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NOTICE OF ENTITLEMENT

I/We W. L. GORE & ASSOCIATES, INC.

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being the applicant(s) and nominated person(s) in respect of an application for a patent for an invention entitled TRIBOELECTRIC FILTRATION MATERIAL (Application No. 47893/93), state the following:

1. The nominated person(s) has/have, for the following reasons, gained entitlement from the actual inventor(s):

THE NOMINATED PERSON IS THE ASSIGNEE OF
THE ACTUAL INVENTOR.

2. The nominated person(s) has/have, for the following reasons, gained entitlement from the applicant(s) listed in the declaration under Article 8 of the PCT:

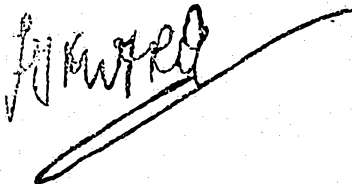
THE APPLICANT AND NOMINATED PERSON IS THE
ASSIGNEE OF THE BASIC APPLICANT.

3. The basic application(s) listed in the declaration under Article 8 of the PCT is/are the first application(s) made in a Convention country in respect of the invention.

DATED: 22 March 1995

W. L. GORE & ASSOCIATES, INC.

GRIFFITH HACK & CO.



Patent Attorney for and
on behalf of the applicant(s)



AU9347893

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(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 669207

- (54) Title
TRIBOELECTRIC FILTRATION MATERIAL
- International Patent Classification(s)
(51)⁵ **B01D 039/16 B03C 003/30**
- (21) Application No. : **47893/93** (22) Application Date : **28.07.93**
- (87) PCT Publication Number : **WO94/12262**
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| 979946 | 23.11.92 | US UNITED STATES OF AMERICA |
- (43) Publication Date : **22.06.94**
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- (71) Applicant(s)
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- (56) Prior Art Documents
AU 32434/93
- (57) Claim

1. An electrically charged filter which comprises a blend of clean expanded porous polytetrafluoroethylene fibers and clean polyamide fibers.

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification⁵: B01D 39/16, B03C 3/30</p>	<p>A1</p>	<p>(11) International Publication Number: WO 94/12262 (43) International Publication Date: 9 June 1994 (09.06.94)</p>
<p>(21) International Application Number: PCT/US93/07084 (22) International Filing Date: 28 July 1993 (28.07.93) (30) Priority Data: 07/979,946 23 November 1992 (23.11.92) US (71) Applicant: W.L. GORE & ASSOCIATES, INC. [US/US]; 551 Paper Mill Road, P.O. Box 9206, Newark, DE 19714 (US). (72) Inventor: WENCHAK, Ray; 92 West Mill Station Road, Newark, DE 19711 (US). (74) Agents: JOHNS, David, J. et al.; W.L. Gore & Associates, Inc., 551 Paper Mill Road, P.O. Box 9206, Newark, DE 19714 (US).</p>		<p>(81) Designated States: AU, CA, DE, GB, JP, SE, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published With international search report.</p> <p>669207</p>
<p>(54) Title: TRIBOELECTRIC FILTRATION MATERIAL</p>		
<p>(57) Abstract</p>		
<p>An electrically charged air filter which comprises a blend of clean expanded porous polytetrafluoroethylene fibers and clean polyamide fibers.</p>		

TITLE OF THE INVENTION

Trieboelectric Filtration Material

FIELD OF THE INVENTION

5 This invention relates to triboelectric filtration media and more particularly to triboelectric air filtration media of blended fibers.

BACKGROUND OF THE INVENTION

10 Triboelectrification is the transfer of electric charge, by contact or by friction, between pieces of different, polymeric materials. Materials may be approximately ranked on a "Trieboelectric scale" regarding their tendency give or receive electrons, i.e., become positively or negatively charged. Such scales vary somewhat by researcher and method, but generally tend to agree. The combination of dissimilar, usually fibrous, materials to create a triboelectric enhancement of air filtration media is known in prior art. On contact of polymers having different tendencies to give or receive electrons, localized electrical charges accumulate. These local electric fields aid in capturing airborne particles and make a blend of dissimilar materials useful as air filters. One such filter, described in British Patent 384052 comprises wool fibers and resin particles. A combination of clean fibers of polyolefin and clean fibers of a polymer consisting of one or more halogen or -CN substituted hydrocarbons is described in Brown U.S. Patent 4,798,850.

25 In an article "Generation of Trieboelectric Charge In Textile Fiber Mixtures and Their Use as Air Filters" from the Journal of Electrostatics, volume 21 (1988), pages 81-98, other fibrous polymers were mixed and tested for their triboelectric effect. The article states that many yarns of different types were tested, the best was found to be a combination of polypropylene with modacrylic fibers and is the one also exemplified in U.S. Patent 4,798,850.

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Fiber mixtures of ordinary polytetrafluoroethylene (PTFE) and glass fibres available commercially and are known to have triboelectric properties. Fibres of ordinary PTFE are commonly made by
5 mixing a dispersion of PTFE particles in water with a cellulosic binder, forcing the dispersion through a spinnerette, and heating the resulting fiber until the PTFE particles become sintered together and the cellulose largely burned away.

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SUMMARY OF THE INVENTION

According to the present invention there is provided an electrically charged filter which comprises a blend of clean expanded porous polytetrafluoroethylene
15 fibers and clean polyamide fibers.

Preferably the polyamide fibers are crimped.

Preferably the polyamide is nylon 66.

Preferably the filter is in the form of a carded
batt.

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Preferably the filter is in the form of a felt.

DESCRIPTION OF THE INVENTION

Expanded porous polytetrafluoroethylene (ePTFE) is a form of PTFE that has been described as "a remarkable new material" and one that "differs from other processed forms
25 of Teflon." Teflon resin is ordinary PTFE. Expanded porous PTFE is made by stretching ordinary PTFE at certain rates and temperatures as described in Gore USP 3,953,566. The resulting ePTFE consists of a microporous structure of nodes interconnected by fibrils.

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To obtain fibers of ePTFE a sheet of ePTFE can be slit in fiber widths. Porous expanded PTFE fibers can be obtained from W. L. Gore & Associates, Inc.

The polyamide fibers used herein can be obtained commercially. Representative polyamide fibers include
35 nylon 66, nylon 612 and the like. Nylon 66 is preferred.



To prepare the mixtures of this invention, fibers of ePTFE and fibers of polyamide are mixed in an ordinary carding machine. The fibers are usually about 5 to 7.6 cm long because most carding machines use that length fiber.

- 5 The ration of the two fibers can be any ration that produces triboelectrification between the fibers. Because carding is usually easier to carry out when the majority of

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fibers are polyamide, it is preferred that a preponderance of the blend be polyamide. The fibers become electrically charged during carding. By "electrically charged" is meant that triboelectrification has occurred and an electrical charge has been transferred. The blended fibers hold the electrical charge and it is this charge that effects capture of small particles during filtration operations.

The PTFE and the polyamide are preferable free of any solvent or lubricant that may be present. Any solvent or lubricant present can be removed by washing in an aqueous surfactant bath and rinsing with deionized water. By "clean" is meant that the fibers have no coating of lubricant or anti-static agent, or that any such coating was removed before blending, for example by scouring with a non-ionic detergent. Preferably also, the nylon fibers are crimped.

The carded blend can be needled to form a felt, if desired.

Example 1

200 grams expanded porous PTFE fiber 7.6 cm long and 3 denier was blended with 1000 g crimped nylon 66 fiber 5.08 cm long and 3 denier (obtained from The DuPont Company) in a standard carding machine, and then lightly needled to form a coherent manageable felt batt.

The fibers became electrically charged by the rubbing and friction that occurs during the carding operation, and remain electrically charged due to their close proximity to one another.

The nylon 66 used had been cleaned by washing the fibers in a hot (about 140°C) aqueous bath containing a non-ionic surfactant followed by washing with deionized water. The ePTFE fibers were already free of solvent or lubricant.

A number of samples were tested as described below.

Comparison 1

Ordinary PTFE fibers 7.6 cm long, 3 denier obtained from the DuPont Co. were mixed with identical nylon 66 fibers as were used in Example 1 at the same weight ratio and by the same carding and

needling procedure. A number of samples were prepared by this procedure.

Test Results for Example 1 and Comparison 1 Samples

5 Several samples in the form of batts of ePTFE/nylon 66 prepared according to Example 1 and several samples in the form of batts of ordinary PTFE/nylon 66 were prepared according to Comparison 1.

10 These samples were tested for air filtration efficiency and quality. The test apparatus was a filter media efficiency tester built by W. L. Gore & Associates, Inc. of the type using solution atomization as the aerosol particle source any laser aerosol spectrometers as the aerosol particle detectors. The aerosol particle source was an atomizer (a Model 3076 made by Thermal Systems Inc. of St. Paul, Minnesota), which used a dilute NaCl solution. The aerosol spectrometers were two Model LAS-X particle counters made by Particle Measuring Systems Inc. of Boulder, Colorado, measuring upstream and then downstream of the filter medium under test. The apparatus was under computer control.

15 In Table 1 below the mass of the sample is recorded, the velocity of the aerosol passing through the sample is recorded, resistance to airflow is recorded, efficiency of capture of 0.1 micrometer sodium chloride particles is recorded, efficiency of capture of 0.3 micrometer particles is also recorded. Because all filter media do not have the same resistance to air flow, it is usually better to compare filter media by quality factor (Q_f), which is an accepted measure of performance as determined by the compromise between particle capture efficiency and air permeability. This data appears in the last column of Table 1. Quality factor is defined as

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$$Q_f = \frac{-\log_{10}(P)}{R}$$

As used herein P is the penetration of .1 μ m diameter particles, and R is the resistance to flow in mm of H₂O at a filtration velocity of 3.2 m/min. (10.5 ft/min.)

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1 355 47800

TABLE 1

Sample	Mass (g)	Mixture	Velocity (ft/min.)	Resistance (mm H ₂ O)	Efficiency @ .1 μm	Efficiency @ .3 μm	Quality Factor
	1	ePTFE/Nylon	10.4	0.3118	94.6%	93.3%	4.07
	2	"	10.3	0.3148	95.3%	94.6%	4.23
5	3	"	10.8	0.2892	95.6%	95.6%	4.72
	4	"	10.4	0.3916	95.8%	95.5%	3.51
	5	"	11.2	0.3283	95.5%	95.3%	4.10
	6	"	10.4	0.3017	95.2%	93.8%	4.38
	7	"	10.9	0.2892	95.6%	96.9%	4.70
10	8	"	10.7	0.2733	96.2%	96.1%	5.21
	1	PTFE/Nylon	10.3	0.3066	77.0%	70.2%	2.06
	2	"	10.0	0.3643	77.0%	68.6%	1.75
	3	"	9.9	0.2523	77.6%	71.1%	2.58
	4	"	10.7	0.3904	79.4%	71.6%	1.76
15	5	"	10.7	0.3044	80.6%	74.7%	2.34
	6	"	10.7	0.3606	77.5%	71.9%	1.79
	7	"	10.7	0.5883	83.2%	77.8%	1.32

As is seen, both the efficiency and the quality factor are superior for the ePTFE/nylon 66 batt of the invention.

Comparison Example 2

5 For many applications, a desirable property of filters is cleanliness, specifically the amount of extractable organic contaminant present in the filter media. A representative triboelectret filter made of a felt of a mixture of a polypropylene and modacrylic fibers (Technostat) was obtained from Hepworth Minerals & Chemicals, Ltd. The felt had a mass of 110 g/m². Several combinations of ePTFE and nylon 66 fibers were produced as in Example 1 to compare to the polypropylene/modacrylic felt. The mixtures tested were 20 weight % and 40 weight % ePTFE to nylon 66 by weight. The nylon had been cleaned by washing in a hot bath of non-ionic surfactant, tetrasodium pyrophosphate and deionized water, and rinsed in deionized water. The fibers were mixed in a carding machine and needled to produce a felt that was about 5 oz/yd² (150 g/m²). To test for presence of extractable organic contaminants, a standard Soxhlet extraction test was performed for thirty minutes using trichlorotrifluoroethane as a solvent to compare the cleanliness of the polypropylene/modacrylic combination versus the ePTFE/nylon 66 combination of the invention. For comparison between materials, samples of 10.937 g were tested (equivalent to 100 square inches (645.1 cm²) of 5 ounce/square yard (169.49 g/m²) of material), and the results are given in terms of mg/m² of 5 ounce/square yard (169.49 g/m²) material. The results of the test are as follows:

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	Material	Milligrams of Contaminate (Per m ² of filter material)
	Polypropylene/modacrylic	362.86
	"	348.59
	"	223.05
5	ePTFE fiber/nylon 66 (20% ePTFE)	19.99
	ePTFE fiber/nylon 66 (30% ePTFE)	16.89
	ePTFE fiber/nylon 66 (40% ePTFE)	10.39

10 The felts of ePTFE fiber and nylon 66 fiber showed much less retention of organic contaminants than did the polypropylene/modacrylic felt combination.

To test for filter efficiency, the felts were tested in the filter efficiency tester used further above. Results were as follows:

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Material	Ratio of ePTFE to nylon 66	Efficiency @ .1 m	Resistance mm H ₂ O	Velocity ft/min	Quality Factor
ePTFE fiber/ nylon 66	20/80	96.24	.2733	10.76	5.22
	40/60	97.63	.2971	14.14	5.47
Polypropylene/ modacrylic	N/A	96.67	.2988	10.19	4.94
	N/A	97.09	.2729	10.00	5.63

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An electrically charged filter which comprises a blend of clean expanded porous polytetrafluoroethylene fibers and clean polyamide fibers.

5 2. The filter of Claim 1 wherein the polyamide fibers are crimped.

3. The filter of Claim 1 or 2 wherein the polyamide is nylon 66.

10 4. The filter of Claim 3 in the form of a carded batt.

5. The filter of Claim 3 in the form of a felt.

6. An electrically charged filter substantially as hereinbefore described.

DATED THIS 29th DAY OF February 1996

15 W. L. GORE & ASSOCIATES

By Its Patent Attorneys:

GRIFFITH HACK & CO.,

Fellows Institute of Patent Attorneys of Australia



INTERNATIONAL SEARCH REPORT I

International Application No
PCT/US 93/07084

<p>A. CLASSIFICATION OF SUBJECT MATTER IPC 5 B01D39/16 B03C3/30</p>		
<p>According to International Patent Classification (IPC) or to both national classification and IPC</p>		
<p>B. FIELDS SEARCHED</p>		
<p>Minimum documentation searched (classification system followed by classification symbols) IPC 5 B01D B03C D04H</p>		
<p>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched</p>		
<p>Electronic data base consulted during the international search (name of data base and, where practical, search terms used)</p>		
<p>C. DOCUMENTS CONSIDERED TO BE RELEVANT</p>		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO, A, 93 12281 (W.L. GORE & ASSOCIATES, INC.) 24 June 1993 see abstract see page 8, line 16 - page 9, line 3 see claims 1-3, 6, 7, 14, 15	1
P, A	---	5
A	FR, A, 1 563 320 (THERMO ELECTRON CORPORATION) 11 April 1969 see page 1, line 1 - line 8 see page 1, line 42 - page 2, line 6 see page 3, line 26 - page 4, line 2 see claims	1, 3
A	EP, A, 0 395 331 (JAPAN GORE-TEX, INC.) 31 October 1990 see claims	1

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<p><input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.</p>		
<p>* Special categories of cited documents :</p> <p>*A* document defining the general state of the art which is not considered to be of particular relevance</p> <p>*E* earlier document but published on or after the international filing date</p> <p>*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>*O* document referring to an oral disclosure, use, exhibition or other means</p> <p>*P* document published prior to the international filing date but later than the priority date claimed</p> <p>*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>*&* document member of the same patent family</p>		
<p>Date of the actual completion of the international search</p> <p>11 November 1993</p>		<p>Date of mailing of the international search report</p> <p>22. 11. 93</p>
<p>Name and mailing address of the ISA</p> <p>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</p>		<p>Authorized officer</p> <p>STEVNSBORG, N</p>

INTERNATIONAL SEARCH REPORT

Int. l. Application No

PCT/US 93/07084

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 488 822 (MITSUI PETROCHEMICAL INDUSTRIES, LTD.) 3 June 1992 see page 2, line 46 - line 55 see claims 1-3; example 1 ---	1
A	DD,A,235 831 (KOMBINAT ILKA LUFT- IND KÄLTETECHNIK) 21 May 1986 see claim ---	1
A	GB,A,2 190 689 (NATIONAL RESEARCH DEVELOPMENT CORPORATION) 25 November 1987 see the whole document ---	1,5
A	WO,A,91 14503 (WHEWAY PLC) 3 October 1991 see the whole document -----	1-3,5

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/US 93/07084

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