

Nov. 28, 1972

J. W. SOEHNGEN ET AL
PROCESS OF MAKING TOBACCO SMOKE FILTERS FROM
EXTRUDED POLYMER AND BINDER

3,704,192

Original Filed Sept. 21, 1966

2 Sheets-Sheet 1

Fig. 1.

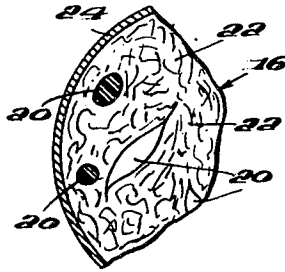
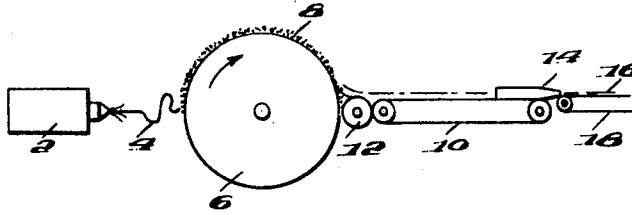


Fig. 2.

Fig. 3.

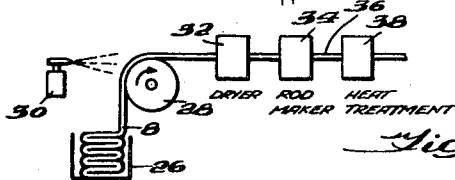
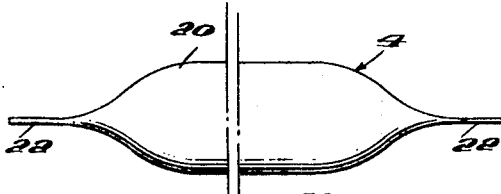


Fig. 4.

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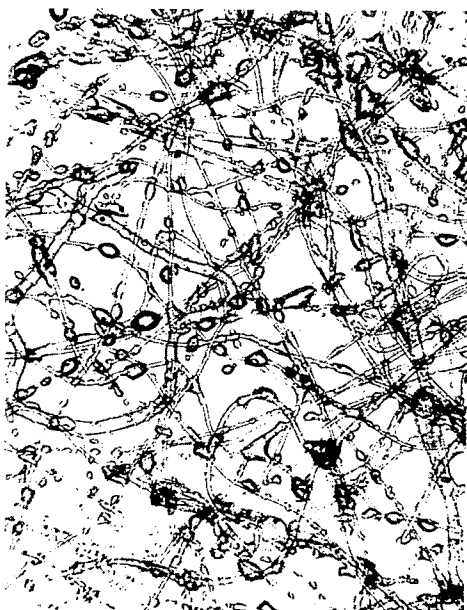


Fig. 5

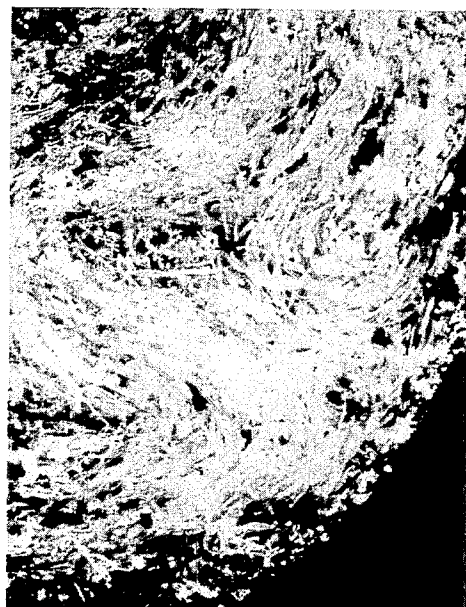


Fig. 6

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3,704,192

PROCESS OF MAKING TOBACCO SMOKE FILTERS FROM EXTRUDED POLYMER AND BINDER

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Original application Sept. 21, 1966, Ser. No. 580,993, now
Patent No. 3,444,863, dated May 20, 1969. Divided
and this application Feb. 6, 1969, Ser. No. 835,827

Int. Cl. D04h 3/16

U.S. Cl. 156—167

6 Claims

ABSTRACT OF THE DISCLOSURE

Cigarette filters are prepared from a random array of continuous spray-spun synthetic filamentary material, the filaments of said filamentary material varying in diameter with the periodicity characteristics of the filament being such that the change in diameter occurs over a relatively long distance along the filament length.

This application is a divisional application of Ser. No. 580,993, filed Sept. 21, 1966, now U.S. Pat. No. 3,444,863.

This invention relates to tobacco smoke filters and more particularly, to cigarette filters containing man-made filamentary material and to processes for making such filters.

Widespread use now is being made of continuous artificial filaments in the manufacture of cigarette filters. In a typical process of the type employed commercially for making such filters, many fine filaments extruded from spinnerette orifices are collected together in the form of a tow of indefinite length and the tow is transformed into filter rod without disrupting its lengthwise continuity. Crimping of the filamentary material usually is considered necessary in order to displace portions of the individual filaments at angles to the axis of the tow so that, in the filter, these displaced portions of the filaments will be disposed across the flow path of the tobacco smoke for intercepting the particles to be filtered from the smoke. In some of the more widely used processes, crimping is accomplished by passing the tow into a stuffer box apparatus, and the crimped tow is opened and manipulated to bring the crimped portions of adjacent filaments of the tow out of registry with each other. The opened tow of crimped continuous filaments is then consolidated laterally, as by passage through scarfing apparatus to give it the desired round or oval cross section, and a wrapper of sheet material such as paper is applied to complete the formation of the filter rod.

The bulk of the commercial production utilizing conventional processes has centered around the manufacture of filters from cellulose acetate filaments. Although proposals have been advanced for handling such filamentary materials as polyethylene and polypropylene, the use of these materials in commercial filters has not been very large.

Although such processes have prove highly useful, they have some objectionable characteristics. The crimping and crimp-deregistering operations are particularly critical to the production of high quality cigarette filters by these processes. Yet, such operations add materially to the cost of production, and unless they are carried out with care, products lacking in uniformity are apt to result. Also, the ability of certain of these conventional filters to remove selectively certain chemical components from a smoke stream is not as great as might be desired.

A primary object of this invention is to provide a new process for making filters in which a number of the steps

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of the conventional process are eliminated without detracting from the desired properties of the cigarette filters produced thereby.

Another object of the invention is to provide tobacco smoke filters which may be produced reliably and economically and in which substantially continuous filamentary material is oriented in a distinctive pattern enhancing the desirability of the products.

Another object of the invention is to provide tobacco smoke filters having improved firmness and selectivity characteristics, by treating the filamentary material thereof with additive compositions that not only serve to bond together the surfaces of contacting filament segments but also contribute a capacity to remove selectively from the smoke stream certain chemical components thereof.

These and other objects of the invention which will become apparent hereinafter are realized in accordance with a preferred embodiment of the invention by spray spinning from a melt substantially continuous filamentary material, collecting the freshly spun filament in the form of a band in which individual filament sections extend randomly in every direction, and laterally displacing portions of the band relative to each other to form an assembly of the cross sectional shapes desired for a cigarette filter. A binder or a plasticizer preferably is applied to the band of filamentary material prior to the shaping operation. When polyolefin material is used, it is especially desirable to add to the filamentary band a polymeric bonding agent that will serve not only to achieve the bonding necessary for filter firmness but also to improve the phenol selectivity of the filter.

The spray spinning preferably is carried out through the use of the techniques and apparatus disclosed in a patent application of Wagner et al. entitled "Methods and Apparatus for Forming Fibrous Structures," which application is owned by the assignee of this application and is being filed concurrently herewith. Molten fiber-forming material is extruded through the orifice of a nozzle as a filament. Attenuation of the incompletely hardened filament is effected by a plurality of high velocity gas streams issuing from gas passages spaced about the extrusion orifice and having axes that converge toward but do not intersect the extrusion orifice axis. The gas flow projects the filament away from the nozzle in a random swirling pattern.

The filamentary material projected from the spinning zone has a combination of characteristics which have been found to be of particular interest in connection with the making of cigarette filters. Although the fiber is like conventionally spun fiber in the sense that it is in the form of a substantially continuous filament, it is characterized by random lengthwise variations in diameter and degree of orientation which result from random variations in the attenuating action of the gas streams contacting the freshly spun filament. These variations in filament properties are of substantial magnitudes and their periodicity characteristics are such as to make them significant in cigarette filters. For example, in a preferred embodiment the cross section of the largest filament portion is more than ten times that of the smallest filament portion, with the mean being about two and one-half times the cross section of the smallest filament portion and with the periodicity of the variations being on the order of at least several inches. In a filter embodying such filamentary material, the small diameter segments present a larger proportion of surface area per unit of volume, while the large diameter segments are relatively stiff and resist crushing of the filter.

Surface effects also have been observed in examining the filament by electron microscopy techniques. The surfaces of the spray spun filaments examined were some-

what rough as compared with conventionally spun filaments.

As the filament is projected away from the spinning nozzle in a random pattern by the gas streams, a collection surface, such as the surface of a roll or a belt, is moved continuously across the path of the projected filamentary material to collect the filamentary material without destroying the random orientation of the filament sections. When the swirling filament intersects the collector surface, it traverses both across and lengthwise of the collector surface, as well as in all intermediate directions. The movement of the collector surface serves to bring new portions of the surface into the path of the filamentary material continuously, so that the collected fiber forms a band or strip that is continuously removed lengthwise from the collection zone. The extrusion rate and the speed of the collector surface are so related to each other as to assure that the collected band will have the desired weight per unit length for use in tobacco smoke filters.

Collection of the filament preferably takes place while the surface portions of the filament are still sufficiently tacky to effect some self-bonding at crossover points between segments of the filament. In practicing the invention, it is necessary that the collected band have tensile properties which will permit handling of the band during subsequent processing without destroying the random orientation of the filament sections within the band. Such properties conveniently are provided by permitting self-bonding to occur concurrently with the filament collection operation.

After collection, the band is consolidated laterally to give it the round or oval cross section customary for cigarette filters. Upon consolidation, the band becomes a three dimensional structure in which the directions of the traverses of the substantially continuous filament are distributed in a random fashion as regards the three coordinates. This arrangement has been found to be a particularly desirable one from the standpoint of achieving in the filter a suitable balance between such properties as filtration efficiency, resistance to gas flow, and crush resistance.

Although the physical arrangement of the filamentary material in the collected band need not be altered prior to consolidation of the band into filter rods, it is frequently desirable to apply additives to the band. For example, filters of improved firmness characteristics may be produced by applying a suitable plasticizer or binder to the band before the band is passed to the rod making apparatus. Also, it may be desirable to add to the band particulate material such as activated carbon and/or compositions which are chemically active to improve filtration performance. These treatments may be carried out without disturbing the desired filament orientation in the band through use of suitable spraying techniques.

In certain of its aspects the invention is especially suited to the production of filters from polyolefin filaments. As conventionally spun, polyethylene and polypropylene filaments are characterized by a highly oriented crystalline structure. These filaments are not readily susceptible to the conventional plasticizers used in making cigarette filters and they exhibit little capacity to selectively remove from a smoke stream certain undesired chemical components contained therein.

It is a feature of this invention that polyolefin fiber assemblies be treated with a bonding agent having a combination of particularly desirable properties. The preferred bonding agent is a polymeric material suitable for application to the fiber assemblies in emulsion form prior to passage of the assemblies into the rod making machine. After the filter rod is formed, the structure is heated to a temperature sufficiently high to cause solvent action between the molten bonding polymer and the surface layers of the polyolefin fiber but not to melt the fiber. The deposits of bonding agent will thus serve to

bond contacting fiber surfaces together. The bonding agent also has incorporated therein components contributing to phenol selectivity and/or toluene selectivity in the filtering action achieved.

The invention is further illustrated in the accompanying drawings, in which:

FIG. 1 is a schematic view illustrative of a process in accordance with this invention;

FIG. 2 is highly diagrammatic in nature and is intended merely to suggest certain functional properties of a transverse cross section through a cigarette filter formed in accordance with this invention;

FIG. 3 is a diagrammatic view which, while not intended as an illustration of structure, suggests the diameter differences which exist as between segments of a filament used in the filter of this invention;

FIG. 4 is a schematic view illustrative of the treatment of a band of filamentary material to add a binder thereto prior to scarfing;

FIG. 5 is a photograph (magnification 40X) showing the randomly arranged filament segments in a band of filamentary material collected in accordance with the invention from a spray spinning zone and having a binder applied thereto; and

FIG. 6 is a photograph (magnification 25) depicting the arrangement of the filamentary material after scarfing; the view being a partial end view of a filter rod without a wrapper or covering on the filamentary material.

The filters of this invention may be made from various fiber-forming materials that may be melted and extruded through an orifice as a filament. Examples of suitable types of fiber-forming materials are polyolefins, polyamides, polyesters, polyacetals and cellulose acetate. Polyolefins such as polyethylene and polypropylene, and polyamides such as nylon 66, have sufficiently low melt temperatures and sufficiently low melt viscosities, and they are therefore particularly convenient to use. Materials which are normally difficult to melt without decomposition, such as cellulose acetate, may be used by adding a high boiling plasticizer as a melt depressant.

Spray spinning nozzle means are indicated schematically at 2 in FIG. 1. Preferred nozzle structures are disclosed in the aforesaid patent application of Wagner et al., and for detailed information on these structures reference should be made to such application. It will be sufficient to point out here that the preferred spray spinning nozzle is provided with an orifice through which the molten fiber-forming material is extruded and is also provided with a plurality of gas passages disposed about the extrusion orifice. The cross sectional shape of the extrusion orifice is determined by the filament shape desired. The usual round or oval filaments are presently preferred, but other shapes may be employed. For example, trilobal filaments may be produced by extruding the material through Y-shaped orifices. The gas passages are inclined to direct heated gas, such as steam, along generally converging paths the axes of which do not intersect the projected axis of the extrusion orifice. The high velocity gas streams issuing from the gas passages attenuate the filament 4 extruded through the extrusion orifice and project the filament away from the nozzle in a random swirling pattern.

Collector means disposed in front of the spray spinning apparatus 2 receives the freshly-spun filamentary material 4. In FIG. 1 the collector has been illustrated as a rotating drum 6 provided with a smooth surface which moves across the path of the filamentary material. It should be understood, however, that other forms of collectors may be employed in practicing the invention. For example, the drum can be replaced by an endless belt guided through a path intersecting the spray pattern from the nozzle 2. It also should be understood that the collector surface need not be smooth. For example, a foraminous collector surface is desirable in some instances in order to decrease the likelihood of displacement of the deposited filament segments due to gas movement. Sim-

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ilarly, it may be desirable in some instances to electrostatically charge the drum or other collection surface.

As the drum 6 is rotated, there is formed a fibrous band 8 of continuous length and of a width corresponding to the diameter at the collection zone of the spray pattern from the nozzle means 2. Band widths in the range from about 4 to about 8 inches normally are preferred.

Each increment of the filament 4 is deflected from the central axis of the extrusion orifice of the nozzle means 2 with continually changing magnitude and direction as it is being projected and attenuated by the gas jets. Consequently, in the collected band 8, the substantially continuous filament makes many traverses both across and lengthwise of the band, as well as at all intermediate orientations. This arrangement is shown in the photograph designated FIG. 5 of the drawing.

In practicing the invention, the random arrangement of the filament sections within the band 8 is preserved throughout the subsequent steps of the process. Since handling of the band ordinarily imposes upon it tensile loads tending to orient the filament segments more nearly parallel to the length of the band, it is desirable that some bonding together of crossing filament segments be achieved at or near the collection zone. In most instances this effect can be produced merely by disposing the collector 6 close enough to the nozzle means 2 to collect the filament 4 while portions of its surface are still somewhat tacky. When the filament 4 is collected prior to complete hardening thereof, self-bonding occurs at crossover points between contacting filament segments. Of course, in instances where self-bonding is impractical or undesirable, a suitable binder material may be sprayed onto the collector along with the filamentary material.

The weight of the band 8 may be between 20,000 denier and 100,000 denier, and preferably lies within the range of from about 40,000 to about 60,000 denier, for optimum cigarette smoke filter characteristics. Control over band weight may be achieved by controlling the extrusion rate at the nozzle 2 and/or controlling the rate of rotation of the collector drum 6.

Referring again to FIG. 1, it will be observed that the band 8 is transferred from the drum 6 to an endless belt 10 by means of a roll 12 which cooperates with the drum 6. The band 8 moves into a scarfing trumpet 14 of a conventional rod making machine. Paper tape is fed to the rod making machine to wrap the filamentary material in the customary manner. The resulting filter rod 16 is carried on a belt 18 and is subsequently cut into suitable lengths for association with cigarette tobacco components.

As the band is drawn past the converging and inclined surfaces of the trumpet 14, portions thereof are displaced laterally relative to each other. This rolling or bunching action yields a three-dimensional structure in which the traverses of the filament segments are distributed in substantially random fashion as regards the three coordinates. In this connection, attention is invited to the photograph designated FIG. 6.

FIG. 3 suggests another significant characteristic of the filamentary material of the cigarette filters of this invention. The filament does not have a uniform diameter. Some sections 20 are much thicker than other sections 22. For example, the smallest sections may have diameters in the range of from about 10 microns to about 20 microns and the largest sections may have diameters in the range from about 50 microns to about 60 microns, depending on spraying conditions. There are in addition filament sections of various intermediate diameters. Moreover, the mean diameter of all the filament sections is closer to the minimum than to the maximum, demonstrating a predominance of fiber sections of smaller diameters.

In suggesting the magnitude of the diameter variations along the length of the filament, FIG. 3 grossly distorts the periodicity characteristic of these variations. As will be seen from the photograph designated FIG. 5, the diameter transitions are not abrupt but are gradual in nature, oc-

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curing over relatively long distances along the fiber lengths. Under a microscope, thick and thin portions of an individual filament section are not apparent. The appearance is rather that of an array of variously sized fibers.

FIG. 2 depicts a filter rod 16 having a paper wrapping 24 enclosing the filamentary material. The showing here of the filamentary material is again highly diagrammatic, and the view is intended merely as a stylized representation emphasizing certain functionally significant distinctions between the filamentary material of the filters of this invention and the filamentary material conventionally employed in cigarette filters. Instead of relying upon crimping for providing filament sections disposed across the flow path of the smoke, segments of the filament making up the filter of this invention are disposed in a random array. Portions of the filament extend lengthwise of the filter, crosswise of the filter, and in every other direction. Also, the filaments used in conventional filters do not have the marked diameter variations of the filament used in this invention. The thick sections 20 lend to structural strength of the filter rod, and resist crushing of the filter unit during assembly and use. The thin sections 22 present a large surface area for mechanically trapping the smoke particles.

The discontinuities in the filamentary material indicated in FIG. 2 should not be construed as discontinuities in the filament itself, but rather as suggestive of departures of the filament from the plane of view. The varying lengths of the filament sections shown in FIG. 2 are intended as indicative of the existence of variations in the lengths of the traverses of the filament in any given direction or plane.

The nature and arrangement of the filamentary material in the filters of this invention make it possible to achieve a satisfactory balance as between the properties normally sought in cigarette filters. Filter rods having the desired circumference and having the firmness characteristics to which smokers are accustomed may be formed without packing in so much material as to make the pressure drop across the filter medium unacceptably high. Furthermore, the filter rods are formed from the filamentary material as deposited directly upon the collector that receives the freshly spun filament. No intermediate mechanical treatment of the filament, such as crimping, is required before forming the band of filamentary material into filter rods. Therefore, this process is very efficient.

Although it will be apparent that various modifications of the process described above are possible, brief references to certain of the more interesting variations will serve to further illustrate the invention.

The drawings show an embodiment in which the band 8 of collected filamentary material is relatively narrow and is suitable for conversion into a single filter rod. However, this is not essential. If desired, a relatively wide band of filamentary material may be collected, as by traversing the collector laterally back and forth in front of the spray spinning nozzle, and the wide band may be slit longitudinally into a plurality of strips each having a width and a weight suitable for the production of an individual filter rod. In this instance, each of the strips is passed to the scarfing trumpet of a conventional rod-making machine in the manner described in connection with the band 8 of FIG. 1.

Additionally, the paper wrapper 24 about the filamentary material is not required in all instances. Most cigarette filters produced at the present time do have paper wrappers about the filamentary material, but proposals have been advanced for eliminating such wrappers. If application of the paper is considered undesirable in some circumstances, it need not be applied to the filamentary material of this invention. The shaped assembly of filamentary material passing out of the scarfing trumpet or other rod-making device, may instead be treated to provide the assembly with a hard outer core in the form of a sheath of coalesced filament segments, or the shaped assembly of filamentary material may be treated in the

manner described in Dunlap U.S. Pat. 3,190,294, granted June 22, 1965.

Moreover, it frequently will be found desirable to combine solid or liquid additives with the filamentary material of the filter. Such additives enhance the desirability of the filter products for some purposes. One may, for example, wish to add one or more compositions for improving the selectivity (e.g., phenol selectivity) of the filtration action, and/or a particulate filtering material such as activated carbon, and/or a flavorant such as menthol.

Various techniques are available for bringing the additives into the desired association with the filamentary material. An additive that is not incompatible with the fiber-forming material or the spray spinning environmental conditions may be incorporated into the molten spinning composition. A wider variety of additives may be accommodated by procedures for applying the additives to the filamentary material after spinning. A spray of additive material may be brought into contact with the filamentary material in the zone where the filamentary material is collected initially into a band, or the additive spray may be applied to the band after its formation.

Binders or plasticizers ordinarily are sprayed onto the filamentary material in instances where these contribute to the structural properties of the filter. For example, included among the various plasticizer compositions suitable for use on cellulose acetate fiber are the conventional cellulose ester plasticizers, such as acetyl triethyl citrate, methylphthalyl ethyl glycolate, tributyl phthalate, tripropionin, triacetin, etc.

Bonding agents of particular value with respect to linear and isotactic polyolefin fiber of relatively high melting point are polymeric materials in which at least part of the structure is closely analogous to the low molecular weight solvents for the polyolefin. These agents may be applied to the filamentary material prior to formation of the fiber rod without adversely affecting the mechanical processing, and then activated by heat after the rod has been formed. The softening temperature of the bonding material is conveniently higher than that at which the deposit is dried on the polyolefin fiber surfaces so as to avoid any interference with consolidation of the filamentary material into the desired rod structure. However, the softening temperature must be sufficiently lower than that of the polyolefin fiber to allow the bonding to take place without any significant loss in the porosity of the structure. Thus, the softening temperature of a bonding material to be used with polyethylene filament fiber should be within the range of from about 60° C. to about 125° C. and that of one to be used with polypropylene should be within the range of from about 60° C. to about 150° C. Known copolymerization techniques are available for use where necessary in improving materials having melting points in the ranges indicated.

A preferred type of binder is one which not only accomplishes the bonding function but also enhances the filtration action of the filter. To this end the bonding material may include compositions containing functional groups which contribute to selectivity for the removal of phenol or other undesirable components of the smoke. For example, suitable polymeric materials which enhance phenol selectivity may be prepared by polymerization of a polymerizable unsaturated organic ester such as vinyl acetate or an acrylate ester, or a vinyl ether.

FIG. 4 illustrates schematically a processing sequence for treating with a binder a band of filamentary material collected as described in accordance with FIG. 1. The band 8 is formed in the same manner as the band of filamentary material in FIG. 1, but FIG. 4 shows this band as coming from a receptacle 26. It is, of course, optional whether the band from the collector drum 6 be processed into filter rods continuously or with interruptions requiring temporary storage of the band.

The band is withdrawn from the receptacle 26 by a

rotating roll 28 and a sprayer 30 is positioned opposite the roll 28 for applying a binder to the filamentary material of the band. As shown in FIG. 4, the binder is applied to only one side of the band 8, but it may be applied to both sides if penetration of the spray into the band from one side is inadequate. Of course, the binder may be applied by any other suitable means, such as by passing the band through a bath containing the binder, or by applying the binder as a powder and thus activating the binder by heat. The amount of binder supplied should ordinarily be such that, after drying in air to remove volatile constituents, the binder deposit constitutes from about twenty to about twenty-five percent of the total weight of the treated band, although as low as five percent of binder may be used.

The treatments which follow application of the binder depend somewhat upon the composition and form of the binder. One preferred binder is an emulsion of polymeric material of the type described above as being particularly advantageous for use with polyolefin fiber, and FIG. 4 depicts the treatment that would be used in connection therewith.

After the emulsion has been applied, the band 8 passes through a dryer 32 where the volatile constituents of the polymer emulsion are evaporated. The temperature in the dryer should be below that at which the binder exerts a solvent action on the polyolefin fiber, e.g. below about 90° C. The band passes through a conventional rod maker 34 which forms the band into a filter rod 36. The filter rod 36 passes through a chamber 38 where it is heated to a temperature below the melting point of the fiber but high enough to soften or melt the bonding polymer and cause solvent action between the binder and surface portions of the fiber. After heating, the filter rod 38 is cooled to provide a stabilized porous structure in which contacting fiber surfaces are bonded together. The stabilized filter rod then is cut into suitable lengths and assembled with tobacco to form cigarettes.

The following examples will serve to further illustrate the invention:

EXAMPLE I

Undried polyethylene was heated to a molten condition in an extruder and the material was extruded through a nozzle having a circular orifice of 0.028 inch diameter at a temperature of about 365° C. The nozzle included three 0.082 inch ports spaced at 120° intervals about the extrusion orifice, directing steam along paths the axes of which converged toward but did not intersect the extended axis of the extrusion orifice. The axes of the steam ports were so inclined as to provide an opening of about 1/16 inch diameter therebetween at the point of closest convergence, which point was located about one inch downstream from the outlet of the extrusion orifice. The steam was supplied at a temperature of about 410° C. and at a pressure of about 25 p.s.i.g.

The high velocity steam attenuated the extrudate and whipped it about randomly with little or no filament breakage. The diameter of the filament varied from about 15 microns to about 55 microns, with the mean diameter being about 25 microns.

The filamentary material was collected from the spinning zone on the surface of a rotating drum spaced approximately 16 inches from the nozzle. The surface speed of the drum was about one meter per minute. The material was collected as a band about 5 inches wide and having a denier of about 50,000. Traverses of the filament extended in every direction within the band.

The band was consolidated laterally into a cylindrical filter rod by passing it through the scarfing trumpet of a rod making machine, and a paper wrapper was applied thereto in a conventional manner. The filter rod was then severed to provide cigarette filter tips of 20 mm. length. An average pressure drop of 55 mm. of water was

observed when air was passed through these filter tips at a rate of 15 cc. per second.

EXAMPLE II

Polypropylene filament was spray spun under the conditions and using the apparatus described in Example I. The filamentary material was collected on the surface of the rotating drum in the form of a continuous band or web about 6 inches wide and 1/8 inch thick at the center thereof. The band weighed about 4.7 grams per meter. This is equivalent in linear density to a 42,000 denier continuous filament tow. The band was sprayed with a polyvinylacetate homopolymer emulsion (available commercially as CL-122, a product of Celanese Corporation), diluted with water to a solids content of 25%. The emulsion was sprayed with a conventional paint sprayer. Sufficient spray was applied to the band to deposit about 20-25% of polyvinylacetate in the band, as measured after the band was dried in air to remove the volatile constituents in the spray emulsion. The dried web was wrapped with cigarette paper in a conventional rod making machine, and the rods were cut into lengths of 12 cm. Then the rods were placed in a forced convection oven at 125° C. for 20 minutes and subsequently cooled to room temperature. The filter tips were cut from the rods in lengths of 20 mm. and were tested in comparison with a commercial filter tip of the same length made from cellulose acetate fibers. The averages of the results were as follows:

	Spray spun filter	Commercial filter, cellulose acetate
Pressure drop, mm. H ₂ O	62	65
Fiber weight, g.	0.094	0.135
Total filter media weight, g.	0.124	0.135
Firmness	82	82
Smoke removal efficiency	54	51
Phenol selectivity	2.8	2.8

Firmness was determined as the percentage of the original filter diameter retained under the application of a one-pound load.

Smoke removal efficiency is determined by drawing 35 cc. puffs of 2 seconds duration from a lit cigarette consisting of a 65 mm. tobacco column attached to a 20 mm. filter at a frequency of 1 per minute and then through a glass microfiber trap (Cambridge filter media No. CM-113, made by Cambridge Filter Corporation of Syracuse, N.Y.) which retains 99.9% of all smoke particles greater than 0.3 micron in diameter. The cigarettes are smoked to 30 mm. overall length. The percent efficiency is then calculated from the following formula:

Percent efficiency

$$= \frac{\text{increase in weight of filter}}{\text{increase in weight of filter} + \text{trap}} \times 100$$

Phenol selectivity is measured by employing a test smoking machine of the type described in Tobacco Science 2, 73 (1958) and a gas chromatographic analysis. Phenol selectivity is determined by smoking therein a cigarette consisting of a 65 mm. tobacco column (0.92±0.3 gm.) attached to a 20 mm. filter, the cigarette being conditioned at 74±2° F. and 60% relative humidity. In the smoking machine 35 cc. puffs of 2 seconds duration are drawn every 58 seconds and the smoking is continued long enough to reduce the overall length of the cigarette to 30 mm. A smoke condensate solution is prepared from the filters (glass micro filters carrying an acrylic binder) by submerging the filter pads in a 2 N caustic solution, adding internal standards (o-chlorophenol), removing all traces

of condensate from the pads by washing with further caustic, pressing the pads, filtering through a fritted glass filter under suction, and extracting the filtrate with successive portions of ether. Subsequent processing includes acidifying the resulting aqueous solution at 0° C. with sulfuric acid; repeatedly extracting the phenols by treating the acidified solution with ether, washing the ether extracts with saturated sodium bicarbonate; evaporating the ether solution to a 5 ml. volume; and analyzing with a gas chromatograph equipped with a flame ionization detector. The ratio of the peak area of phenol to that of o-chlorophenol affords a measure of the phenol in smoke. The amount of phenol in the smoke condensate is calculated by the following equation:

Micrograms phenol/cigarette

weight phenol/weight internal standard

$$= \frac{\text{X micrograms of internal standard added}}{\text{number of cigarettes smoked to produce the sample}}$$

In a similar manner, an 85 mm. non-filter reference cigarette (same tobacco column as in filtered cigarette test) is smoked and the condensation collected on Cambridge glass fiber filter pads. Phenol in the condensate is determined as described above.

The amount of phenol, in micrograms, per milligram of condensate for the reference all-tobacco cigarette relative to the amount of phenol per milligram of condensate for the filter cigarette yields the phenol selectivity number, S for the filter tip.

$$S = \frac{\left[\frac{(\text{Micrograms phenol per cigarette})}{(\text{Milligrams condensate per cigarette})} \right] \text{All tobacco reference cigarette}}{\left[\frac{(\text{Micrograms condensate per cigarette})}{(\text{Milligrams condensate per cigarette})} \right] \text{Filter cigarette}}$$

EXAMPLE III

The procedure of Example I was employed to produce a spray spun polyethylene band. A portion of the collected band of filamentary material was sprayed with a styrene-butadiene copolymer emulsion (Darex 511 L, a high styrene latex of W. R. Grace & Co.), and dried in air at room temperature. This portion of the band contained 20-25% by weight of the styrene-butadiene copolymer. It was consolidated into filter rod, covered with cigarette filter paper and cut into 20 mm. lengths. Some of these filters were then exposed to heat treatment at a temperature of 116° C. for 17 minutes. Other portions of the spray spun band were processed into filters without the addition of the copolymer emulsion, and some of these filters were heat treated at 116° C. for 17 minutes. A comparison was made between the spray spun polyethylene with and without the emulsion and the heat treatment.

Treatment:

Firmness

Original spray-spun web	71
Spray-spun web heated at 116° C.	73
Web sprayed with styrene-butadiene latex	72
Web sprayed with styrene-butadiene latex and heat treated at 116° C.	83

The filter tips containing the styrene-butadiene copolymer also showed an ability to remove toluene selectively from cigarette smoke.

EXAMPLE IV

The procedure of Example II was repeated using a low molecular weight polyethylene emulsion for the binder spray (Spencer Chemical Poly-Em 41 Nonionic). The spray spun filament was formed of secondary cellulose acetate of acetyl value 55, in which 16% dimethoxyethyl phthalate had been incorporated by hot-roll blending of the polymer prior to spray spinning. The filter rods were heated at 125° C. for ten minutes. The resulting filter rods produced filters having a firmness of

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77% as compared with 56% for similarly formed filters without the heat treated additive.

EXAMPLE V

A spray spun band of nylon 66 continuous filament having an average diameter of about 3 d.p.f. and exhibiting a pebbled surface under electron microscopy, was formed into a conventional cigarette filter rod having the following characteristics:

Average pressure drop	mm	50
Phenol selectivity		1.8
Average weight	gm	.207
Phenol delivery	μg	.28

A filter prepared from 3 d.p.f. conventional nylon staple exhibited a phenol selectivity value of 1.1.

These examples demonstrate that filters produced in accordance with this invention directly from spray spun filament bands, without mechanical crimping treatments, compared favorably with conventionally formed filters. The random arrangement of the filamentary material in the filters of this invention gives the filters suitably high smoke removal efficiencies without requiring that the filters be so densely packed with fiber as to raise the pressure drop to objectionable levels. The additional treatments described herein may be employed to improve the firmness of the spray spun filter tips and their selectivity for specific smoke components. Accordingly, improved filter tips may be produced economically and efficiently.

The condensed bands of filamentary material of this invention may find utility as air or smoke filters in analogous environments.

While this invention has been illustrated and described in certain preferred embodiments, it is recognized that variations and changes may be made therein without departing from the invention as set forth in the claims.

What is claimed is:

1. A process for making an intermediate product suitable for processing into tobacco smoke filters comprising extruding a molten polymer through an orifice of a nozzle as a filament, attenuating said filament with a plu-

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rality of high velocity gas streams, collecting the filament as a random web on a moving collector surface while said filament is sufficiently tacky to effect some self-bonding at crossover points between said segments of said filament, the conditions employed in said process being such as to produce a randomly oriented filamentary web having a denier of about 20,000 and 100,000, and treating said web with from 5 to about 25% based on the total width of the web of a binder having a softening temperature within the range of about 60° C. to about 150° C.

2. The process of claim 1 wherein said polymer is a polyolefin polymer.

3. The process of claim 1 wherein said web at collection has a width of from about 4 to about 8 inches.

4. The process of claim 1 wherein said binder comprises a polymer of an unsaturated organic ester.

5. The process of claim 1 wherein said binder is selected from the group consisting of vinyl acetate, styrene and copolymers of styrene and butadiene.

6. The process of claim 1 wherein said web is subsequently condensed into a tobacco filter rod.

References Cited

UNITED STATES PATENTS

2,916,038	12/1959	Wade	131—10
3,148,101	9/1964	Allman, Jr. et al.	156—167
3,016,945	1/1962	Wexler	156—166
3,039,908	6/1962	Parmelee	156—166
3,227,791	1/1966	Kiefer et al.	156—167
3,501,361	3/1970	Touey et al.	156—167

FOREIGN PATENTS

1,103,093	2/1968	Great Britain	156—166
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