



PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

Filamentary Material

WE, EASTMAN KODAK COMPANY, a Company organized under the Laws of the State of New Jersey, United States of America, of 343 State Street, Rochester, New York 14650, United States of America (Assignees of George Patrick Touey and Robert Chester Mumpower II), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a material that is particularly useful for the manufacture of tobacco smoke filters and to tobacco smoke filters made therefrom.

There exist in the vapour phase of tobacco smoke certain small, but yet highly important, quantities of various gaseous and solid compounds which exhibit a specific toxic action on the human tissue with which they come into contact. Thus, in an effort to reduce or, if possible, completely eliminate the presence of these deleterious tobacco smoke constituents the tobacco industry has extensively investigated the use of various types of tobacco smoke filters. Of all these various types of tobacco smoke filters tested those made from crimped textile-like strands of cellulose acetate are by far the most widely accepted.

There are a number of reasons why cellulose acetate or other like spun fibres have enjoyed such popularity in the tobacco filter industry. For example, cellulose acetate is relatively inexpensive and readily available and can be continuously and rapidly processed into filter rods on existing commercial equipment. Another important attribute of cellulose acetate filter tow is its sensitivity to liquid, essentially non-volatile, plasticizers. By this it is meant

that during the continuous processing of the filter rods a nonvolatile, oily, liquid plasticizer such as glycerol triacetate can be added to the tow to produce a firm filter element. This firmness of the cellulose acetate filter element is produced by the plasticizer liquid within the filter at random points of contact. Such a rigid filter structure can then be easily and cleanly cut into smaller segments the size of a filter tip.

The use of such a plasticizer hardening or bonding agent in producing tobacco smoke filter elements has many obvious advantages over the use of conventional adhesives of the type which make the tow tacky as soon as they are applied. For example, the use of tacky adhesives is objectionable because they cause the rapidly moving tow to adhere to the various parts of the filter making machines. Also, the use of such conventional tacky adhesives requires the removal of water or an organic solvent from either the tow or the finished filter rod.

Of course a cellulose acetate filter tow has numerous other features which are highly desirable, if not mandatory, for any tobacco smoke filter, especially those to be used on cigarettes. These additional features include being nontoxic, nonodorous, white, and stable to storage under varying conditions of humidity and temperature. However, even in view of these many attributes, filter tow made from cellulose acetate or other like spun filamentary materials leave something to be desired in reference to their general physical characteristics. For example, these filamentary materials are inherently straight with very smooth outer surfaces. In addition to this they are relatively weak and, in the case of cellulose acetate, somewhat more expensive to produce than certain other types of synthetic materials.

[Price 4s. 6d.]

gradually dissolving and bonding together adjacent fibres

It is now well known in the art of tobacco smoke filtration that a fibre which has a smooth outer surface and is inherently straight cannot successfully be used without physical modification in a filter since the fibres will lie in a more or less parallel arrangement and thus permit smoke particles to channel through its length and into the smoker's mouth. The first efforts by the industry to alleviate this basic and undesirable characteristic of such straight, smooth filamentary materials was by subjecting them to a crimping operation. This crimping operation was accomplished through the use of various methods and apparatus designed for placing any desired crimp count (defined as one-half the number of fibre bends per linear inch) into a substantially straight filamentary material. For instance, these crimps have been produced mechanically by means of intermeshing gear-like elements, by means of a "stuffer-box" type crinkler, or by twisting the fibres, setting the twist, and then untwisting. Needless to say, although this crimping operation is somewhat time consuming and adds to the over-all expense of the finished filtered product it nevertheless has been reasonably successful and is widely accepted by the industry since it not only results in the breaking up of those channels which would otherwise exist in the interstices between the fibres, but also increases the bulk factor of the fibres. This crimping of fibrous tow materials is not, however, without its own drawbacks. Besides the added time and expense required for producing such crimped tow, these crimped tows, when made of cellulose acetate or a like material that is relatively weak, generally do not have a crimp count in excess of about 16 per inch. Furthermore, the low strength of the filter tow creates special handling and processing problems while the low crimp count capability of the tow limits its bulk factor to a value below that normally desired.

In accordance with the present invention there is provided a filamentary material for filters comprising a continuous mass of crimped filamentary-like ribbons formed from a molecularly oriented film composed of 40-70 percent of polyolefin and 30-60 percent of polystyrene, said ribbons being discontinuously and randomly separated from each other yet interconnected with the mass, the flat filaments as a whole being in substantial alignment longitudinally of the mass and each flat filamentary-like ribbon having integral hair-like protrusions extending therefrom. According to the present invention there is also provided a tobacco smoke filter comprising a structurally unitary rod-like mass of filamentary-like ribbons and a wrapper encircling the mass, the ribbons

being formed from a molecularly oriented film composed of 40-70 percent of polyolefin and 30-60 percent of polystyrene, each flat filamentary-like ribbon of the mass being crimped and substantially coextensive and interconnected with the filamentary mass, the ribbons as a whole being aligned substantially longitudinally of the mass, with each ribbon having a plurality of integral hair-like protrusions, and a plurality of the ribbons being bonded to contiguous ribbons at random points of contact by surface solvation bonds from the action of a plasticizer.

In the accompanying drawing:

Figure 1 is a diagrammatic illustration, to an enlarged scale, of a bundle of smooth surface filaments of known type, such as cellulose acetate; and

Figure 2 is a diagrammatic illustration, to an enlarged scale, of a bundle of fibrillated and bonded polyolefin-polystyrene filaments made in accordance with the present invention;

Referring to Figure 1, reference numeral 10 designates a bundle of crimped cellulose acetate fibres. As will be noted, the outer surface of each fibre 12 is substantially smooth and thus large channels or openings 14 exist between the various fibres through which the tobacco smoke may pass without encountering any filtering action. For purposes of illustration the fibres are shown bonded together at 16 by a suitable high boiling, oily plasticizer.

Figure 2 illustrates a bundle 18 of fibres taken from a highly crimped filter tow formed in accordance with this invention from a multiplicity of highly fibrillated continuous fibres composed of a composition comprising from 40 to 70 percent polyolefins and from 60 to 30 percent polystyrene. As will be observed, the individual filaments 20 of this type tow are entirely different in appearance from the smooth surfaced, substantially rounded filaments 12 of Figure 1, obtained from a conventional wet, dry, or melt spinning process. The polyolefin-polystyrene fibres 20 are flat and ribbon-like in appearance with a thickness of from 15 to 100 microns which permits them to be fibrillated to produce the desired results. Since the fibres 20 are fractured during their preparation, numerous fine hair-like fibrils 22 of varying thickness and length will be produced and will extend from their surface in all directions. As can be seen, these fine splinter fibrils 22, which have a thickness from about 0.5 microns to about 15 microns, extend into the spaces 24 that exist between the various fibres 20 and therefore allow less of the solid tobacco smoke constituents carried in the effluent stream of smoke to pass therethrough. This coupled with the fact that these fine fibrils 22 are uniformly

dispersed between the coarser trunk or continuous portion of the fibres 20 is believed to be the reason why filters formed from this tow can remove a higher percentage of the smoke particles (tar) found in cigarette smoke than other similar heretofore known filter materials without at the same time producing such a high pressure drop (resistance to draw) that they would be impractical for use on cigarettes. Furthermore, since these hair-like protruding fibrils 22 are firmly attached by one end to the larger trunk portions of the filament and are, therefore, an integral part of the main ribbon-like filaments 20 they do not fall off nor can they be wiped off as the tow 18 passes through the many parts of the cigarette filter rod making machine.

The filaments 20 may be fibrillated to the point that the splinter or fibril filaments 22 represent from 10 to 70 percent of the total make-up of the crimped filter tow 18. Below this value of fibrillation there are not enough of these fine fibrils in the tow to render it very effective as a filter material while above this value the main continuous trunk portion of the filaments 20 is so reduced in size as to substantially weaken the tow thereby making it too weak to be processed into filter rods on standard equipment. The preferred ratio of fibrillation of the tow has been found to be 20 to 50 percent splinter fibrils and 50 to 80 percent ribbon-like filaments. The total number of "filaments" in the filter tow, which includes both the ribbon-like filaments 20 and fibrils 22, can vary over a wide range since their thickness can vary from 0.5 to about 15 microns for the fibril type to over 100 microns for the heavier ribbon-like type. Therefore, in terms of total denier it can be said that the tow 18 may vary from about 20,000 total denier to 150,000 total denier.

As is well known, polyolefin fibres *per se* cannot be bonded together with a high boiling, oily plasticizer. Thus, to avoid the use of tacky adhesives the filaments 20 were made plasticizer sensitive by placing at least 30 percent by weight of polystyrene in the polyolefin spinning dope. Because of the presence of the plasticizer-soluble polymer in the filaments 20, the tow 18 is rendered plasticizer sensitive and can be bonded together by a plasticizer as illustrated at 26 in Figure 2. As will be appreciated by those working in the filter art, this capability of the polyolefin-polystyrene filter material of this invention is of utmost importance. The percentage of polystyrene added is somewhat critical with the optimum amount of polystyrene additive having been found to be between 35 and 50 percent by weight. Lower amounts of polystyrene give a filter tow that cannot be hardened by the application of a plasticizer while, conversely, too much poly-

styrene (over 60 per cent) makes the tow too weak to withstand the tensions applied to it during the filter rod making process.

The type of polyolefin in the mixture of polyolefin and styrene from which the filaments of the tow are made can be any of the film-forming types with molecular weights from about 10,000 to 900,000. Preferably the molecular weight should be between 25,000 and 200,000. Their softening points should be between 70° and 160°C. Of the several polyolefin materials produced industrially the preferred ones are polyethylene and polypropylene or copolymers of ethylene and propylene.

Concerning the polystyrene, it can have a molecular weight of from 5,000 to 500,000; the preferred range in molecular weight being from 10,000 to 100,000. The softening point of the polystyrene should fall between 100°C. and 160°C., and preferably between 130°C. and 150°C. Both polymers may contain substituent grouping providing such groupings do not change the softening points beyond those stipulated.

The fibrillated filaments from which the filter tow of the invention is made can be produced by any of the known techniques for fracturing elongated polymeric strands so that splinter type fibrils extend from them. However, the preferred technique is the one disclosed in our co-pending application No. 14439/66 (Serial No. 1145976). This method of fibrillating ribbon-like filaments is simple, economical, and can be carried out in one continuous operation. For example, a blend of the polyolefin and the polystyrene is melt extruded into a film which is then continuously drafted and fibrillated into fractured, ribbon-like filaments. A number of these filaments are gathered together to produce the tow of this invention.

For best results the polyolefin-polystyrene filter tow should be crimped. The number of crimps placed in filter tow 18 should be sufficiently high to raise the bulk factor of the tow to the point where it fills out the cigarette rod. This crimp value has been found to be between 15 and 50 crimps/inch with the preferred number of crimps being between 20 and 30. These crimps can be applied to the tow by any suitable method, such as a conventional "stuffer box" crimping device known in the textile art.

A further understanding of the invention will be had from a consideration of the following examples that may be used in actual commercial practice and are set forth to illustrate certain preferred embodiments.

Example 1

Highly fibrillated filaments formed from a melt extruded film of 50 percent polypropylene (mil. wt. 32,000) and 50 percent



polystyrene (mol. wt. 10,000) were prepared according to the procedure given in the aforementioned Application No. 14439/66 (Serial No. 1145976). Microscopic examination of the filaments indicated that there were many tiny hair-like projections extending outwardly from the larger trunk portion of the continuous filaments and that these fibrils had a thickness of from 1 to 10 microns. A 250-ft. length of tow of 60,000 total denier was produced from these filaments and was given 30 crimps/inch through the use of a stuffer box. The crimped tow was then processed into filter rods (25.4 mm. circumference). During the passage of the tow through the filter making machine it was opened or bloomed out at one point and sprayed with diethyl phthalate. The plasticizer treated and paper (cigarette paper) wrapped rods contained 10 percent diethyl phthalate; after 60 minutes they had sufficient rigidity so that they could be cleanly cut into 15 mm. filter segments.

Several of these filters were attached to standard cigarettes which had been 85 mm. in length but which were shortened to 70 mm. to compensate for the length of the filters. These filter cigarettes (now 85 mm.) were smoked to 35 mm. butt lengths on an automatic smoking machine. The weight percentage of smoke particles (tar) removed by the filters is shown in Table 1. The pressure drops (resistance to draw) of the

filters were also obtained and the average value was calculated (Table 1).

Example 2

Highly fibrillated filaments formed from a melt extruded film of 65 percent polyethylene (mol. wt. 42,000) and 35 percent polystyrene (mol. wt. 12,500) were prepared according to the procedure described in Application No. 14439/66. (Serial No. 1145976). A 200-ft. crimped (25 crimps/inch) tow of 65,000 total denier was prepared from these filaments which consisted of about 35 percent of the fibril type and 65 percent of the heavier and parallel type. During the passage of the tow through the cigarette filter rod making machine it was bloomed out at one point and sprayed with dibutyl phthalate. The plasticizer treated and cigarette paper wrapped rods contained 8 percent plasticizer. About 45 minutes after the filter rods were made they were firm enough to be cleanly cut into 15 mm. filter tip segments.

Several of these filter tips were tested for pressure drop and for their effectiveness in removing tar from cigarette smoke in the manner described in Example 1. The results obtained are listed in Table 1. Commercial 15 mm. filters made from a tow of cellulose fibres (5 den./fil) were also tested for pressure drop and for their effectiveness in removing tar from cigarette smoke. For comparative purposes these results are also listed in Table 1.

Table 1

Filter (15 mm.)	Average Pressure Drop of Ten Filters (Inches H ₂ O)	Percent Tar Removed
Fibrillated filaments of polyethylene and polystyrene	2.0	41
Commercial cellulose acetate filter of 5 denier/filament fibres	2.0	25

Example 3

Highly fibrillated filaments formed from a melt extruded film of 45 percent polypropylene (mol. wt. 60,000) and 55 percent (mol. wt. 15,000) were prepared according to the procedure disclosed in Application No. 14439/66. (Serial No. 1145976). A 200-ft. crimped tow (30 crimps per inch) of 70,000 total denier was prepared from these filaments which consisted of about 50 percent the fibril type and 50 percent the heavier parallel type. During the passage of the tow through the rod making machine it was bloomed out at one point and treated with 10 percent di-(2-ethylhexyl) phthalate by means of a wicking device which wiped the

plasticizer on the filaments. The final paper wrapped filter rods were firm enough to be cleanly cut into small 15 mm. filter segments about 1 hour after they were prepared.

Several of these filter tips were tested for pressure drop and for their effectiveness in removing tar from cigarette smoke in the manner described in Example 1. A 15 mm. commercial filter of cellulose acetate (1.6 denier/filament fibres) was tested in a similar manner. The results shown in Table 2 indicate that a filter of the finest commercially available cellulose acetate fibres is still not quite as effective as one which can be prepared from the inexpensive tows disclosed in this invention.

acetate



Table 2

	Filter (15 mm.)	Average Pressure Drop of Ten Filters (Inches H ₂ O)	Percent Tar Removed
5	Fibrillated filaments of polyethylene and polystyrene	3.5	55
	Commercial cellulose acetate filter of 5-denier/filament fibres	3.4	50

10 The highly-crimped, fibrillated, plasticizer sensitive polyolefin-polystyrene filter tow of this invention offers numerous advantages over those tows heretofore known and used to produce tobacco smoke filter elements. For example, not only is the tow easily 15 bonded by known and commercially acceptable plasticizer, thus alleviating the need for using heavy wrapper papers or tacky adhesives, but the polyolefin-polystyrene, ribbon-like filaments are strong enough to 20 permit high fibrillation and easy processing in existing commercial equipment. This high degree of fibrillation results in a more effective tar and nicotine filter than was heretofore thought possible. Furthermore, 25 not only are the filaments less expensive to produce than conventional cellulose acetate type fibres, but the high fibrillation and extremely high crimp count that can be placed in the new filaments greatly increases 30 the bulk factor of a filter tow made from the fibres. The desirable ramifications of this high bulk factor, including the fact that with a higher crimp level less fibres of a given denier/filament are required to fill out the 35 filter element to its proper size, is readily apparent to those working in the filter tow art. The other advantages of the new polyolefin-polystyrene tow, such as its low moisture retention characteristic and ease of 40 production to name only a few, will also be appreciated by those working in the field.

WHAT WE CLAIM IS:—

1. A filamentary material for filters 45 comprising a continuous mass of crimped filamentary-like ribbons formed from a molecularly oriented film composed of 40-70 percent of polyolefin and 30-60 percent of polystyrene, said ribbons being discontinu- 50 ously and randomly separated from each other yet interconnected with the mass, the flat filaments as a whole being in substantial alignment longitudinally of the mass and each flat filamentary-like ribbon having 55 integral hair-like protrusions extending therefrom.

2. A filamentary material according to Claim 1, wherein the polyolefin has a 60 molecular weight from 10,000 to 900,000 with a softening point between 70°C. and 160°C.,

and the polystyrene has a molecular weight of from 5,000 to 500,000 with a softening point between 100°C. and 160°C.

3. A filamentary material according to Claim 2, wherein the polyolefin is selected 65 from the group consisting of polypropylene and polyethylene.

4. A filamentary material according to any one of the preceding claims, wherein the total denier of tow falls between 20,000 and 70 150,000.

5. A filamentary material according to any one of the preceding claims, wherein the tow has a crimp count between 15 and 50 75 crimps per inch.

6. A filamentary material according to any one of the preceding claims, wherein the fractured ribbons are 15 to 100 microns wide with a plurality of hair-like protrusions 80 0.5 to 15 microns thick.

7. A filamentary material according to any one of the preceding claims, wherein the fine splinter fibrils represent from about 10 to 70 percent of the total number of filaments 85 in the tow.

8. A tobacco smoke filter comprising a structurally unitary rod-like mass of filamentary-like ribbons and a wrapper encircling the mass, the ribbons being formed 90 from a molecularly oriented film composed of 40-70 percent of polyolefin and 30-60 percent of polystyrene, each flat filamentary-like ribbon of the mass being crimped and substantially coextensive and inter-connected with the filamentary mass, the ribbons as a 95 whole being aligned substantially longitudinally of the mass, with each ribbon having a plurality of integral hair-like protrusions, and a plurality of the ribbons being bonded to contiguous ribbons at random points of 100 contact by surface solvation bonds from the action of a plasticizer.

9. A tobacco smoke filter according to Claim 8, wherein the ribbons are 15 to 100 105 microns in width with a plurality of hair-like protrusions being 0.5 to 15 microns thick and represent 10 to 70 percent of the total number of filaments within the filter.

10. A tobacco smoke filter according to Claim 9, wherein the filaments have 15 to 110 50 crimps per inch.

11. A filamentary material according to

polypropylene

1.6

10,000



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Table 2

Filter (15 mm.)	Average Pressure Drop of Ten Filters (Inches H ₂ O)	Percent Tar Removed
Fibrillated filaments of polyethylene and polystyrene	3.5	55
Commercial cellulose acetate filter of 5-denier/filament fibres	3.4	50

The highly-crimped, fibrillated, plasticizer sensitive polyolefin-polystyrene filter tow of this invention offers numerous advantages over those tows heretofore known and used to produce tobacco smoke filter elements. For example, not only is the tow easily bonded by known and commercially acceptable plasticizer, thus alleviating the need for using heavy wrapper papers or tacky adhesives, but the polyolefin-polystyrene, ribbon-like filaments are strong enough to permit high fibrillation and easy processing in existing commercial equipment. This high degree of fibrillation results in a more effective tar and nicotine filter than was heretofore thought possible. Furthermore, not only are the filaments less expensive to produce than conventional cellulose acetate type fibres, but the high fibrillation and extremely high crimp count that can be placed in the new filaments greatly increases the bulk factor of a filter tow made from the fibres. The desirable ramifications of this high bulk factor, including the fact that with a higher crimp level less fibres of a given denier/filament are required to fill out the filter element to its proper size, is readily apparent to those working in the filter tow art. The other advantages of the new polyolefin-polystyrene tow, such as its low moisture retention characteristic and ease of production to name only a few, will also be appreciated by those working in the field.

WHAT WE CLAIM IS:—

1. A filamentary material for filters comprising a continuous mass of crimped filamentary-like ribbons formed from a molecularly oriented film composed of 40-70 percent of polyolefin and 30-60 percent of polystyrene, said ribbons being discontinuously and randomly separated from each other yet interconnected with the mass, the flat filaments as a whole being in substantial alignment longitudinally of the mass and each flat filamentary-like ribbon having integral hair-like protrusions extending therefrom.

2. A filamentary material according to Claim 1, wherein the polyolefin has a molecular weight from 10,000 to 900,000 with a softening point between 70°C. and 160°C.,

and the polystyrene has a molecular weight of from 5,000 to 500,000 with a softening point between 100°C. and 160°C.

3. A filamentary material according to Claim 2, wherein the polyolefin is selected from the group consisting of polypropylene and polyethylene.

4. A filamentary material according to any one of the preceding claims, wherein the total denier of tow falls between 20,000 and 150,000.

5. A filamentary material according to any one of the preceding claims, wherein the tow has a crimp count between 15 and 50 crimps per inch.

6. A filamentary material according to any one of the preceding claims, wherein the fractured ribbons are 15 to 100 microns wide with a plurality of hair-like protrusions 0.5 to 15 microns thick.

7. A filamentary material according to any one of the preceding claims, wherein the fine splinter fibrils represent from about 10 to 70 percent of the total number of filaments in the tow.

8. A tobacco smoke filter comprising a structurally unitary rod-like mass of filamentary-like ribbons and a wrapper encircling the mass, the ribbons being formed from a molecularly oriented film composed of 40-70 percent of polyolefin and 30-60 percent of polystyrene, each flat filamentary-like ribbon of the mass being crimped and substantially coextensive and inter-connected with the filamentary mass, the ribbons as a whole being aligned substantially longitudinally of the mass, with each ribbon having a plurality of integral hair-like protrusions, and a plurality of the ribbons being bonded to contiguous ribbons at random points of contact by surface solvation bonds from the action of a plasticizer.

9. A tobacco smoke filter according to Claim 8, wherein the ribbons are 15 to 100 microns in width with a plurality of hair-like protrusions being 0.5 to 15 microns thick and represent 10 to 70 percent of the total number of filaments within the filter.

10. A tobacco smoke filter according to Claim 9, wherein the filaments have 15 to 50 crimps per inch.

11. A filamentary material according to



* C L M S *

Claim 1, substantially as hereinbefore described.

12. Tobacco smoke filters according to Claim 8, constructed substantially as herein-
5 before described.

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