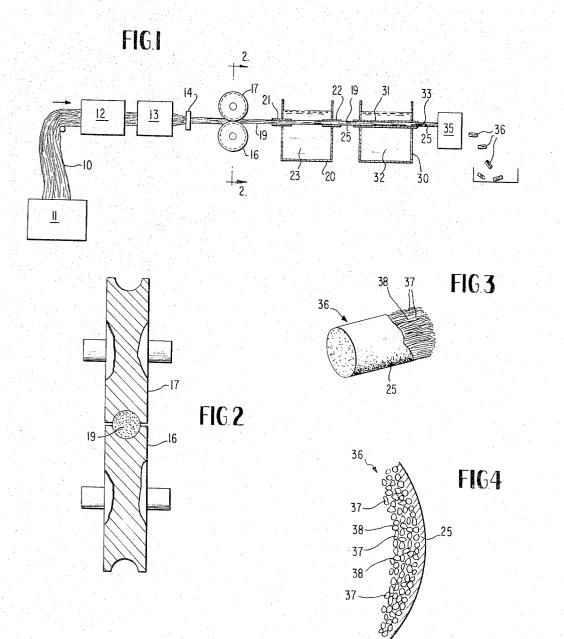
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G. A. WATSON PAPERLESS CIGARETTE FILTER Filed Sept. 27, 1965



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3,361,137 PAPERLESS CIGARETTE FILTER George A. Watson, Charlotte, N.C., assignor to Celanese Corporation of America, New York, N.Y., a corporation of Delaware Filed Sept. 27, 1965, Ser. No. 490,509

3 Claims. (Cl. 131-10.9)

This invention relates to novel, paperless, shape-retaining cigarette filters and to a process for forming them. 10 More particularly, it relates to the formation of plasticcoated cigarette filter plugs from thermoplastic filamentary material.

Thermoplastic fibers or filaments, notably cellulose acetate, have gained wide acceptance as filter elements for 15 tobacco smoke, being incorporated in the form of a plug or mouthpiece in cigarettes. To make such filter tips or plugs it is common to form them from a tow or bundle of several thousand continuous filaments which are spread flat, usually sprayed with a plasticizer such as glycerol 20 triacetate, formed into a continuous rod of predetermined shape by passage through a correspondingly shaped aperture in a device referred to as a "condenser." The rod is then fed into a conventional cigarette wrapping machine together with cigarette paper to be wrapped therein. After 25 cutting the paper-wrapped filter rod into plugs of the desired length the plugs are usually allowed to cure to effect stiffening of the filter through bonding of the filaments at their point of contact and the paper-wrapped plugs are incorporated into cigarettes in a well-known manner by 30 being wrapped in cigarette paper with tobacco.

By proceeding as outlined, valuable time is consumed in the initial wrapping of the tow in paper and, where the paper is secured by gluing, in the application of glue to seal the paper and in the drying of the glue. In addition, 35 the paper and glue used in such prewrapping of the filter plug add significantly to the cost of the filter without contributing anything of value to its effectiveness as a filter.

In other filter manufacturing processes a bundle of pre- 40 ties to the cigarettes in which such are included. plasticized filaments is heated while being passed through a shaping member, sufficient heat being applied to form an impervious sheet by fusion of the surface filaments. Such heat serves to retain the shape without a paper wrapping but the process requires the maintenance of relative-45 ly high temperatures and relatively slow production speeds in order to assure proper fusion and the filaments employed in forming the sheet lose their filamentary character and lose their efficacy as filter elements. The filtration efficiency of such superficially fused filters is thus lower than the efficiency of paper wrapped filters containing the same number of filaments. Moreover, unless such surface fusion is most carefully controlled, substantial fusion of filaments throughout the entire body of the rod tends to take place, thereby resulting in a further loss in filtration efficiency. In aggravated cases, such extensive obstruction of the normal smoke passages can result that smoking of a cigarette provided with such a filter becomes undesirably difficult.

It is an object of the present invention to provide a novel filter rod or plug which requires neither a paper wrap nor fusion of the filaments. Another object is to provide a process for forming cigarette filter plugs at high production rates which does not require processing tem-peratures above about 150° C. Still another object is to provide plastic-coated filter rods which are easy to cut into predetermined lengths and which comprise a coherent superficial plastic skin that can be easily applied to the tow from which the rods are formed. These and other objects, as well as the nature, scope and mode of operation of the invention, will become apparent from the description which follows. In the absence of indications to

the contrary it will be understood that all percentages and proportions of materials are expressed herein on a weight basis.

In accordance with this invention novel paperless filter plugs and filter rods are produced by a novel process which permits high production speeds and results neither in any substantial fusion or bonding of the filaments constituting the bundle nor in any substantial blockage of the passages therebetween. More particularly, filter rods produced in accordance with the present invention comprise a tow or bundle of crimped thermoplastic filaments which are sheathed or held together in the desired shape in a resinous thermoplastic film or skin. Still more particularly, in a preferred embodiment of the present invention a continuous tow of crimped, limp cellulose acetate filaments, which may be plasticized with a plasticizer such as glycerol triacetate in an otherwise conventional manner, is passed through a bath of a molten mixture or solution of wax and an ethylene-vinyl acetate copolymer to apply a liquid coating to the tow, and the applied liquid coating is rapidly solidified on the tow by cooling, e.g., by passage of the freshly coated tow or rod through an externally cooled tube having an internal diameter or crosssection corresponding to the desired shape of the filter rod.

It has particularly been discovered that by proper formulation of the resinous mixture, the present invention permits the production of resin-coated filter plugs by applying the required coating in the liquid state at moderate processing temperatures without substantial penetration of the liquid coating medium into the interior portions of the tow, the resulting plastic skin being substantially nonsticky at temperatures to which finished cigarettes are normally exposed (e.g., 0° to 40° C.) and being easy to cut in customary cigarette making equipment as well as physiologically approved. In contrast, other plastic coatings tend to cause process difficulties in their application to the tow or in the subsequent mechanical handling of the resulting plugs, or they tend to impart undesirable proper-

For instance, wax alone tends to produce coatings on filter rods that penetrate deeply into the rods and make such rods difficult to cut. Also, such coatings tend to soften objectionably when a cigarette of which such a plug is a part is being smoked. Other resinous compositions available for melt application either require unduly high bath temperatures, or result in undue penetration and hence in undue impairment of eventual filter action, or they result in a tacky coating which interferes with the high 50 speed operation customarily used to incorporate filter plugs in cigarettes, or they represent a health hazard.

The resinous sheath composition which has been found advantageous in the practice of the present invention comprises a mixture which is solid at temperatures up to 55 about 40° C. or higher and which contains more than 50 and up to 75% petroleum wax, preferably about 60 to 75% petroleum wax, and correspondingly about 25 to less than 50%, or about 25 to 40% of a thermoplastic ethylenevinyl acetate copolymer.

The petroleum wax can be either microcrystalline or 60 paraffin wax. For instance, it can be either a microcrystalline or an amorphous wax having a melting point from about 50° to about 85° C., or a mixture of several waves. The wax may contain up to about 0.5% mineral oil by weight without detriment to the final coating properties 65 of the wax-copolymer composition used in this invention, Commercially available paraffin wax having a melting point between about 50° and 55° C., of the kind commonly used to seal jars in home canning, and having FDA approval, is particularly suitable. 70

As is well known, such a wax can be prepared from a slack wax from the dewaxing of lubricating oil or from

topping a high wax content crude oil, fractionally distilling under reduced pressure, and dissolving an appropriate distillate fraction in a solvent such as a mixture of methylethyl ketone and benzene, and recovering the wax which separates upon cooling of the solution. Similarly usable in the present invention are mixtures of waxes such as those described in United States Patents No. 3,013,709, 3,069,346, and 3,090,709.

The ethylene-vinyl acetate copolymer useful in the present invention is a thermoplastic solid containing about 10 50 to 85% combined ethylene and corresponding about 50 to 15% of combined vinyl acetate, preferably about 60 to 80% ethylene and about 40 to 20% vinyl acetate. Copolymers having an ethylene content of less than about 60% tend to be relatively non-resilient, particularly at 15 low temperatures. The copolymers are typically characterized by a melt index between about 2 to about 1000 or preferably between about 5 to 200, a tensile strength between about 400 and 3000 p.s.i., a density of about 0.94-0.955 g./cc., a softening point (Ring and Ball) of about 85 to 190° C. and an intrinsic viscosity at 30° C. 20 (0.25 weight percent in toluene) of about 0.7 to 0.95. Particularly suitable are commercial copolymers having a vinyl acetate content of between about 24 and 30, a melt index between about 5 and 30 (ASTM D-1238-57D), tensile strength of between about 700 and 3000 lbs./sq. in. and an elongation at break of about 700 to 1000%. For instance, a copolymer commercially available as "Elvax 250," having a melt index of from 12 to 18 and a vinyl acetate content of from about 27 to 29%, is convenient to use in a paraffin wax system. For use in a microwax system, a copolymer commercially available as "Elvax 350," having a melt index of from about 16 to 22 and a vinyl acetate content of from about 24 to 26%, is recommended.

As is well known, such copolymers can be prepared by copolymerizing a mixture of ethylene and vinyl acetate in the presence of a catalyst such as oxygen or an organic peroxide such as tertiary butyl hydroperoxide, in a tubular reactor at a pressure of from about 15,000 to about 40 30,000 p.s.i.g. and at a temperature from about 150° to 250° C. and then separating the resultant copolymer from the reaction mixture by flashing off unreacted monomers. By varying the conditions of pressure, temperature, catalyst concentration and vinyl acetate content in the 45 monomer mixture, one can obtain copolymers varying in melt index from about 0.5 to above about 1000. A different method for preparing suitable ethylene-vinyl acetate copolymers is described in United States Patent No. 2,200,429. The preparation of various blends of wax 50and ethylene-vinyl acetate copolymers is described in United States Patent No. 2,877,196.

The particular kind of resin-containing wax composition required in the present invention can be advantageously prepared by dissolving a minor proportion of the 55 copolymer, e.g., from about 25 to 49%, preferably about 30%, of the copolymer in molten paraffin wax while stirring. Solution can be accomplished, for instance, at temperatures between about 70° and about 175° C., the minimum practical temperature obviously depends somewhat on the particular wax and copolymer used.

The invention will now be described more fully with reference to the accompanying drawing wherein:

FIG. 1 is a schematic elevation of an apparatus for making cigarette plugs in accordance with the present $_{65}$ invention;

FIG. 2 is a section taken through the forming rolls along the line 2-2 of FIG. 1;

FIG. 3 is a perspective view of a coated plug produced by the apparatus of FIG. 1 with a portion of the coating 70stripped off; and

 \overline{FIG} . 4 is a transverse section on a greatly enlarged scale through a portion of the plug shown in FIG. 3.

Referring now more particularly to FIG. 1, there is shown a crimped cellulose acetate tow 10 composed of 75

from about 2,000 to 30,000, e.g., 8,000 to 20,000 filaments of about 1.5 to 25 denier per filament (total denier about 30,000 to 120,000) and about 6 to 15 crimps per inch of tensioned tow. For instance, the tow 10 may be composed of about 9,000 continuous filaments of 8 denier and about 9 crimps per inch. The tow 10 is removed from its shipping container 11 and is opened or spread flat by opener 12 of conventional construction and conveniently comprising means whereby the filaments in the tow are displaced longitudinally relative to each other so as to keep adjacent crimped filaments from being closely nested, the tow then passes continuously through box 13 into which a plasticizer such as glycerol triacetate (triacetin) may be sprayed as a mist, e.g., in an amount such that the cellulose acetate fibers pick up about 3 to 12%plasticizer by weight of fiber. However, such plasticizing, while often advantageous, is not an essential feature of the present invention. The plasticized tow next advances through a circular guide or condenser 14 of conventional construction where it is condensed into a predetermined transverse shape such as a circle, ellipse, etc. in a manner otherwise well known. Instead of, or in addition to, such circular guide the tow may also be passed at ambient temperature through other condensers such as one or more pairs of small opposed grooved rollers or wheels 16 and 17 having a peripheral groove as further shown in FIGURE 2.

The condensed tow 19 next is fed through the molten wax-resin bath 23 which is contained in tank 20. For instance, the tow can be passed through the tank below the surface of the bath via inlet and outlet tubes 21 and 22 of about 3/8" internal diameter, there being a gap between tubes 21 and 22 such that the tow becomes exposed to the molten wax composition as it passes from tube 21 to tube 22. By making one or both of the tubes 35 longitudinally movable the distance between them can be adjusted such that more or less tow is exposed to the molten solution. Thereby the amount of coating deposited on the tow can be controlled at the desired level, e.g., between about 1 and 10 ml., preferably between about 3 and 5 ml., while running at constant speed, e.g. at 25 linear feet per minute. The temperature of the resinous solution is maintained at or above its melting point, temperatures up to about 20° C. above the melting point of the mixture being convenient to use. Higher temperatures may be used but usually offer no advantage and tend to make subsequent solidification of the coating more difficult to achieve.

When the tow coated with the molten solution emerges from tube 22 the coating 25 is promptly solidified by cooling or refrigeration, e.g., by passage through tube 31 $(\frac{3}{5})$ or $\frac{1}{4}$ I.D.) which is externally cooled by means of an ice water bath 32 maintained in tank 30 or by other suitable cooling means.

As the tow emerges from tube 31 it is now in the form of a paperless, shape-retaining cigarette filter rod 33 which has a thin coherent resinous skin or coating 25 on the exterior surface thereof. This rod then passes to cutter 35 of a conventional cigarette making machine where it is severed into filter plugs 36. These plugs can be incor-60 porated in cigarettes in the same manner heretofore employed with paper wrapped plugs. Though the present invention does not require this, the rods or plugs can be cured in an otherwise conventional manner if it is desired to stiffen the inner filaments of the rod before using them to make cigarettes. A proper cure should cause the filaments to become lightly bonded to each other without substantial loss of their filamentary character. When a plasticizer such as triacetin is used, sufficient curing takes place on aging the plugs at room temperature. Curing can be accelerated by heat but in such a case caution should be exercised to keep the curing temperature below the melting point of the outer resinous coating as well as to avoid excessive fusion of the filaments themselves.

As shown in FIG. 4 on an enlarged scale, the plug 36

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is composed of a multiplicity of crimped, wavy filaments 37 extending longitudinally. Because of the crimps the filaments contact each other at a plurality of locations such as 38 while being held together by the exterior resinous film 25 without loss of their filamentary character. As particularly shown in FIG. 4 the resinous coating is applied essentially only as a surface skin 25, without any appreciable penetration into the internal portions of the filamentary filter and hence without any interference with the normal filtering action of the filaments which con-10 stitute the filter.

The non-penetrating character of the coating solution can be demonstrated, for instatuce, by incorporating a bright, oil soluble dye in the molten solution 23. When such dyed or brightly colored solution is applied to the 15 white cellulose acetate tow a brightly colored and sharply defined skin 25 is formed around the periphery of the tow without any noticeable penetration of the colored resinous material into the inner portions of the tow. If desired, colored resinous compositions can thus be used 20 which will produce colored filter plugs and add a pleasing decorative effect when incorporate in cigarettes.

It should be understood, of course, that while the method of application of the wax-copolymer coating by passing the compacted tow through a molten bath of coating com- 25 position has been found very effective, other methods of application can be used. For instance, the coating composition in liquid form may be sprayed onto the compacted tow or filter rod from a plurality of nozzles distributed about its circumference, and thereafter dried to 30 form the desired coating.

The tow can be formed of any thermoplastic filamentary material such as nylon, linear polyesters such as polyethylene terephthalate, polymers and copolymers of vinylidene compounds such as ethylene, propylene, acrylonitrile, vinyl chloride, vinyl acetate, and the like. However, the preferred materials are cellulose esters of organic carboxylic acids and particularly cellulose acetate having an acetyl value in the range from about 50 to about 62.5% by weight calculated as acetic acid.

As already stated, in making a filter for a cigarette of conventional size, the tow can range in total denier from about 30,000 to 120,000, preferably 35,000 to 80,000, and the individual filaments thereof can range from about 1.5 to 25 denier, preferably 1.5 to 15 denier. The tow em-45 ployed is preferably a crimped tow having from about 6 to 15 crimps per inch of tow length, the length being determined by placing a specimen of the tow under sufficient tension to remove the crimps therefrom.

The following example is given by way of illustrating a 50 preferred mode of the present invention.

EXAMPLE

A 3.2/54,000 tow, i.e., a tow of about 54,000 total denier and about 3.2 denier per filament, having about 10 55 crimps per inch of tensioned length of tow and composed of cellulose acetate of an acetyl value of about 55%, is opened and spread out to a width of about 13 inches. Glycerol triacetate is applied thereto by spraying, to the extent of about 5% by weight of the tow and the tow is thereafter condensed to form a filter-size rod of about 22 to 26 mm. in circumference, i.e. about 7 to 8.5 mm. in diameter, by being pulled through a circular guide or condenser of conventional construction.

The condensed tow is then pulled at a rate of about 25 feet per minute in sequence through tubes 21 and 22

inside a heated container 23 substantially as shown in FIG. 1 of the attached drawing. The tubes 21 and 22, each having an internal diameter of 3% inch and located below the surface of the molten solution, are spaced from each other such as to leave a gap of about 1/8 inch between the outlet end of tube 21 and the inlet end of tube 22, thereby causing the tow to become exposed to the molten solution with which container 23 is filled. The molten mixture or solution comprises about 30% ethyl-ene-vinyl acetate copolymer ("Elvax") and about 70% paraffin wax (domestic canning grade, M.P. about 65° C.) and is maintained in tank 23 at a temperature a few degrees above its melting point. From tank 23 the tow with its surface deposit of the solution passes through tube 31 which has an internal diameter of 1/4 inch and is externally cooled by ice water. This "freezes" the rod in its final diameter. No significant penetration of the coating into the internal portions of the filamentary rod is observed.

The resinous coating thus applied to the tow may have a weight of from about 2 to 25 grams per square meter, e.g., 12 g./m.2, or a thickness of from about 3 to 30 microns, e.g., 13 microns. The coated and cooled rod, which is now a shape-retaining rod of about 25 mm. in circumference is cut into plugs about 100 mm. or 4 inches long.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that variations and modifications thereof may be made without departing from the spirit of this invention. The invention which is to be secured by Letters Patent is particularly pointed out in the appended claims.

What is claimed is:

1. A shape-retaining paperless cigarette filter plug comprising a bundle of crimped thermoplastic filaments of total denier ranging from about 30,000 to 120,000 and of individual denier ranging from about 1.5 to 25, all of said filaments because of their crimp contacting other filaments at a plurality of spaced locations, said bundle of filaments being sheathed in a coherent solid coating com-40 posed of a major proportion of petroleum wax and a minor proportion of a resinous copolymer consisting essentially of from about 5 to 50% combined vinyl acetate and correspondingly from about 95 to 50% combined ethylene, said coating acting to retain the shape of said plug in the absence of a paper wrapper.

2. A paperless plug according to claim 1 wherein said filaments are composed of cellulose acetate and said coating comprises from about 60 to 75% paraffin wax and about 40 to 75% of an ethylene-vinyl acetate copolymer having a vinyl acetate content of between about 24 and 30 and a melt index between about 5 and 30.

3. A filter-tipped tobacco cigarette comprising a filter plug as defined in claim 1.

References Cited

UNITED STATES PATENTS

| 60 | 3,025,861 3,190,294 | 3/1962 6/1965 | 2 Cobb 5 Dunlap | | _ 131—267 _ 131—267 | |
|----|------------------------|------------------|--------------------|--|------------------------|--|
| | | FOREIGN PATENTS | | | | |
| | 693,929 | 9/1964 | Canada. | | | |

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