Oct. 10, 1967 R. N. THOMSON 3,346,682

METHOD FOR MAKING A FILTERING MEDIUM FROM PLEXIFILAMENTARY MATERIAL

Filed March 16, 1962

FIG. 1

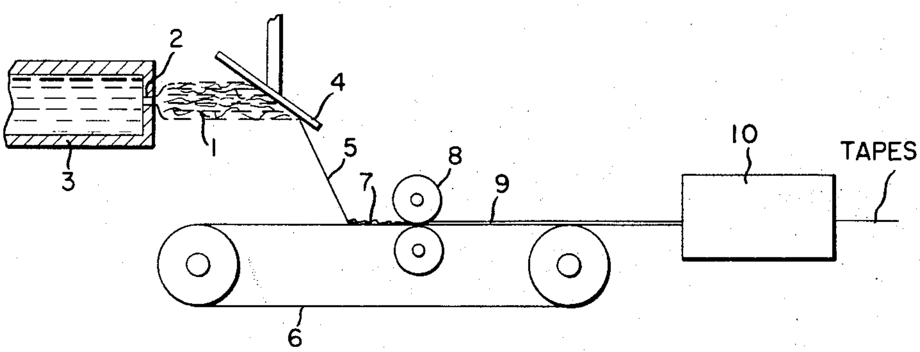

FIG. 2

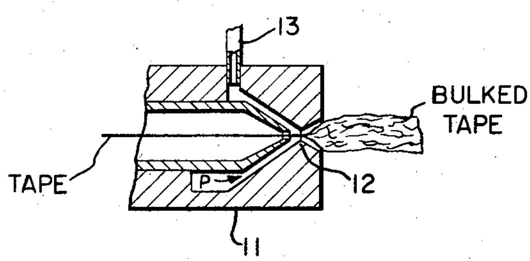

FIG. 3

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United States Patent Office

3,346,682

Patented Oct. 10, 1967

3,346,682

METHOD FOR MAKING A FILTERING MEDIUM FROM PLEXIFILAMENTARY MATERIAL

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Filed Mar. 16, 1962, Ser. No. 180,079

4 Claims. (Cl. 264—93)

This invention concerns non-woven tapes which may be passed through a turbulent fluid jet to form low density, highly efficient cigarette filters.

The cigarette filter art has grown rapidly in recent years. The art is complex because there are so many important aesthetic and practical features which must be built into any cigarette filter if it is to be accepted commercially. For example, the filter must effectively remove particulate matter without removing too much of the desired taste components. At the same time the filter must not require an excessive pressure drop to get an acceptable flow of smoke vapor; otherwise the smoker may have to drag excessively on the cigarette.

In the past it has been common to prepare cigarette filters from fiber tows, or from sheets of a creped paper which are stacked, crumpled or rolled-up to form the filter element.

Belgian Patent 568,524, corresponding to U.S. Patent 3,081,519, issued March 19, 1963, describes plexifilaments of linear polyethylene which may be used as filters for tobacco smoke in cigarettes. The plexifilaments which are used in these filters are strands composed of a three-dimensional network of extremely thin film-fibrils with very high surface area. While this product has remarkable filtering efficiency, it is difficult to process into cigarette filters. To begin with, in the process of the Belgian patent, the strand is spun at such high speeds that it cannot be wound up in a conventional manner. If the strand is collected in the form of a cake, considerable difficulty is encountered in back-winding the strand because of its tendency to cling to itself and to entangle. Moreover, the provision of strands in the proper size for the preparation of the filters requires great care.

A flexible coherent non-woven sheet product may be made by collecting the product of Belgian 568,524 on a moving belt so that the strand overlaps itself in a random fashion. This sheet is too weak to handle in continuous process cigarette making equipment. On the other hand, if the sheet is calendered and cigarette filters cut therefrom, it is found that the pressure drop across such filters is excessive when used in quantity sufficient to provide adequate filtration.

An object of this invention is to provide a plexifilamentary material in a form suitable for use in a cigarette filter-making process.

Another object is to provide a process for converting a plexifilamentary material to a tobacco smoke filter without encountering entanglement or slub formation during processing.

These objects are accomplished by preparing a plexifilamentary material in the form of a continuous thin tape having a denier between 10,000 and 50,000 and a density of 0.15 to 0.75 g./cm.^{3}, passing the tape through a turbulent slot-shaped fluid jet to obtain a bulky tow having a cross-sectional area which is 5 to 100 times as large as the cross-sectional area of the unbulked tape.

FIGURE 1 is a flow chart of the tape preparation, illustrating spinning, sheet formation, calendering and cutting.

FIGURE 2 shows the manner and apparatus in which the tape is bulked.

FIGURE 3 is a cross-sectional view of a plexifilamentary tape at about four-fold magnification with the strands partially separated.

The plexifilamentary tape employed in this invention is made from plexifilamentary strands, which are characterized morphologically by a three-dimensional network of film-fibril elements. The film-fibril elements are extremely thin, being less than 4 microns thick. In the preferred products, the film-fibrils are less than two microns thick. The film-fibril elements are at least five times as wide as they are thick, the actual width being between about 1 micron and about 1,000 microns.

Preferably, more than half of the fibrils have lengths under 1.5 cm. (i.e., between points of attachment). The tie points, being spatially arranged in various planes along the width, length, and depth of the strand are responsible for the three-dimensional structure which results.

The predominantly longitudinal orientation of the film-fibrils of all plexifilament strands is readily apparent from the fact that all such strands are much more resistant to tearing or breaking transversely than to splitting lengthwise. The general coextensive alignment of the fibrous elements in the direction parallel to the strand axis is easily discernible to the naked eye for most plexifilamentary species.

The plexifilamentary strands of the invention are made of crystalline polymer. It has been found, quite unexpectedly, that the pellicular material in the as-spun strand when consisting of a crystalline polymer is substantially oriented as measured by electron diffraction, i.e., it has electron diffraction orientation angles smaller than 90°. It is believed that the high strength of the plexifilamentary strand as spun is closely related to the crystalline orientation within the film-like ribbon and in the structural arrangement of the fibrils themselves in the strand. In the preferred crystalline oriented products of the invention, the film-fibrils have electron diffraction angles of less than 55°. The orientation of the crystallites in the film-fibrils is in the general direction of the film-fibril axis.

In accordance with the present invention, the plexifilamentary strands are first assembled into the form of a loose sheet wherein the film-fibril networks are laid one on top of the other in overlapping multidirectional, intersecting patterns.

The sheet may be prepared by hand or mechanically. In an especially preferred direct lay-down process a solution of a synthetic organic crystallizable polymer under pressure and at a temperature well above the boiling point of the solvent is spun through an orifice, whereupon the solvent evaporates instantaneously and forms a strand consisting of a three-dimensional film-fibril network. The strand as it forms strikes a deflector which spreads it out into a web-like form many times greater in width than the original strand. The web-like form is directed immediately to a moving belt without any intervening hold-up of the strand between the deflector and the moving belt. The deflector may be any flat surface such as a metal plate set at an angle to the issuing stream of polymer and at a point within a half inch of the extrusion orifice. FIGURE 1 shows polymer stream **1** issuing from orifice **2** of autoclave **3** and striking deflector plate **4**. The web **5** which leaves the deflector plate is collected on moving belt **6** as sheet **7**. The material in the autoclave is a proper spinning composition as shown in the Belgian patent. The continuous plexifilament strand may of course be cut into suitable lengths at a time prior to laydown on the moving belt.

The loose batt product may be treated in several ways. It may be compacted in a direction normal to the plane of the batt by means of coacting, compressive plates or rollers. Thus in FIGURE 1 the deposited uncompressed sheet 7 on moving belt 6 is calendered at rolls 8 to form compressed sheet 9. Pressure is uniformly applied at the calender rolls. On leaving the calender rolls, the sheet may be fed directly to a cutter 10 to be cut into tapes of the desired width. The plexifilamentary sheet which is cut into tape is used in its lightly compacted form where it has a density of about .034 g./cm.^{3} but is preferably used in denser calendered forms having a density between 0.15 g./cm.^{3} and 0.75 g./cm.^{3}. The maximum sheet thickness however, is 0.5 cm. (200 mil). In a preferred embodiment, the sheet is less than 0.125 cm. (50 mil) thick.

For the purposes of preparing cigarette filters, however, it is preferred that the original sheet be composed of plexifilaments with film-fibrils having a thickness less than 4 microns.

In the practice of this invention plexifilamentary sheets having weights in the range from about 0.25 oz./yd.^{2} to about 10 oz./yd.^{2}, preferably 1 to 3 oz./yd.^{2}, are cut into tapes of uniform width, by means of a cutting device. For example, a 40-inch sheet may be simultaneously slit into 40 one-inch tapes which may be wound up continuously on separate reels using a cutting (slitting) machine of the type sold by Hobbs Manufacturing Co. having a series of parallel score cutting discs. These tapes may be wound on reels resembling those used for movie film. Alternatively, many separate rolls may be wound up on the same cylindrical core after slitting. The tapes may be between 0.3 inch and 10 inches in width. However, the tapes are preferably between 0.5 and 2 inches in width since they may then be easily processed into cigarette filters after bulking in an air jet without folding or rolling.

One of the preferred sheet forms is a moderately soft sheet which has been embossed by means of a patterned roll. This sheet has dense lines, dots or other patterns impressed upon it. The resulting embossed or "quilted" sheet may be transparent at the dense spots (wherever the raised portion of a patterned calender roll has pressed the sheet) but is otherwise opaque. The tape may be embossed either by a room temperature process or by hot processes. The embossed tapes are particularly useful for uniformizing the product, giving after bulking a fluffy tow without knots or slubs even when very high fluid pressures are used for bulking.

The tape which is to be used for the filter element may be formed by cutting a wide sheet for example 40 inches wide or it may be formed directly as a narrow ribbon simply by flash spinning onto a belt which is relatively close to the spinneret. It is essential, however, that the sheet or tape be calendered by either a smooth or by an embossed roll before bulking in a jet; otherwise the product will be too weak to process continuously.

The plexfilamentary tapes which are useful for cigarette filters are made from crystalline synthetic polymers including polyhydrocarbons such as linear polyethylene and polypropylene, polyethers such as polyoxymethylene, polyamides such as polyhexamethylene adipamide, and polyesters such as polyethylene terephthalate. It is preferable, however, to make the tapes from plexfilaments composed of crystalline oriented polyhydrocarbon film-fibrils and particularly of linear polyethylene.

In practicing the bulkin process of this invention the plexifilamentary tapes are converted to a bulky voluminous tow by forwarding them through a jet 11 such as shown in FIGURE 2 having a slot-shaped passage 12. The passageway at its narrowest point has thickness and width which are no greater than twice the tape thickness and width, respectively. The air flow P against the tape in the jet illustrated is directed at an angle to the plane of the tape between 10 and 45 degrees. The air pressure in the supply pipe 13 to the jet is between 30 and 90 p.s.i.g. Other types of fluid jet bulking devices are also suitable. In operating the bulking process the plexifilamentary tape is fed into the jet at speeds of 5 to 150 yards per minute or even faster. The tape speed into the jet is controlled by a positively driven pair of rolls. A bulky tow appears immediately as the tape exits from the jet, the cross-sectional area being increased as much as 100-fold. Surprisingly, the cross section is increased in this dramatic fashion without any appreciable change in length and without the fibrils being broken to any appreciable extent. The fluffy tow which is obtained by the jet treatment has a density between 0.001 and 0.120 g./cm.^{3} and has a surface area of at least 2 meter^2/g.

The bulked tow is pulled away from the jet at approximately the same speed ($\pm 20\%$ and preferably $\pm 5\%$) as it is fed into the jet. Regardless of whether the take-up rate is faster or slower than feed rate, a bulky product is obtained.

In one form of the process the bulked tow passes directly from the jet into the gathering flume of a cigarette-making machine. By using a coupled bulking and filter rod-making process, the tow may be used in its fluffiest form. Since there is no need for shipping or transporting the tape in the bulky form, there is no danger of losing the bulky character which is essential for uniform filtration and uniform pressure drop. In this coupled process the tow is reduced in size as it passes through the flume and is formed into the shape of a cigarette filter. The filter usually has the same diameter as the tobacco portion of the cigarette. The density of the finished filter may be varied depending on the efficiency of filtration desired. The density will nevertheless be in the range 0.018 g./cm.^{3} to 0.08 g./cm.^{3}.

EXAMPLE I

A 14% solution of linear polyethylene having a melt index of 0.6 was prepared using fluorotrichloromethane, as solvent. The solution was prepared in an autoclave at 185° C. and 640 p.s.i.g. (autogenous pressure). The solution was allowed to pass through an adjustable needle valve with a small pressure drop, into a pipe having an L-bend leading to a spinneret. The solution passed through the spinneret in a horizontal direction. The spinneret had a round orifice with a diameter of 0.032 inch and a passage length of 0.062 inch.

The flowing stream of solvent vaporized instantly as the solution flowed from the spinneret and a highly fibrillated strand formed. This strand issued horizontally and impinged against a baffle arranged at an angle of 70° to the axis of the strand and was directed downward to a moving belt. The baffle oscillated in such a manner that a uniform sheet was formed on the moving belt. The belt, moving at 25 ft./min., carried the sheet to the nip of a pair of rolls which lightly compacted the sheet at room temperature, the force between the rolls being about 10 lbs./inch of roll width.

The sheet which was obtained after compaction by the rolls had an apparent density of about 0.252 g./cm.^{3}. It was a soft, white opaque sheet product about 60 cm. wide having a thickness of 0.0435 cm. (17.1 mil) and was composed entirely of continuous strands of highly fibrillated material. The sheet product was cut lengthwise into 26 separate ribbons each 2.25 cm. wide by passing through a multiple disc cutter. The individual ribbons or tapes were wound simultaneously as separate reels. The denier of the tapes was about 22,000 and the apparent density 0.252 g./cm.^{3}. When strands were removed from the tape and examined under the microscope, they were found to be composed of an interconnecting three-dimensional network of film-like fibrils. Essentially all of the fibril ends were found at the edges of the tape where cutting had occurred.

The tape was fed into a jet of the type shown in FIGURE 2 in which the dimensions of the rectangular slot at its narrowest point were 1 inch x 0.025 inch. The air pressure in the supply pipe to the jet was 75 p.s.i.g. The tape speed into the jet was 12 ft./min. and the speed at which the yarn was removed was also 12 ft./min.

The fluffy tow which issued from the jet had a roughly square cross section, being about 1.7 cm. wide and 1.7 cm. thick. The density of the fluffy tow was about 0.009 g./cm.^{3}.

The fluffy tow passed directly into a cigarette-making machine wherein the fluffy tow was gathered together and compacted lightly as it passed through a round lightly tapered funnel. The tow passed then into a wrapping device which placed a paper wrapper around the condensed tow and glued it in place. The resulting wrapped continuous filter rod was approximately 0.74 cm. in diameter. It passed through a cutter then which prepared 3.0 cm. lengths of the filter. These were finally cut in half by another cutter to form filters of 1.5 cm. length ready for use in cigarettes. This filter element contained 0.030 g. of plexifilamentary material. The density of the filter element exclusive of the wrapper was therefore about 0.042 g./cm.^{3}.

EXAMPLE II

Linear polyethylene was flash spun as in Example I and collected as a sheet material which was lightly compacted as before by a roll pressure of 10 lbs./in.

The sheet product was then embossed by passing between a pair of rolls one of which was covered with burlap. The average force exerted in this second rolling operation was 20 lbs./in. of width. A white paperboard-like material was obtained having transparent spots wherever the pressure was greatest in the calendering operation. It had a texture very similar to burlap with tiny transparent spots derived from the points where warp and fulling yarns crossed in the fabric used for embossing the sheet. This embossed sheet still had much of the soft feel of the sheet in Example I. After embossing the sheet had a thickness of .0446 cm. (17.6 mil). The sheet was cut into tape 2.25 cm. wide. This tape had a denier of about 24,000 and a density of 0.265 g./cm.^{3}.

The embossed tape was bulked by the same process as Example I and a fluffy tow was obtained which was 1.3 cm. thick and 2.0 cm. wide. The density of the two was 0.10 g./cm.^{3}.

The bulky tow passed directly into a cigarette-making machine as in Example I which was used to form the filter elements in the proper shape for use in cigarettes. The filter elements had a diameter of 0.74 cm. and a finished length of 1.5 cm. The density of the filter exclusive of the wrapper was 0.047 g./cm.^{3}.

EXAMPLE III

The filter elements prepared in Examples I and II were tested for pressure drop at flow rates of 1050 cm.^{3}/min. in cigarettes. The filter elements were also tested independently without tobacco. The two polyethylene filters were compared with a filter made of standard cellulose acetate round fibers with a denier per filament of 6. Table I summarizes the data for this experiment. The Cambridge test for measuring tar passed by the filter is described by W. B. Wartman, Jr., et al., Analytical Chem. 31, No. 10, 1705–1709 (1959).

TABLE I

Type Filter	Filter Length, mm.	Filter Wt., gm.	Pressure Drop, Inches of water		Passed Tar, mg.
			Filter Only	Whole Cigarette	
Unembossed Plexifilament	15	29.5	3.0	5.0	20.5
Embossed Plexifilament	15	33.6	3.0	5.1	24.9
Acetate round Filament	13	91.0	3.3	4.9	27.1

The polyethylene plexifilamentary filters, although being much lighter in weight than the acetate were found to be almost equivalent in pressure drop. On the other hand they were found to be much superior to the acetate in their ability to absorb tars. In summary it may be concluded that even when using only one-third as much material in the filter, more efficient filtration was obtained with the plexifilamentary tow and there was no sacrifice of the desirable aesthetic values associated with low pressure drop.

It is believed that the plexifilament tape of this invention will have wide significance in the field of cigarette filters, enabling filters to be constructed having efficient filtration. One of the advantages of the invention is that filtration efficiency may be controlled at will by controlling the weight and bulk of the material used in the filter. The tape-like materials are unusual in that they can be bulked to very high specific volumes without the formation of large channels or holes in the filter structure; consequently, filtration efficiency is very high after the bulking operation and a pleasant more saleable cigarette filter is obtained. Another important advantage is the convenience of shipping which is obtained by using the compressed form of the tape and bulking the tape at the cigarette manufacturing location. The tape is free of loose ends and is therefore much superior to staple materials which tend to clog the jets during normal opening operation.

While the above examples show filters of very specific construction, it will be obvious to anyone skilled in the art that a wide variety of materials can be used and the construction can be varied greatly to obtain a range of filtration efficiencies and range of smoke tastes.

What is claimed is:

1. A process for making a tobacco smoke filtering material comprising flash spinning a crystalline synthetic polymer through an orifice to form a plexifilamentary strand, intercepting the strand as it issues from the orifice by an oscillating surface set at angle to the direction of travel of the strand in order to spread the strand into a web and direct the web downward toward a moving collection surface, collecting the web on the collection surface in overlapping, multi-directional, intersecting layers to form a sheet, calendering the sheet to reduce its thickness to below about 0.5 cm. at a basis weight of between about 0.25 oz./yd.^{2} and 10 oz./yd.^{2}, slitting the pressed sheet into tapes of between about 0.3 and 10 inches in width, feeding a tape into a jet, impinging a stream of gas against the plane of the tape and at an angle of between about 10° and 45° thereto increase the cross-sectional area of the said tape at least 5 times and reduce its density to between about 0.001 and 0.120 gm./cm.^{3} and collecting the bulked tape.

2. The process of claim 1 wherein the plexifilamentary material is a crystalline hydrocarbon.

3. The process of claim 2 wherein the plexifilamentary material is linear polyethylene.

4. The process of claim 1 wherein the sheet is calendered to reduce its thickness to below about 0.125 cm. at a basis weight of between about 1 oz./yd.^{2} and 3 oz./yd.^{2} and wherein the pressed sheet is slit into tapes of between about 0.5 and 2 inches in width.

(References on following page)

References Cited

UNITED STATES PATENTS

2,416,390	2/1947	Hitt	264—210 XR
2,794,239	6/1957	Crawford et al.	28—81
2,900,988	8/1959	Crawford et al.	
2,914,835	12/1959	Slayter et al.	28—72
2,953,838	9/1960	Crawford et al.	28—81
2,982,082	5/1961	Pool.	
2,997,771	8/1961	Martyn.	
3,019,507	2/1962	Maragliano et al.	28—72
3,025,197	3/1962	Sheidley	264—119
3,026,190	3/1962	McMahon et al.	264—204
3,055,080	9/1962	Claussen et al.	
3,079,929	3/1963	Mueller.	
3,081,519	3/1963	Blades et al.	28—81
3,099,594	7/1963	Caines et al.	
3,144,025	8/1964	Erlich.	
3,156,752	11/1964	Cope	264—280 XR
3,180,911	4/1965	Mueller.	

FOREIGN PATENTS

568,523	12/1958	Belgium.
568,524	12/1958	Belgium.
864,695	4/1961	Great Britain.

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