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(71) Applicant *(for all designated States except US):* **NORWEX HOLDING AS** [NO/NO]; Industriveien, N-2072 Dal (NO).

(72) Inventors; and

(75) Inventors/Applicants *(for US only):* **MING, Gao** [CN/CN]; 2-301, Siping Road Lane 184, Shanghai 200086 (CN). **PETERSEN, Jørgen** [DK/DK]; Harløsevej 299, DK-3320 Skaevinge **(DK).**

(74) Agents: **ONSAGERS AS** et al; P.O. Box 6963 St. Olavs Plass, N-0130 Oslo (NO).

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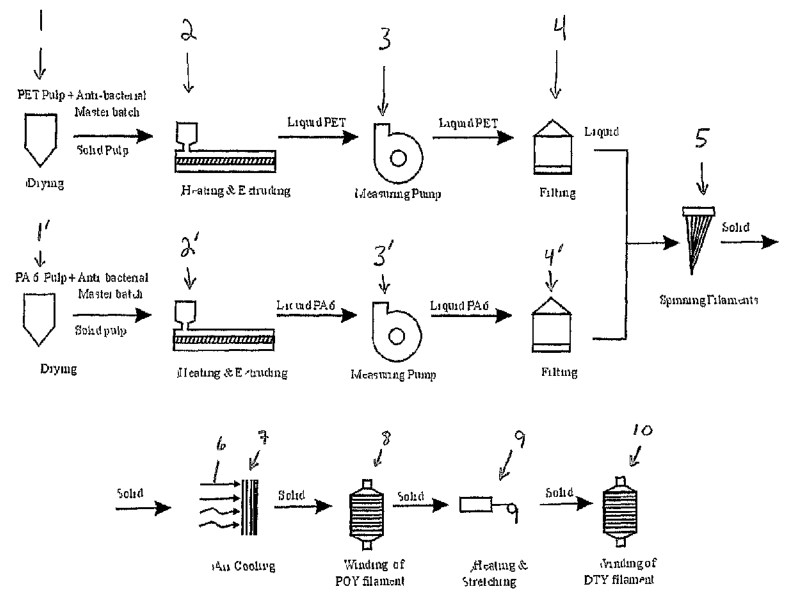
(54) **Title:** ANTI-BACTERIAL MICRO-FIBRE AND PRODUCTION THEREOF



(57) Abstract: According to one aspect of the invention, there is provided a micro fiber comprising an antibacterial agent that meets the following criteria: The agent must withstand (i.e. not loose its antibacterial activity) temperatures of at least 300 degrees C; The particles of the agent must have an average size in the range from lOOnm to 900nm, preferably from 400nm to 900nm, and most preferably from 500nm to 900nm; The agent must be suitable for use in a consumer product, i.e. be non-toxic, non allergenic and environmentally safe. The agent should preferably be a substance already approved by regulatory bodies for such use. The invention also provides a method of production and products made from the micro fibers .

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ANTI-BACTERIAL MICRO-FIBRE AND PRODUCTION THEREOF

BACKGROUND - FIELD OF THE INVENTION

This invention relates generally to anti-bacterial filaments useful in the production of textiles such as cleaning products and clothes, more particularly to bi-component filaments used to make micro-fibres and methods of manufacture thereof. More particularly, this invention relates to a bi-component filament or staple fibre of less than 2.5 denier ("D") linear density produced with an anti-bacterial agent, designed to be split into micro-fibres, during the finishing process of the final product, of less than 0.3 D. This can be achieved by producing a filament or a staple fibre with 2 polymer components or more that can be separated under certain conditions.

DEFINITIONS

The following terms have the following definitions as used throughout this specification:

Fibre: Basic unit of raw material having suitable length, pliability, and strength for conversion into yarns and fabrics. A fibre of extreme length is a filament. Fibres can occur naturally or can be produced artificially

Bi-component fibre or filament: a fiber or filament comprising a plurality of individual fibers or filaments of two different polymers coexisting in the same cross-sectional structure, having a size 2.5 denier or less

Staple-fibre: Bi-component fibre with a finite length of size 2.5 denier or less

Micro-fibre: One component single fibre or filament of size 0.3 denier or less, generally a single fibre that has been split from a bi-component fibre or filament

MF Yarn: A bundle of filaments or staple-fibres spun together

Construction yarn: A yarn normally comprised of fibres or filaments of significantly greater diameter than a MF_5 used as the lattice onto which MFs are knitted or woven, for example to form terry loops.

Acronyms:
PET: Polyester fibre
PP: Polypropylene fibre
DTY: Drawn Textured Yarn
FDY: Full Drawn Yarn
POY: Partly Oriented Yarn
ABA: Anti Bacterial Agent
Denier: D
MF: Micro-Fibre

BACKGROUND - PRIOR ART.

Microfibres

A MF generally comprises a filament or fibre having a linear density of 2.5 D or less, and preferably less than 0.3 D. A MF is produced by splitting a multi-component filament or fibre. Figures 1 -10 and 14 illustrate several different types of multi-component fibres that can be split into MF' s. The example shown in Figs 1 and 2 is known as the "sea-island type", the example shown in Figs 3 and 4 is known as the "wedge/orange type", and the example shown in Figs 5 and 6 is known as the "Hollow Segmented Pie" type, the example shown in Figs 7,8 and 14 is known as the "Pie Wedge" type, and the example shown in Figs 9 and 10 is known as the "Conjugate" type. Most MFs today will have a size of 0.13 D, 0.20 D or 0.26 D depending on the design of the filament or staple-fibre.

Bi-component filaments or fibres that can be split into MFs can be made of any two synthetic polymers as well as a combination of some two natural polymers like a Viscose and/ or a Protein fibre. After splitting the bi-component filament or fibre, the resulting MF's therefore usually consists of two fibres of different Polymers, but in some cases where Sea-Island filament or fibre design is used, the final MF can consist of only one fibre of one Polymer. MF filaments can then be spun together in a spinneret to form a yarn useful in the manufacturing of fabrics.

The production technology to produce micro-fibres can be divided into several kinds, each being suitable to produce certain kinds of chemical fibres. MFs can be produced according to the melt spinning method, for example to produce polyester or polyamide fibres or can be produced using the wet spinning method, for example to produce acrylic, viscose or rayon fibres. The most popular MF design used for cleaning products is the orange type, composite filament of PET and PA6 produced by the melt spinning method

More and more, micro-fibres are being used in the manufacturing of cleaning products such as cleaning cloths. A MF cleaning cloth is generally produced by knitting or weaving MF' s onto a lattice comprised of a construction yarn of significantly greater diameter. While the cleaning effect of MF products is significant, there is a need for them to have an anti-bacterial activity. Such antibacterial activity is useful for keeping the cleaning product itself clean, as well as perhaps improving the cleaning effect of the product.

One example of known anti bacterial fibres is described in EP 144 3099. The fibre disclosed in EP 144 3099 is an antibacterial PET yarn that is used as the construction yarn of a finished cleaning product that is knitted together with a non-ABA-containing MF in the terry loops. A major disadvantage of this type of 100% PET yarn is that the fibre is comprised of only one component/polymer, and thus cannot be split into MF's. As a result, this type of ABA fibre is primarily used as the construction yarn of the final product. The construction yarn does not come into direct contact with the surface being cleaned as is the case with the MF filaments of a cleaning product. The anti-bacterial effect of this type of fibre is therefore significantly reduced. In addition, a recent trends in the manufacture of MF cleaning products is to use MF filaments also in the construction yarn, in which case the ABA filaments disclosed in EP 144 3099 could not be used in the product at all.

Another example of a known technique for providing an anti-bacterial effect in cleaning products is described in GB 241 1 184. This patent describes a coating method to coat the finished fibre with an ABA. The disadvantage of this technique is that the coating will have a very limited duration as it is removed during cleaning and laundering. Another disadvantage with the above mentioned production method is that the ABA will create a film on the surface of the final MF fabric thus reducing or remove the added benefit of MF compared to other conventional fibres.

Until now it has been difficult to produce durable anti-bacterial micro-fibres of the required quality. There are several reasons for this:

- First the linear density (size) of micro-fibre is very small hence the addition of an antibacterial agent having too large a particle size will result in filaments or fibres that degrade the physical and mechanical properties of the fibres. Specifically the difficulty is due to the fact that the ABA's particle size has traditionally been too large to be able to incorporate into a MF of size 0.3D or less without affecting the strength or durability of the fibres. For example, the average particle size non-organic ABA's is 25 µm.
- Secondly, the particles in the ABA cannot be too small, or else the particles will attract each other to form larger particles that clog the holes in the spinneret and as such making it impossible to produce a yarn. Particles that are too small have the additional disadvantage that they may be released from the fibre into the environment (so-called "leave-on" effect). Particles of this size are often described as being based on nanotechnology. Examples of agents having too small a particle size include:
    - NanoSilver from JR Nanotech (http://www.jrnanotech.com/nano_silver.html)
    - NanoSilver from Fine Polymer
    - Antibacterial Colloidal Silver master batch avg. size of 30nm
    - Nanover from Nanogist with an average particle size of 7nm

- Thirdly it is difficult to find a suitable anti-bacterial agent with the necessary combination of properties, such as strong antibacterial effect, durability, heat resistance, resistance to colour change, compatibility with polymers, being environmentally friendly, wide anti-bacterial scope, non-toxic to humans, etc. For example, in an ABA with Ag (silver) combined with Zeolite, the Zeolite will attract moisture and accelerate the oxidation process inside the polymer reducing breaking strength of the filament. This again prevents the filament to be used to complete the final production by knitting or weaving. Other examples include ABA's based upon alcohol or enzymes, the activity of which will be destroyed at the temperatures employed in the production process for the MF.

SUMMARY OF THE INVENTION

The present invention seeks to address the above-identified problems.

Micro fibres

According to one aspects of the invention, there is provided a MF comprising an antibacterial agent that meets the following criteria:

1) The agent must withstand (i.e. not loose its AB activity) temperatures of at least 300 degrees C.
2) The particles of the agent must have an average size in the range from lOOnm to 900nm, preferably from 400nm to 900nm, and most preferably from 500nm to 900nm
3) The agent must be suitable for use in a consumer product, i.e. be non-toxic, non allergenic and environmentally safe. The agent should preferably be a substance already approved by regulatory bodies for such use.

As an example an anti-bacterial agent meeting the above criteria comprises a mixture of silver phosphate and metal oxide or a mixture of silver sodium zirconium phosphate and titanium dioxide. Guanidine Salt from PreTech AS in Denmark also meets the above criteria.

The MF filament preferably has a linear density of 2.5 D or less, and preferably less than 0.3 D after the bi-component fibre is split. Most preferably the MF filament will have a size of approximately 0.13 D, 0.20 D or 0.26 D depending on the design of the filament or staple-fibre. The bi-component fibre can be made of any two synthetic polymers as well as a combination of some two natural polymers such as a viscose and or a protein fibre. After splitting the bi-component fibre, the final MF filaments usually consists of two filaments of different Polymers, but in some cases where Sea-Island filament or fibre design is used, the final MF filament can consist of only one filament of one Polymer. Preferably, the bi-component fibre comprises a combination of PET and PA 6 fibres combined with an ABA. Either or both of them could contain an ABA. The bi-component fibre desirably comprises 60-100% by weight of polyester and 0-40% by weight of polyamide to which the ABA is added. The preferred composition of a bi-component fibre is 70% to 80% PET and 20% to 30% PA6.

Production method

According to another aspect of the invention, there is provided a method for producing micro-fibres wherein an anti-bacterial agent meeting the criteria disclosed above is introduced into the production process.

According to a preferred method of producing the MF according to the invention, an anti-bacterial master batch is first made by mixing the AB agent with the fibre pulp. The mixture of the anti-bacterial agent and the fibre pulp is then melted together, extruded and drawn into a filament. The filament is preferably a bi-component filament made with the melt-spinning method, the filament comprising two kinds of polymer conveyed to a shared spin pack and combined herein, then spun but from the shared spinneret holes and formed into filaments. These filaments are thereafter split into MF filaments containing the AB agent.

In a preferred method , a silver-based AB meeting the above described criteria is mixed with PET since most of the silver based ABAs have better colour resistance properties or light stability in PET than PA..

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described in detail, with reference to the drawings in which:

Figure 1-10 shows prior art types of bi-component fibres and the resulting MFs after splitting.

Figure 11 is a flow diagram showing the production method of a micro-fibre according to the invention, and

Figure 12 shows a knitting method for SUPERPOL terry cloths.

Fig 13 is a microscope image of a microfiber according to the invention containing particles of an antibacterial agent

DETAILED DESCRIPTION OF THE INVENTION

Method of production

Referring to Figure 11, an antibacterial master batch of silver sodium zirconium phosphate and titanium dioxide is added to the solid PET pulp and dried in a hopper, 1, at a temperature of 120°C to 160^{0}C for 3 to 6 hours. The mixture is then heated to a temperature between 285°C to 295^{0}C and extruded in an extruder 2. The molten mixture of PET and ABA is then pumped by a measuring pump, 3, into a reservoir, 4. Optionally at the same time, an anti-bacterial master batch of silver sodium zirconium phosphate, titanium dioxide or Guanidine Salt and PA6 can added to the solid PA6 pulp for an increased antibacterial effect, and a smaller quantity of the mixture is dried in a hopper, V, at 100^{0}C to 120^{0}C for 10 to 14 hours. The mixture is then heated to a temperature of 270^{0}C to 29O^{0}C and extruded in an extruder, 2'. The molten mixture of PA6 and ABA is then pumped by a measuring pump, 3', into a reservoir, 4'. The PET/ABA and PA6/ABA molten mixtures are then passed to a filament spraying combine, 5, and spin into a filament in a ratio of 70-80% PET/ABA and 20-30% PA6/ABA. The ratio can be controlled by regulating the speed of the pumps 3 and 3'. The mixtures of PET/ABA at 285°C to 295^{0}C and the mixture of PA6/ABA at 270°C to 290°C are combined in the combine, 5, and squeezed out through a specially designed spinneret to form the composite filaments, 6, which is then cooled in an air flow, 7, at 2O^{0}C to 30^{0}C. Once the filaments have been cooled, they are wound onto a drum 8 which is driven at 3000m/min to 3300 m/min with a predetermined stretch to keep the filaments at the desired linear density, for example 160D. The result of this process is either POY or FDY filaments.

For the POY, the POY filament is unwound from the drum, heated and stretched at a station, 9, into DTY filament which is wound onto a drum, 10.

The new development of the ABA according to the invention makes it possible to manufacture the MF with the ABA incorporated in the MF during the production of the filament.

Micro-fibre itself

The result of this production process is a filament or a staple-fibre. The MF after splitting the filament from this process will be of size 0.05D to 0.23D, and the exact linear density and design of the filament will depend on the spinneret used.

An example of a MF according to the invention will now be described in detail:

Example

A mixture of 80% by weight of PET and ABA and 20% by weight of PA6 were dried separately at 120-160^{0}C and 100-120^{0}C. The ABA consisted of a mixture of 30% by weight of silver sodium zirconium phosphate and 70% by weight of titanium dioxide. The PA6 Pulp was dried for 10 hours at a temperature of 100^{0}C.

The PA6 Pulp and the PET and ABA master batch was then separately heated to 278^{0}C and 290 ^{0}C and then separately extruded. After extrusion the two molten polymers was sent separately to the same spinning pack to form the filaments and then cooled down by an air flow at a temperature of 27 ^{0}C. After being cooled down the filaments were combined into a bi-component filament of PET/ABA and PA6 in the form of a POY. Then the POY was heated to a temperature of 180 ^{0}C. After heating the POY was drawn and spun into DTY of 150D. The filament design was an orange/wedge type, in which the MFs have a average linear density of 0.23 D. The resulting filament has the following specification:

DTY filament, PET/ABA 80%, PA6 20%, 150 D / 72 filaments X 9 pieces

The yarn containing the ABA was tested for its effect by applying bacteria (S.Aureus, E.CoIi and E.Pneumoniae) to the yarn and cultivating the yarn of 18 hours at a temperature of 37 ^{0}C. The Japanese testing standard JIS1902.2000 was used as a basis for the evaluation of the anti-bacterial fibre. The test results showed that the effectiveness was rated as SEK Orange grade for normal anti-bacterial use. The SEK Orange classification also includes SEK Blue grade classification, but not SEK Red grade classification for use in hospitals etc. This test proves the effectiveness of the anti-bacterial fibre before splitting.

The same test was done on the final fabric made with antibacterial MF together with a fabric of non antibacterial fiber. It was expected that the non antibacterial fabric would have an increased amount of bacteria after incubation while the antibacterial MF would have a decreased amount of bacteria after incubation. The tests conducted proved the effectiveness of the antibacterial MF in the cleaning cloth.

To show durability of the antibacterial property of the MF yarn the same tests were conducted on a microfiber AB Superpol knitted cloth after laundry. It was expected that the AB effect of the laundered MF Superpol cloth would not be significantly reduced compared to the tests done with a new AB Superpol knitted MF Cloth. The test results shows no significant reduction from the tests done on new AB MF Superpol cloth.

MF Products

According to another aspect of the invention is provided textile products made from the above-described micro-fibre. The textile product may be made from knitted, woven or non-woven micro-fibres. For example, the anti-bacterial yarn can be used to knit Superpol terry cloths, in which the anti-bacterial fibre will be exposed on the surface of the cloth. The terry cloth made according to this aspect of the invention, uses anti-bacterial MF, in which the fibres therefore are on the cloth surface.
According to another aspect of the invention, an antibacterial MF cleaning product is provided in which the construction yarn is woven from MF filaments containing an AB agent.

CLAIMS

1. A microfiber having a linear density of 2.5D or less, comprising an anti-bacterial agent having an average particle size in the range of 100-900nm and able to withstand exposure to temperatures of at least 300 degrees C and still maintain its antibacterial activity, said antibacterial agent further being non-toxic to humans.

2. A microfiber according to claim 1, wherein the microfiber's linear density is 0.3D or less.

3. A microfiber according to claim 2, wherein the average particle size of the agent is between lOOnm to 900nm, preferably from 400nm to 900nm, and most preferably from 500nm to 900nm.

4. A microfiber according to claim 3, wherein microfiber's linear density is between 0.13D and 0.26D.

5. A microfiber according to claim 4 wherein the antibacterial agent comprises silver.

6. A microfiber according to claim 5, wherein the antibacterial agent comprises a mixture silver phosphate and metal oxide or a mixture of silver sodium zirconium phosphate and titanium dioxide.

7. A microfiber according to claim 4, wherein the antibacterial agent comprises a guanidine salt.

8. A bi-component fiber comprising a plurality of individual microfibers according to any of claims 1-7.

9. A bi-component fiber according to claim 8, wherein the individual microfibers comprise polyester and/or polyamide.

10. A bi-component fiber according to claim 9, wherein polyester comprises 60-100% by weight, and polyamide comprises 0-40% by weight.

11. A bi-component fiber according to claim 10, wherein the antibacterial agent is present only in the polyester microfibers.

12. A method of producing a microfiber containing an antibacterial agent by the melt-spinning method, wherein a non-toxic antibacterial agent having an average particle size in the range of 100-900nm and able to withstand exposure to temperatures of at least 300 degrees C and still maintain its

antibacterial activity is added to a polymer pulp prior to said pulp being melted.

13. A method according to claim 12, wherein the average particle size of the agent is between lOOnm to 900nm, preferably from 400nm to 900nm, and most preferably from 500nm to 900nm.

14. A textile cleaning product comprising a plurality of microfibers according to any of claims 1-7 knitted into loops onto a lattice of a construction yarn.

15. A woven textile cleaning product comprising a plurality of microifbers according to any claims of 1-7.

16. A cleaning product according to claim 14, wherein the construction yarn comprises a plurality of microfibers according to any of claims 1-7 woven together.

17. A cleaning product according to claim 15, wherein the construction yarn comprises a plurality of microfibers according to any of claims 1-7 woven together.

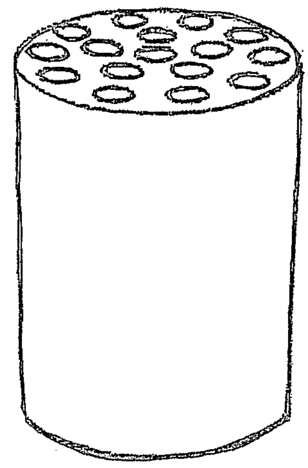

Fig 1

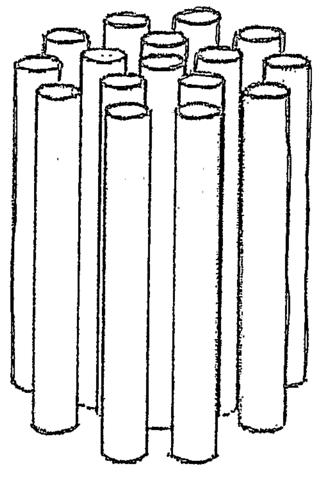

Fig 2

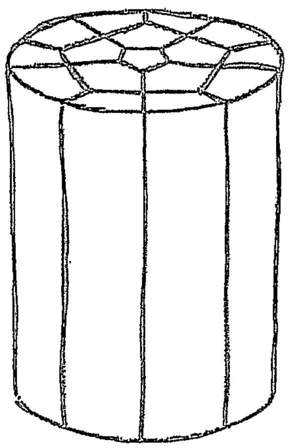

Fig 3

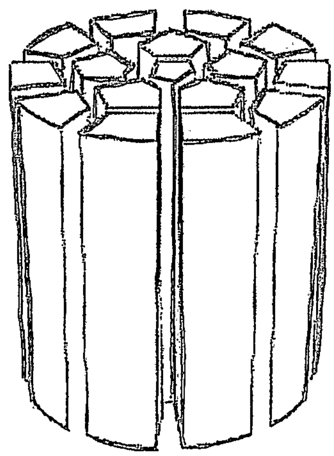

Fig 4

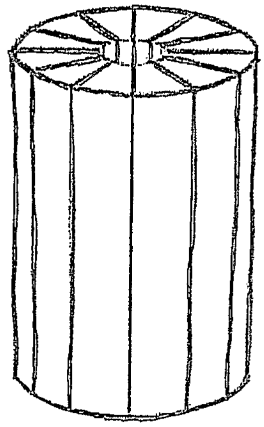

Fig 5

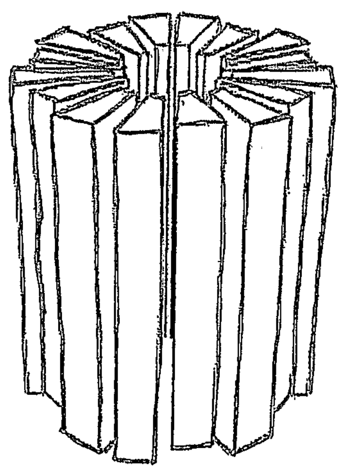

Fig 6

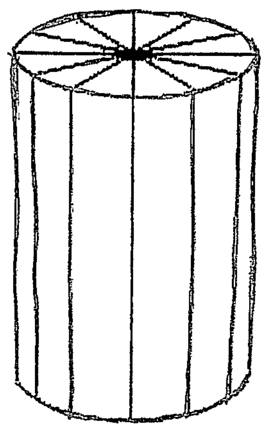

Fig 7

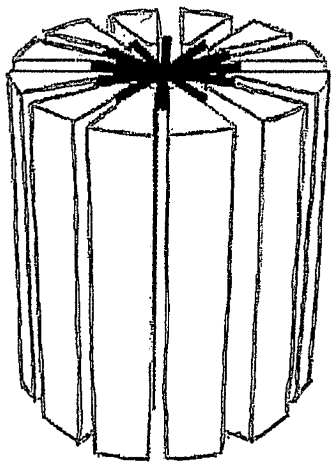

Fig 8

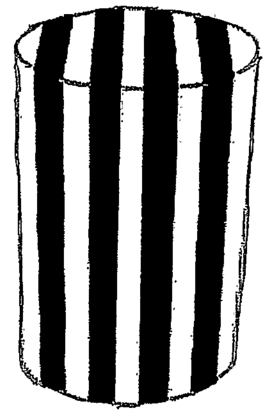

Fig 9

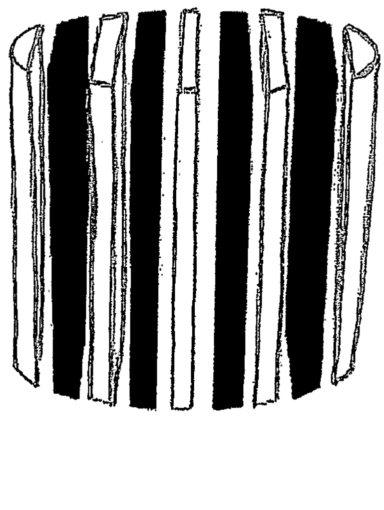

Fig 10

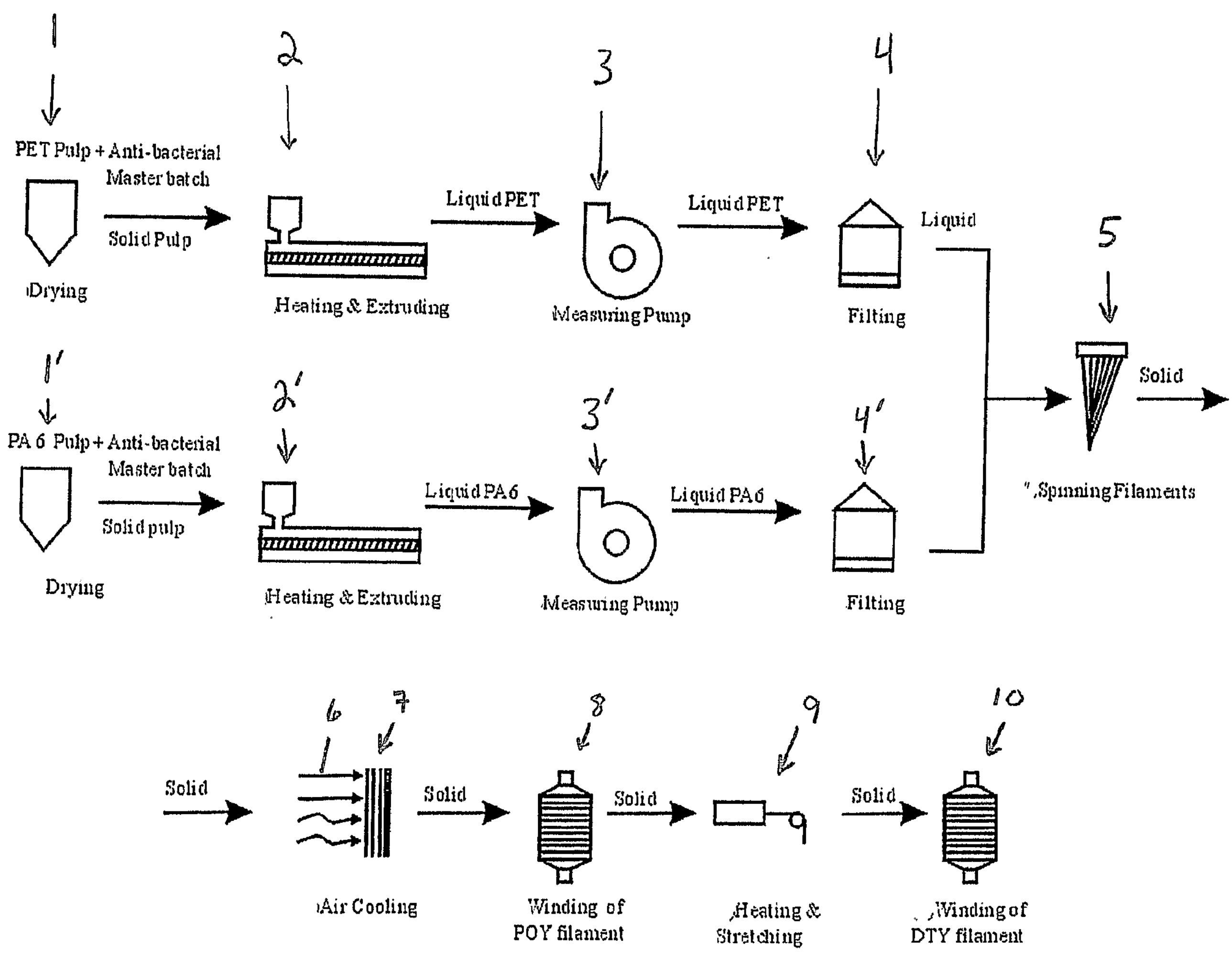
1
PET Pulp + Anti-bacterial Master batch
Solid Pulp
Drying
2
Liquid PET
Heating & Extruding
3
Liquid PET
Measuring Pump
4
Liquid
Filting
5
Solid
Spinning Filaments
1'
PA 6 Pulp + Anti-bacterial Master batch
Solid pulp
Drying
2'
Liquid PA6
Heating & Extruding
3'
Liquid PA6
Measuring Pump
4'
Filting
Solid
6
7
Air Cooling
Solid
8
Winding of POY filament
Solid
9
Heating & Stretching
Solid
10
Winding of DTY filament

Fig 11

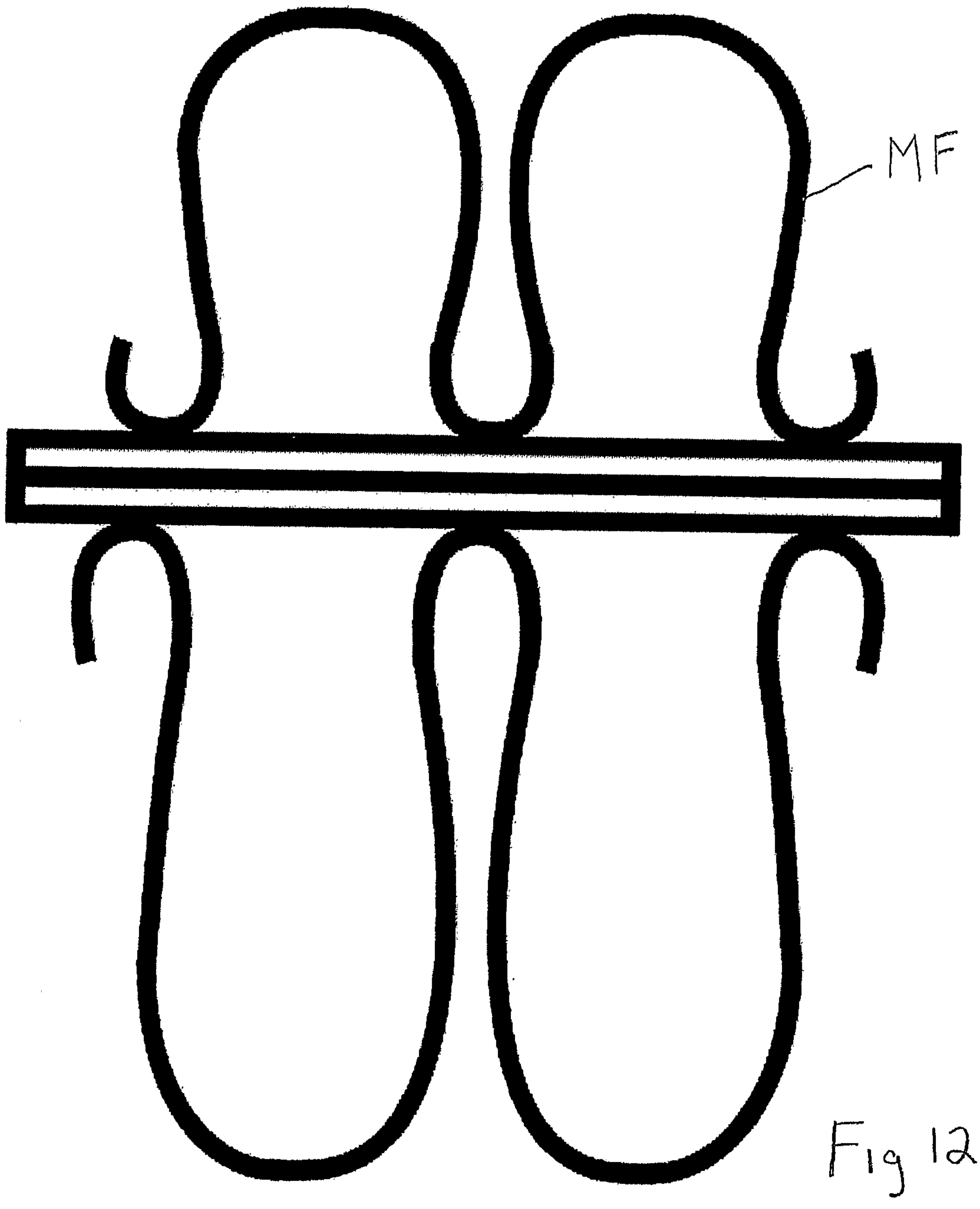

Fig 12

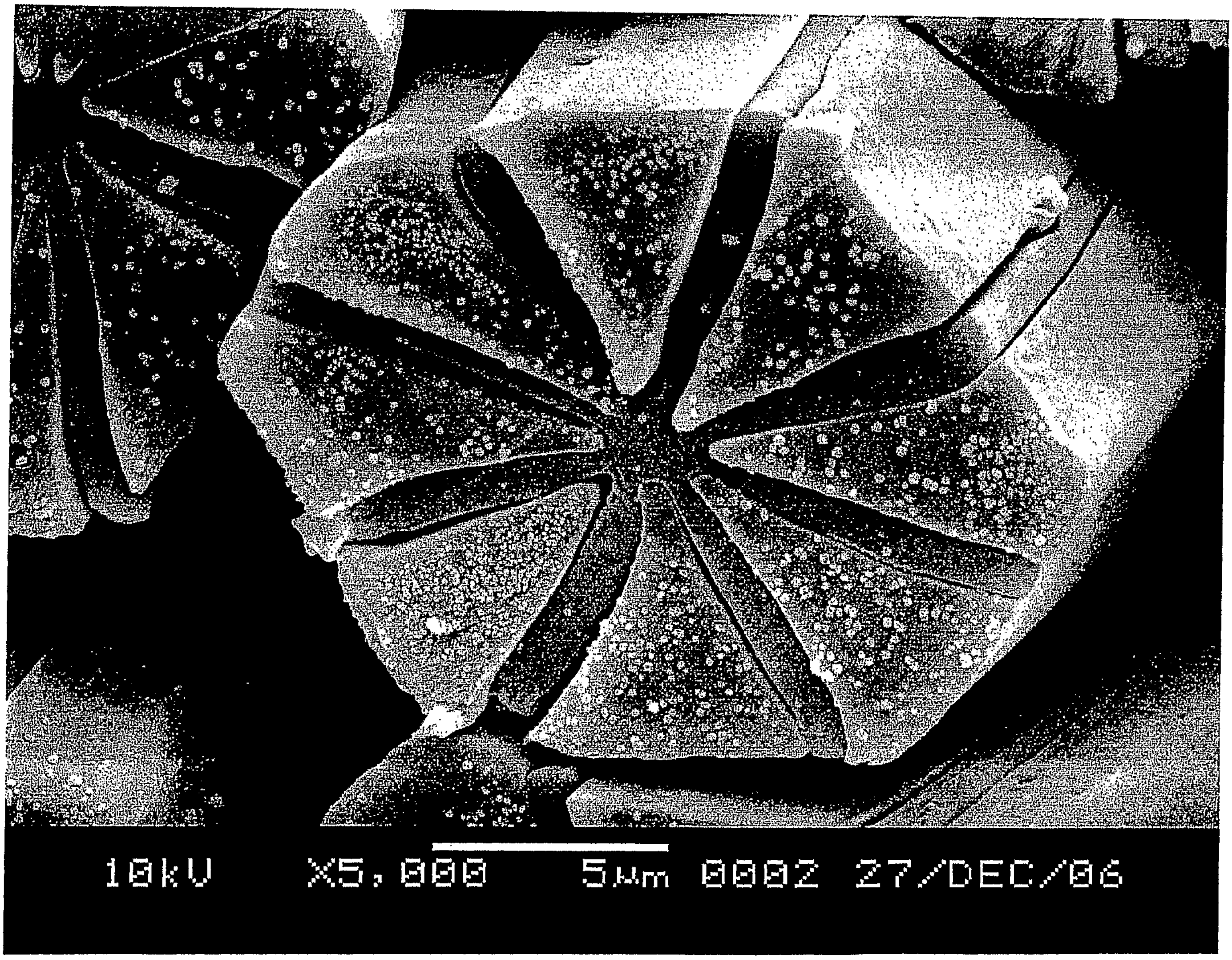

Fig 13

INTERNATIONAL SEARCH REPORT

International application No
PCT/NO2007/000001

A. CLASSIFICATIONOF SUBJECT MATTER ,
INV. DOIFI/IO D01F8/12 D01F8/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
DOIF

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal , WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
X Y	EP 0 360 962 A (KURARAY CO [JP]) 4 April 1990 (1990-04-04) page 3, line 55 - page 4, line 6 page 4, line 42 - line 54 examples	1-5, 12-17 6-11
X Y	JP 09 241957 A (KURARAY CO) 16 September 1997 (1997-09-16) abstract	1,3,5,6, 12,13 6
Y	JP 09 324350 A (TORAY INDUSTRIES) 16 December 1997 (1997-12-16) abstract	7
Y	JP 06 248519 A (DAIWA SPINNING CO LTD) 6 September 1994 (1994-09-06) abstract	8-11
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[X] Further documents are listed in the continuation of Box C

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* Special categories of cited documents

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Date of the actual completion of the international search	Date of mailing of the international search report
4 April 2007	17/04/2007

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P B 5818 Patentlaan 2 NL - 2280 HV RIJSWIJK Tel (+31-70) 340-2040, Tx 31 651 epo nl, Fax (+31-70) 340-3016	Fiocco, Marco

INTERNATIONAL SEARCH REPORT

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PCT/NO2007/000001

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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