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[54] **CIGARETTE SMOKE FILTER MATERIAL**
4 Claims, 3 Drawing Figs.

[52] **U.S. Cl.** **131/266,** 57/140, 161/180
[51] **Int. Cl.** **A24f 07/04**
[50] **Field of Search** 161/170, 173, 174, 175, 177, 179, 180; 131/261, 266, 267, 268, 269, 10

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ABSTRACT: A cigarette filter plug is disclosed which is made from filaments characterized by alternating regions of large and small diameter prepared by dry spinning a solution of cellulose ester containing a plasticizer and solid solvent adsorbent particles and then passing the resultant filaments over a hot surface. The solid particles found suitable may be activated charcoal, other types of activated carbon, silica gel, alumina or clay.



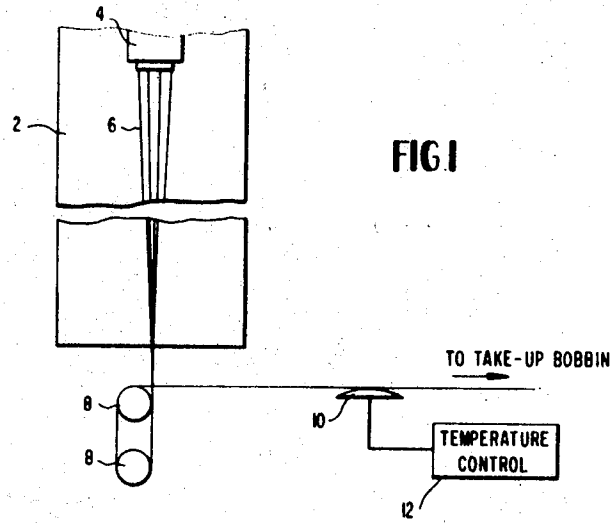
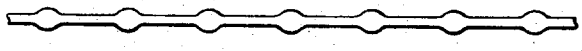


FIG. 1

FIG. 2



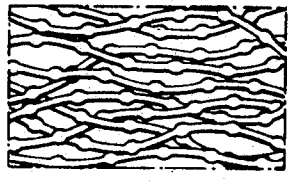


FIG. 3

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CIGARETTE SMOKE FILTER MATERIAL

This application is a divisional application of Ser. No. 495,895, filed Oct. 14, 1965, now U.S. Pat. No. 3,422,176.

This invention relates to a filter material. More particularly, the invention relates to a collection, tow or bundle of thermoplastic filaments suitable for use as a tobacco smoke filter rod.

Among the wide variety of materials which have been recommended as tobacco smoke filters, one type in particular is ideally suited for this purpose from a manufacturing standpoint. This type is a continuous crimped tow material, preferably a continuous crimped cellulose acetate textile tow. One reason for the widespread use of cellulose acetate is the ease and rapidity at which it can be processed into firm or rigid filter rods on equipment known to the cigarette industry. Thus, for example, a continuous crimped cellulose acetate tow of 5,000 to 50,000 filaments, depending on the thickness of the spun fibers, can be bloomed out, sprayed with a non-volatile liquid plasticizer type of bonding agent, recompacted and pulled through a rod-forming and paper-wrapping device to produce 90 mm. filter rods at the rate of several hundred per minute. Such rods become quite firm on standing for a period of time.

Although continuous crimped textile tow is a useful filter material from a processing standpoint as explained above, it has a certain disadvantage from the standpoint of its ability to remove the tiny nicotine and tar particles from cigarette smoke. This aspect is the somewhat parallel relationship and surface smoothness of many of the filaments or fibers which constitute the filtering element. As a result, a certain amount of the nicotine and tar particles are capable of passing between the substantially longitudinally aligned smooth fibers without touching them. These particles, therefore, are not trapped. One method which has been suggested for circumventing this difficulty is to use a very fine denier fiber in the preparation of the tow. This would mean that it would require a considerably larger number of fibers in the tow to fill the filter to the desired circumference of a cigarette, thus giving a higher surface area and a much smaller space between the parallel fibers. Such an approach, however, gives a filter tip which may have a higher pressure drop (high resistance to the passage of air through the filter).

Another method which has been suggested for producing a textile tow type of filter, which is highly effective for the removal of nicotine and tar particles and yet does not produce an excessively high pressure drop, is to dust the tow with certain harmless powders such as starch powders or powders of cellulose or cellulose derivatives at some point along the assembly which processes it into a filter. The dusting produces an irregular surface on the substantially parallel fibers. The particles of the powdered additive function as bumps or protrusions which extend into the spaces between the fibers in the finished filter rod. These protrusions make the path of the smoke particles through the filter more difficult and part of the nicotine and tar particles which might channel through the filter are impinged on these protrusions.

Still another method which has been suggested for overcoming the above problem is to incorporate crystallizable types of compounds into the fiber-forming polymeric substance, which is spun into fibers in the conventional manner. When these fibers are formed, the crystallizable compounds gradually exude to the surface of the fibers whereupon they crystallize forming rough irregular protrusions on the fiber surface.

While the addition of protrusions on the fiber surface either by dusting or by crystallization improves filtration of the tobacco smoke, there are still certain disadvantages associated with the production of these tows. For example, the dusting technique requires the use of additional dusting equipment, forces the use of special precautions to prevent dust explosions and also presents problems in restricting the dust to the dusting area. The crystallization technique usually requires the addition of expensive crystallizable materials, and also generally necessitates an additional curing time for the solids to exude to the surface of the fiber and crystallize.

Accordingly, the primary object of the present invention is to provide a filter having integral protruberances on the fibers thereof.

Another object is to provide a filter plug which is highly effective for removing aerosol particles from tobacco smoke without incurring the disadvantages mentioned above.

An additional object is to provide a new fiber, for use in a filter plug, having alternating regions of large and small diameters without having to dust or crystallize particles on the surface of the fiber.

The fibers of the present invention are formed by a a usual, employing a dope comprising a solution spinnable thermoplastic filamentary material dissolved in a suitable organic solvent, solvent absorbent solid particles and a plasticizer is spun into filaments in a conventional spinning device. After the spun filaments or fibers are formed, and while they still contain a substantial amount of solvent, the fibers are passed over a heated stationary or moving surface, such as a plate or roller, to evaporate substantially all of the solvent from the fibers. Prior to passage over the heated surface the spun fibers are of a usual, substantially uniform diameter. However, after passage over the heated surface and evaporation of the solvent, the filaments have spaced, alternating regions of large and small diameters. In fact, the fibers appear to have beads positioned along the length thereof. The required number of "beaded" fibers are combined into a tow, which may be subsequently used as an aerosol filter element, and particularly a tobacco smoke filter plug.

It is important to note that the above fluctuating regions on the filament surface are not caused by bonding or crystallization of extraneous particles on the surface of the filaments. On the contrary, the beads appear to be swollen cavities within portions of the fiber material itself with the solid particles being uniformly dispersed throughout the filament. By producing the homogeneous beaded fiber there is no need to worry about the beads sifting out of the cigarette filter as sometimes happens when extraneously added particles are bonded or crystallized on the fiber surface.

While it is not certain, it is believed that the beaded effect is caused by the discharge of the absorbed solvent as a fiber passes over the heated surface. For this reason, it is important that the fibers be passed over the hot surface, sometimes hereinafter referred to as a hot shoe, while they still contain a substantial amount of solvent. Further, it is important that the solids present in the dope be capable of absorbing the organic solvent. Hence, the solids are referred to as solvent adsorbent solid particles. In addition, the solids must not be soluble in the materials contained in the spinning solution so that they can retain their adsorptive characteristic.

The fibers may be formed of any solution spinnable thermoplastic filamentary material. The term "solution spinnable" is meant to include the various polymeric substances which are soluble in organic solvents, e.g., cellulose derivatives such as cellulose acetate and triacetate, polymers of acrylonitrile, vinyl chloride, vinyl esters and ethers, and the like. The preferred fiber forming materials are cellulose esters of organic acids, and particularly cellulose acetate, the acetyl value of which can range from about 50 to 62.5 percent by weight calculated as acetic acid.

The thermoplastic filamentary material and the plasticizer are usually present in the spinning solution or dope in a combined concentration ranging from about 10 to 35 percent by weight of the spinning solution, and preferably from about 15 to 30 percent.

The presence of a plasticizer in the spinning solution is very important. Passage of the fiber over the hot surface causes intermittent expansion of the fiber. Because of the presence of the plasticizer, the swollen fiber areas or beads do not burst. The plasticizer may be any of the ones normally used with the aforesaid thermoplastic materials in the conventional filament spinning processes. With cellulose acetate the preferred

plasticizers include swelling agents such as glycerol triacetate (triacetin), triethyl citrate, dimethoxy-ethyl phthalate, dimethyl phthalate, methyl phthalyl ethyl glycolate, o-phenyl phenyl-(bis) phenyl phosphate, and the like. The preferred plasticizers when using cellulose acetate as the thermoplastic filamentary material are dimethyl phthalate and triacetin. The plasticizer may be added to the spinning solution in a concentration ranging from about 15 to 25 percent by weight of the thermoplastic filamentary material present therein, and preferably from about 18 to 22 percent, i.e., in an amount sufficient to prevent rupture or bursting of the fiber surface on sudden thermal discharge of the adsorbed solvent.

Any of the volatile organic solvents normally used with the above-mentioned filamentary materials during the spinning process are suitable for use. For example, solvents such as acetone, methylene chloride, dioxane, dimethyl formamide, methanol and the like, either alone or in combination, may be used. When the filamentary material is secondary cellulose acetate the preferred solvent is acetone or a mixture comprising a major proportion, e.g., 70 to 95 percent methylene chloride and a minor proportion, e.g., 5 to 30 percent methanol. The latter mixture is also preferred when cellulose triacetate is the filamentary material. The solvent is normally present in a concentration ranging from about 65 to 90 percent by weight of the spinning solution, and preferably from about 70 to 85 percent. Desirably, the solvent should comprise at least one component which is rather strongly adsorbed by the solids present in the spinning solution under the conditions of filament formation.

As previously indicated, the primary purpose of the nonsoluble, adsorbent solids is to extend the presence of a substantial amount of the solvent in the spun fibers until they pass over the hot shoe when the volatile solvent relatively suddenly evaporates upon contact of the fiber with the shoe.

The particle size of the nonsoluble, adsorbent solids should be small enough so as not to interfere significantly with the continuity of the supporting filamentary material or fiber structure being formed. Preferably, the diameter of the individual solid particles must be such that they comprise from about 10 to 20 percent of the diameter of the eventual filament in which they become occluded. Normally, the solids range in diameter from about 0.01 to 5 microns, and preferably from about 0.05 to 1 micron. Suitable solids which are nonsoluble in the spinning solution materials and which may be used in the present invention are charcoal, preferably activated charcoal, or other types of activated carbon, silica gel, alumina, and clay.

When cellulose acetate is the filamentary material and acetone or a mixture of methylene chloride and methanol is the solvent a preferred nonsoluble solid is activated charcoal. The nonsoluble solids may be present in the spinning solution in an amount ranging from about 1.5 to 3 percent by weight of the spinning solution, and preferably from about 2 to 2.5 percent. Generally, the solids comprise from about 5 to 10 percent by volume of the eventual fiber in which they are occluded. Because of the need to produce a continuous fiber structure, the amount and the diameter of the solids, within the above mentioned ranges, will be related to the fiber area or fineness of the product spun fiber, i.e., the capacity of the solid particles to disrupt the fiber structure is inversely related to the fiber area.

As previously mentioned, it is important to realize that the solids do not gravitate or flow to the surface or any particular region of the spun fibers, but are present uniformly throughout the fiber material. The spun fiber is an essentially uniform mixture of solids, filamentary material and plasticizer.

For a better and more complete understanding of the present invention, its objects and advantages, reference should be had to the following detailed description and to the accompanying drawings in which:

FIG. 1 is a schematic elevational view showing an exemplary spinning system suitable for use in making the filter fiber material of the present invention;

FIG. 2 is a sketch of an individual beaded filament produced by the disclosed process; and

FIG. 3 is a highly magnified longitudinal view of a filter plug or element comprising a collection of the beaded filaments produced by the said process.

Referring to FIG. 1 there is shown schematically a spinning cabinet 2 with a spinneret 4 located at its upper end and a multiplicity of filaments 6 moving downwardly from the spinneret 4.

A spinning solution comprising the aforementioned components, e.g., cellulose acetate, a solvent mixture of 90 percent methylene chloride and 10 percent methanol, activated charcoal particles and dimethyl phthalate plasticizer, is introduced by means not shown into the spinneret 4. The solution is spun under conventional conditions for solution spinning, for example, a temperature in the range of from about 30 to 50° C., and an extrusion rate in the range of from about 25 to 150 meters per minute, and preferably 40 to 60 meters per minute.

The spun filaments 6 pass downwardly through the spinning cabinet 2 at the same rate at which they were extruded. The temperature in the spinning cabinet may be in the range of from about 20 to 100° C., and preferably 30 to 50° C.

The spun filaments are removed from the cabinet 2 and while still containing solvent in the range of from about 10 to 50 percent by weight of the filaments, are passed over a pair of conventional skew rolls 8. The spun filaments are then passed over a hot shoe 10, which has been heated to a temperature above the evaporation temperature of the solvent and below the charring temperature of the filamentary material. Normally the hot shoe 10 is maintained at a temperature in the range of from about 175 to 205° C., and preferably 180 to 190° C. The hot surface or hot shoe 10 may be heated in any convenient manner, e.g., an electrically heated shoe may be used. As illustrated in FIG. 1, the hot shoe 10 is connected to a conventional temperature control box 12. The heated surface is preferably of cylindrical shape, however, a flat surface may also be used. The hot shoe may be stationary or moving, such as a heated rotating roller.

As previously mentioned, it is important that the filaments be contacted with the hot shoe while still containing a substantial amount of solvent. This condition is facilitated by proper control of the spinning cabinet temperatures within the above mentioned ranges and by the speed of withdrawal of the filaments from the cabinet. The speed of travel of the filaments is normally controlled by the pair of skew rolls 8 and a conventional take up bobbin, not shown on the drawing. The optimum cabinet temperature and filament withdrawal speed will also depend on the type of filamentary material, type and amount of solids and type and amount of solvent. However, a few simple preliminary runs with the desired spinning solution will quickly determine the best conditions to achieve the desired beaded filaments.

Returning to the hot shoe 10, the solvent-laden filaments are passed over the shoe so as to cause substantially all of the solvent to evaporate therefrom. Generally, contacting the filaments with the hot shoe for a period of time in the range of from about one-fiftieth to one-twentieth of a second will achieve the desired results.

As a result of contact with the hot shoe, the filaments recovered therefrom consist of alternating regions of large and small diameters, the individual filaments having a weight within the range of from about 3 to 10 denier. More specifically, the filaments have the appearance of a fiber containing beads more or less uniformly spaced thereon as illustrated in FIG. 2 of the drawings.

The filaments prior to contact with the heated surface may have any shape conventionally found in spun fibers. Normally, the spun fibers have substantially bulbous cross sections, such as result from dry spinning through circular orifices.

The filaments are passed from the hot shoe 10 without any stretch of the filaments occurring and are collected on a take up bobbin in the conventional manner, The desired amount of

beaded filaments are collected and subsequently processed in conventional filter plug making equipment to produce the product filter plugs, as illustrated by FIG. 3 of the drawings.

The beaded filamentary material of this invention may be used for filtration of any aerosol particles, however, the preferred use is as tobacco smoke filter plugs. In addition, the beaded fibers may be used in textiles due to optical and tactual features of the fiber structure or to its insulating or adsorptive properties.

The following example is given by way of illustrating the process of the present invention:

EXAMPLE

A slurry of the following composition was agitated for 5 days on a ball mill:

fifteen parts by weight of activated charcoal (Darko KB), having a surface area of 1589 square meters per gram and a pore volume of 2,077 milliliters per gram;

two parts by weight of secondary cellulose acetate flake;

one hundred thirty-one parts by weight of methylene chloride;

two parts by weight of methanol.

A mixture was prepared of 118 grams of the above slurry, plus 111 grams of secondary cellulose acetate flake, 6.8 grams methanol, 296 grams methylene chloride, and 24 grams dimethyl phthalate. This mixture was agitated by tumbling overnight and then extruded through a stainless steel spinneret of five holes, 0.042 mm. in diameter. The filaments were conveyed while still solvent-laden at fifty meters per minute over a hot shoe at a temperature of 182 to 185° C., from which they were taken up without stretch. The filaments obtained were 6 denier per filament and had essentially the shape illustrated in FIG. 2 of the drawings.

Firm hand-rolled filter plugs were made of these fibers and required only 0.115 grams of fiber per each 17 millimeter plug, which is no more than is required to form firm commercial plugs or tips that are presently in use. The filter plug is illustrated by FIG. 3 of the drawings.

The principle, preferred construction, and mode of operation of the fibers of the invention have been explained and what is now considered to be its best embodiment has been described in the foregoing specification. However, it should be understood that the invention which is intended to be protected herein may be practiced otherwise than as specifically illustrated and described without departing from the scope of the appended claims.

I claim:

1. A tobacco smoke filter plug comprising a collection of filaments of a denier per filament of about 3 to 10, having spaced alternating regions of large and small diameters, said large diameters being from about 0.05 to 50 microns, said filaments comprising an essentially uniform mixture of a solution spinnable thermoplastic filamentary material, solvent adsorbent solid particles selected from the group consisting of particles of activated charcoal, activated carbon, silica gel, alumina and clay and a plasticizer.

2. The filaments of claim 1 in which the thermoplastic filamentary material comprises a cellulose ester of an organic acid.

3. The filaments of claim 1 in which the thermoplastic filamentary material comprises cellulose acetate.

4. The filaments of claim 1 in which the adsorbent solid particles comprise activated charcoal.