%, preferably 5 - 12 wt% of the ethylene rubber copolymer.
- 15
4. A fibre according to any one of claims 1 to 3, **characterised in that** the heterophasic propylene copolymer has an MFR of from 0.1 - 50g/10min, preferably 2.5 - 30 g/10 min.
- 20
5. A fibre according to any one of claims 1 to 3, **characterised in that** the heterophasic propylene copolymer has an MFR of from 200 - 2000 g/10 min.
- 25
6. A fibre according to any one of claims 1 to 5, **characterised in that** the heterophasic propylene copolymer has an overall ethylene content of from 1.0 - 15.0 wt%.
- 30
7. A fibre according to any one of claims 1 to 6, **characterised in that** the heterophasic propylene copolymer has a first  $T_g$  in the range of from -15 to +5 °C, preferably around 0 °C and a second  $T_g$  in the range of from -35 to -65 °C, preferably from -40 to -60 °C and more preferably around -50 °C.
- 35
8. A fibre according to any one of claims 1 to 7, **characterised in that** the heterophasic propylene copolymer contains 80 - 95 wt% of a matrix phase comprising a propylene random copolymer with from 1.0 - 15.0 wt% of ethylene and 5 - 20 wt% of a disperse phase comprising an ethylene propylene rubber with from 20 - 40 wt% of ethylene and 80-60 wt% of propylene, the heterophasic propylene copolymer having an overall ethylene content of from 4.0 - 12.0 wt% and two distinct  $T_g$  peaks.
- 40
- 45
- 50
- 55
9. Articles comprising fibres according to any one of claims 1 to 8, especially nonwoven fabrics, e.g. diapers, medical gowns and masks; woven fabrics, e.g. upholstery, clothing, ropes, twines and carpets.

Fig. 1

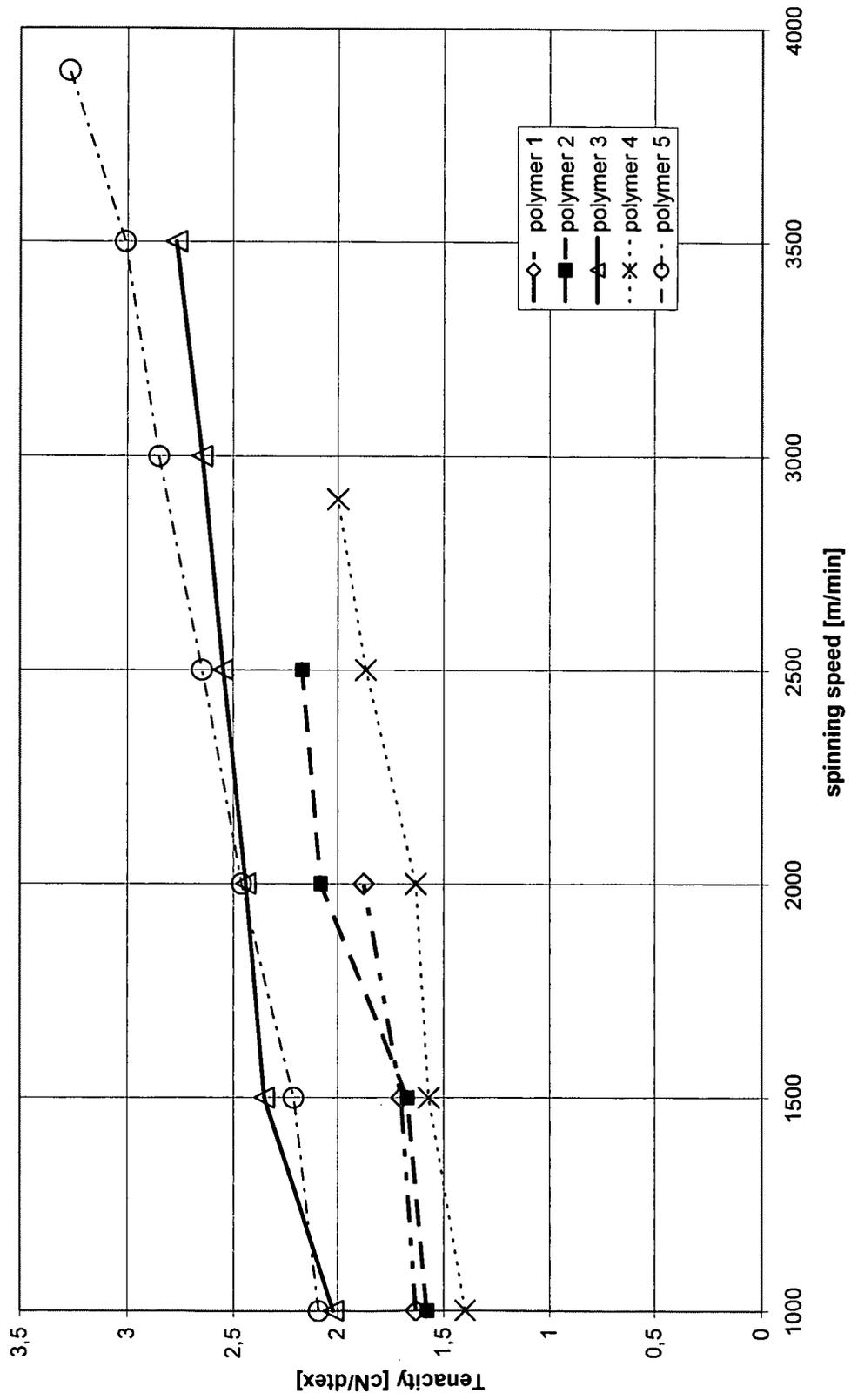


Fig. 2

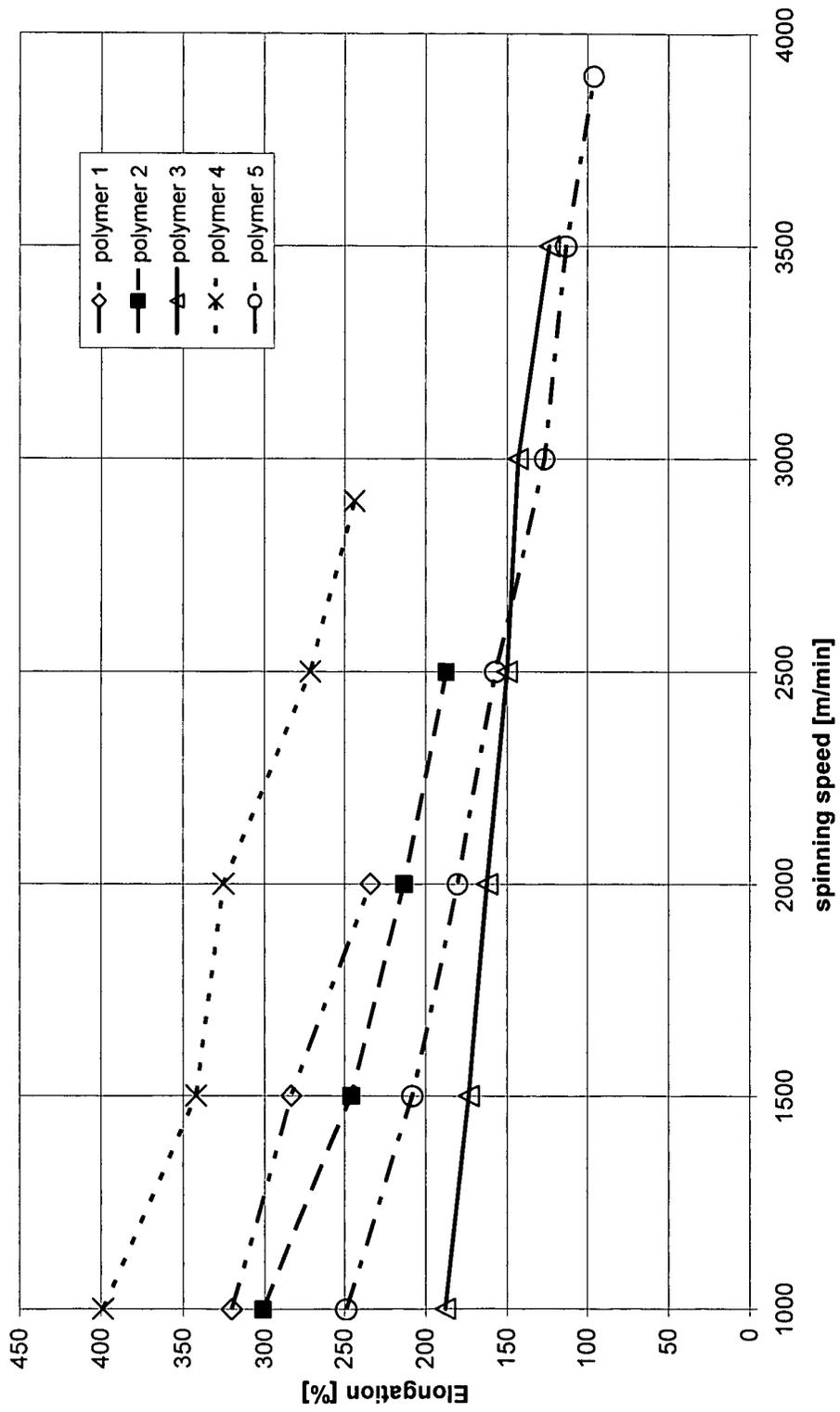


Fig. 3  
Polymer 2 DMTA @ 1 Hz

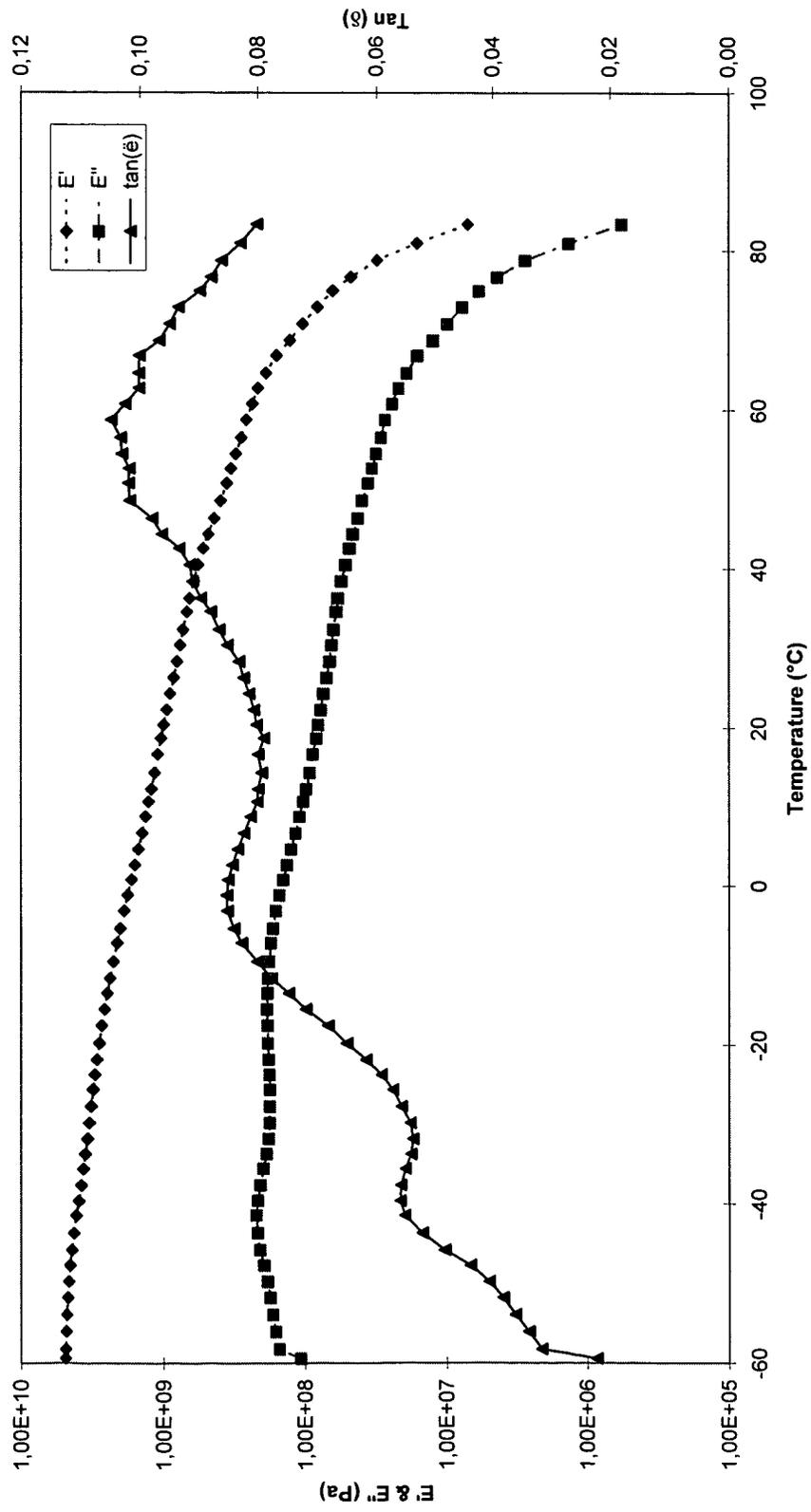
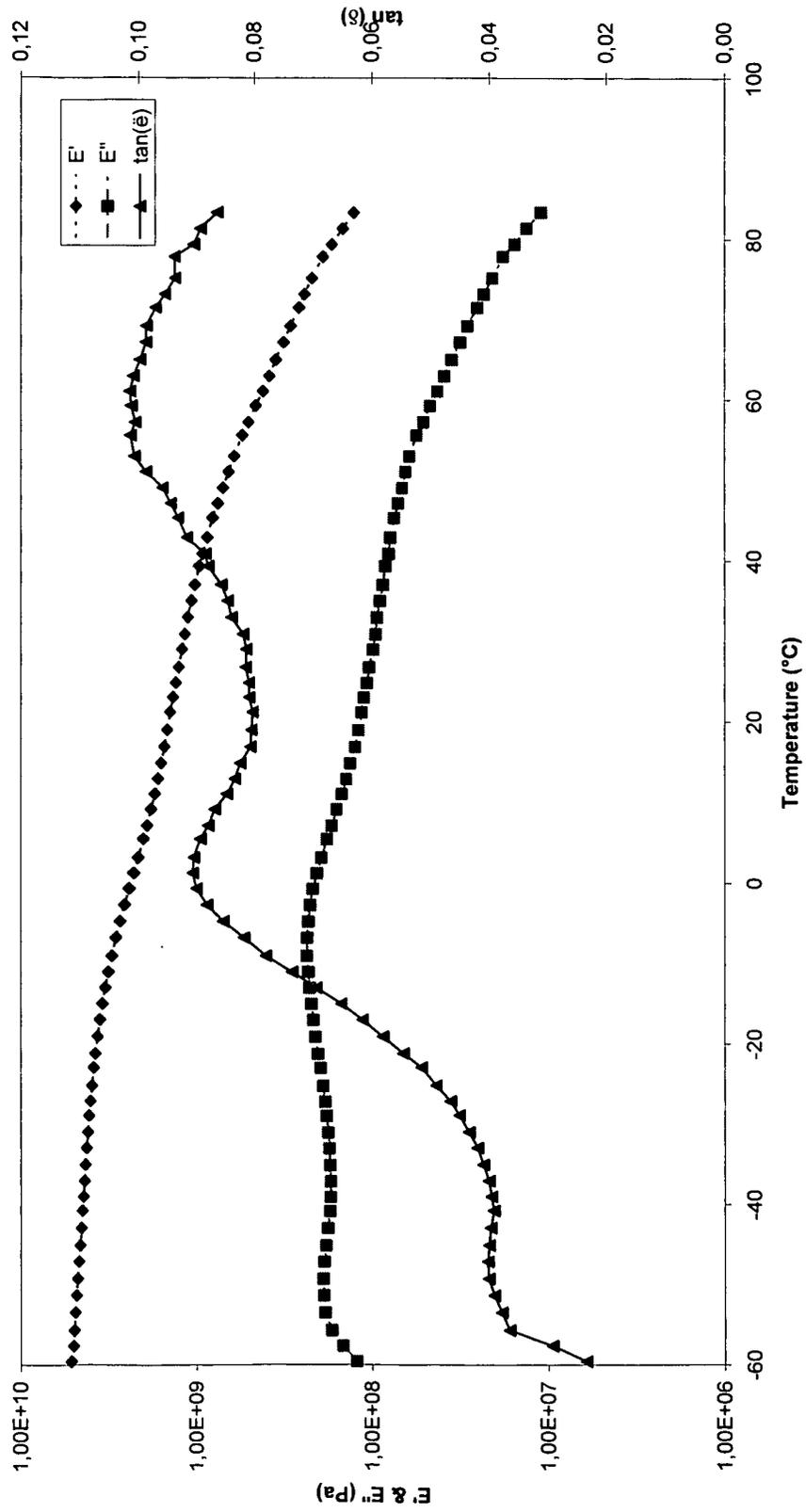


Fig. 4  
Polymer 3 DMTA @ 1Hz





European Patent  
Office

EUROPEAN SEARCH REPORT

Application Number  
EP 03 00 4115

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
A,D	US 6 218 010 B1 (CHIEN WILLIAM MOA-TSENG ET AL) 17 April 2001 (2001-04-17) * column 3, line 12 - line 16 * * column 5, line 44 - line 67 * * examples * ---	1-9	D01F6/46
A	EP 0 632 147 A (HIMONT INC) 4 January 1995 (1995-01-04) * page 2, line 27 - line 50 * * example 3 * ---	1-9	
A	EP 0 552 810 A (HIMONT INC) 28 July 1993 (1993-07-28) * example 8 * ---	1-9	
A	EP 0 987 287 A (SUMITOMO CHEMICAL CO) 22 March 2000 (2000-03-22) * page 3, line 15 - line 40 * * page 17, line 9 - line 16 * * examples * ---	1-9	
A	EP 1 041 111 A (SUMITOMO CHEMICAL CO) 4 October 2000 (2000-10-04) * page 3, line 5 - line 28 * * examples * -----	1-9	TECHNICAL FIELDS SEARCHED (Int.Cl.7) D01F C08L
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
THE HAGUE	29 July 2003	Fiocco, M	
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

EPO FORM 1503 03 82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 03 00 4115

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

29-07-2003

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