



- (51) International Patent Classification:  
D01D 5/24 (2006.01) D01D 5/253 (2006.01)
- (21) International Application Number:  
PCT/US2022/034824
- (22) International Filing Date:  
24 June 2022 (24.06.2022)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:  
63/202,792 24 June 2021 (24.06.2021) US
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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IQ, IR, IS, IT, JM, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(54) Title: HIGH POPULATION OF CLOSED C-SHAPED FIBERS

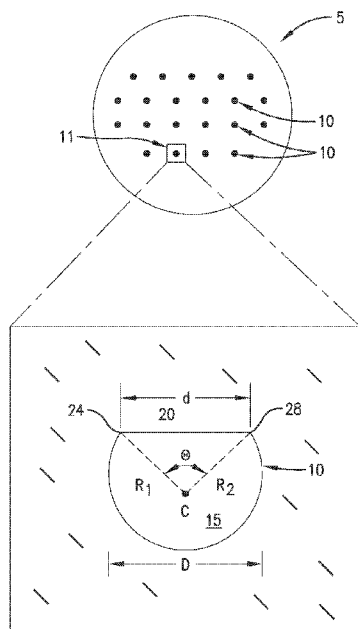


Fig. 1.

(57) Abstract: A high population density of closed C shaped cellulose acetate filament can now be reliably produced. The closed C filaments are made from a spinneret having a substantially D shaped orifice under particular processing conditions depending on the dpf of the filament, the theta angle of the D shaped orifice, and dope temperature. The process can make a high population count of closed C shaped fibers in a tow band, and article are not provided having the high population density of closed C shaped fibers.



**(84) Designated States** (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

**Published:**

— *with international search report (Art. 21(3))*

## HIGH POPULATION OF CLOSED C-SHAPED FIBERS

### FIELD OF THE INVENTION

5       **[0001]**   The present invention relates to a synthetic filament having a cross-sectional closed C shape geometry, a process for manufacturing the filament, and articles made with such filaments.

### BACKGROUND OF THE INVENTION

10       **[0002]**   Various processes and apparatus have been provided for the production of synthetic filaments of various cross-sections. Generally, these manufactured cross-sections include filament shapes of round, crenulated, Y, or multi-lobed configurations and other related variations produced by physically deforming the filament after it has assumed its normal shape as it  
15       issues from the spinning cabinet. Typical methods and apparatus for dry spinning solutions into synthetic filaments are disclosed in U.S. Patent Nos. 2,000,047 and 2,000,048, issued May 7, 1935, to H. G. Stone. These patents describe methods including forcing a heated cellulose ester spinning solution through a spinneret having a plurality of separated round orifices and into a  
20       drying chamber containing an evaporation atmosphere maintained at a suitable drying temperature. By such controlled conditions, round filaments can be consistently formed.

25       **[0003]**   The creation of a hollow filament is difficult to obtain due to the complexity in the spinneret design and the process conditions relative to the anticipated denier per filament of the fiber (“dpf”) produced. We have discovered that the method for producing a consistent hollow filament is more complex when using a dry-spun spinning process because of the solvent evacuation differential from the filament outer surface relative to the inner surface, the shape of the fiber exiting the spinneret, the draw ratio, and process  
30       condition variabilities relative to the dpf of the fiber.

5 [0004] In the area of filtration for aerosolized tobacco products, such as a cigarette or electronic cigarette or liquid vapor producing device, a tow band used for a tobacco filter segment or plug is required to have an appropriate ventilation resistance. In preparing the filter segment, it is important to balance the degree of filtration, filament density, and ventilation resistance so that the filter segment is not easily crushed and retains an ability to draw smoke through the filter. Measurements of filter segments performance depend on the particular application of the filter, but typically filter segments for cigarettes may range in length from 80 mm to 140 mm, and from 16 mm to 27 mm in circumference. For example, a typical filter segment having a 100 mm length and a 24.53 mm circumference may exhibit a pressure drop of from 200 mm to 400 mm of water as determined at an airflow rate of 17.5 cubic centimeters per second (cc/sec) as determined using an encapsulated pressure drop tester, sold commercially as Model No. FTS-300 by Essentra PLC, Milton Keys, UK.

10 [0005] One problem utilizing dry spinning techniques for making the aforementioned shaped filaments is that it is difficult to form yarns where the concave portions and convex portions are uniform in number due to the drying state of the solvent. Thus, the inclusion of filaments in a bundle, such as tow, which are non-uniform in cross-section and are considerably deviated from an intended form, happens with some degree of regularity.

15 [0006] Hollow cellulose acetate filaments could be very useful in the construction of nonwovens and woven products. In nonwovens, they could be used to construct filtration materials for aerosol filtration, liquid-liquid separation, solids-liquid separation, gas-liquid separation, and solids-gas separation. Non-limiting examples of products that could include at least a portion of the filaments includes surgical masks, face masks, surgical gowns and medical packaging. Round hollow filaments can provide a soft feel in a textile and provide more fluid holding and fluid transfer capacity in both nonwoven and woven materials compared to other cross sections. In the use as a cigarette or e-cigarette filter, the filament can provide a lighter weight product having a high surface area and a lower shape factor which may provide higher filtration efficiencies, lower pressure-drop across the filter, and better

filter performance than filaments having a different filament cross-section and similar media weight.

5 **[0007]** In U.S. Patent No. 1,773,969, the technique of the extrusion of filament forming solutions through circular orifices into evaporative atmospheres is disclosed. As described, it is suggested that the outer layer of the stream of cellulosic material, which is initially circular in cross-section as it issues through the spinneret orifices, hardens or solidifies thereby first forming a skin that is tougher and less fluid than the interior. After this initial hardening of the outer surface, the interior of the filament is precipitated or dried and thereby shrinks while the outer layer is further hardened. The outer shell of the filament being tougher and more defined in shape than the interior, the contraction of volume of the interior causes the outer film to collapse and to assume a very irregular cross-section, which is in the form of a figure of many indentations of varying sizes and shapes.

10 **[0008]** U.S. Patent No. 3,340,571 discloses a variety of spinneret extrusion orifices or openings. In one aspect, the '571 patent describes a spinneret having the shape of a circle segment the straight boundary wall of which (constituted by the respective diameter of the circle) is provided with an either centrally or eccentrically located protuberance extending toward the curved wall of the opening. The '571 patent discloses geometric shapes having cross-sections appearing as a "9" or "6", depending upon orientation and a pinched horseshoe or "U" shape where opposite ends are generally disposed next to each other and only very slightly out of contact, i.e., the arms of the U are substantially of equal length. Moreover, the filament cross-sections have relatively uniform wall thicknesses and are symmetrical relative to their longitudinal center lines. Large bundles or tows of filaments can be formed directly from the spun filaments or alternatively by combining several smaller bundles, such large bundles being especially useful for forming cigarette filters or forming staple fibers by cutting. However, we have found that the U shaped orifices do not consistently and reliably produce a large population of closed C shaped filaments.

5 [0009] U.S. Patent No. 5,707,737 issued to Mori et al. discloses a cellulose acetate filament having a non-circular cross-section and having from 1 to 4 cross-sectional axes of symmetry selected from cocoon-shaped, crisscross or X-shaped, Y-letter-shaped, C-letter-shaped, or I-letter-shaped. In forming a C-letter-shape, the '737 patent discloses solubilizing 5 to 40 parts by weight of a plasticizer such as polyethylene glycol (PEG) per 100 parts by weight of cellulose acetate in a solvent to make the dope for spinning. The filament was produced using spinneret having fan-shaped orifices. The filament shapes are predictable based on the orifice shape because the solvent can evolve from the interior of the filament without deforming the filament since the formation of a hard outer shell is reduced due the plasticizing effect of the plasticizer. There remains, however, a persistent problem to consistently make a high population of closed C shaped filaments using a spinning dope with minor or no amount of plasticizer.

10 [0010] EP3821733 to Daicel Corporation discloses a filament formed from a notched spinneret having a general fan-shaped configuration having a notch. The spinning hole has a peripheral shape corresponding to a circular contour and a line segment having two radii  $r_1$  and an arc  $M$  between the line segments. The circle has a center point  $O_1$ . The obtuse angle of the spinning hole is set to a value from  $180^\circ$  to  $270^\circ$ . However, we have found that such wedge-shaped spinneret holes do not reliably produce a high population of closed C shaped fibers. Also disclosed in Figures 7 and 8 of EP3821733 is a D shaped spinneret orifice from which a portion of the periphery is notched to form a circular smooth cusp indentation into the filament. This type of filament shape, however, is not configured to provide a hollow or closed C shaped filament, as discussed below.

25 [0011] Accordingly, there is a need for a filament bundle comprising multiple filaments which are relatively uniform and substantially the same in cross-sectional form having a closed C cross-sectional contour. It would be desirable to produce a bundle of filament that are relatively uniform wherein greater than 90 percent of the filaments per square centimeter ( $\text{cm}^2$ ) have substantially the same closed C cross-sectional configuration.

[0012] This is a further need for a process by which such closed C shaped cross section filaments can be manufactured from a spinneret having a more simplistic design and greater consistency in filament formation.

[0013] There is also a need for an improved method of consistently producing filaments having a closed C shaped cross section form over time on a commercial spinning line.

### SUMMARY OF THE INVENTION

[0014] There is now provided a process for making a closed C shaped filament having cross-sectional configuration having proximal first and second ends and a hollow core, comprising:

- a. providing a cellulose acetate dope comprising cellulose acetate and solvent;
- b. extruding the dope through at least one spinneret having at least one D-shaped orifice to form a wet filament; and
- c. drying the wet filament in an apparatus adapted for removing solvent from the wet filament to form a closed C shaped filament in which:
  - A. at least a portion of a first proximal end is either:
    - i. oriented toward at least a portion of the second proximal end; or
    - ii. contacting a portion of the second proximal end; and
  - B. the first and second proximal ends form either:
    - i. a channel defined by a gap between the first and second proximal ends having a transverse distance  $D_1$ , wherein the channel leads from an outer surface of the filament to the hollow core defined by an inner filament surface and having a diameter  $D_2$ , and wherein  $D_2/D_1 > 1$ , or

- ii. no channel or passageway resulting from at least a portion the first proximal end contacting at least a portion of the second proximal end.

**[0015]** There is also provided a process for making a closed C shaped filament comprising dry spinning a cellulose acetate dope through a spinneret having at least one D shaped orifice having a theta (" $\theta$ ") angle to make a closed C shaped filament having a dpf, wherein:

- a. the  $\theta$  angle is at least  $90^\circ$ , and
- b. the ratio of  $\theta$  angle to the dpf of the filament is less than 33:1, and
- c. the cellulose acetate dope temperature is not more than  $62^\circ\text{C}$ , provided that
  - i. if the ratio of  $\theta$ :dpf is at or lower than 19:1, the dope temperature is less than  $62^\circ\text{C}$ ,
  - ii. if the ratio of  $\theta$ :dpf is more than 19:1, the dope temperature is less than  $60^\circ\text{C}$ , and
  - iii. if the dpf of the filament is less than 6, the dope temperature is less than  $60^\circ\text{C}$ .

**[0016]** There is also provided process for making a cellulose acetate filament comprising continuously dry spinning dope to make a tow band comprising cellulose acetate filaments, wherein at least 50% of the filaments produced over an 8 hour period have a closed C shape.

**[0017]** There is further provided an article comprising cellulose acetate fibers, wherein at least 50% of the cellulose acetate fibers are closed C shaped based on:

- d. 150 cellulose acetate fibers, or
- e. 150 C shaped cellulose acetate fibers.

**[0018]** Examples of suitable articles include bundles, tow bands, yarns, textiles, fabrics, and filters.

**BRIEF DESCRIPTION OF THE DRAWINGS**

**[0019]** Figure 1 is a top view of a spinneret 5 having a plurality of orifices 10 with an enlarged view of a portion 11 of the spinneret 5 illustrating in greater detail the D-shape configuration of one of the orifices 10.

5 **[0020]** Figure 2 is a cross-sectional view of a cellulose acetate filament prepared by dry spinning a cellulose acetate solution through a spinneret having orifices of Figure 1.

**[0021]** Figure 2A is a cross-sectional view of a cellulose acetate filament illustrating metrics for determining a closed C shape.

10 **[0022]** Figure 2B is a cross-sectional view of a C shaped cellulose acetate filament that is not a closed C shaped filament.

**[0023]** Figure 2C is a cross-sectional view of a cellulose acetate filament illustrating a closed C configuration having a partial co-extensive orientation of the proximal ends.

15 **[0024]** Figure 2D is a cross-sectional view of a single opposed embodiment of a cellulose acetate filament illustrating a single opposed orientation of one of the proximal ends.

**[0025]** Figure 2E is a cross-sectional view of another embodiment of a cellulose acetate filament illustrating first and second planar surfaces, s1 and s2, where s2 exhibits a co-extensive orientation of proximal ends.

20 **[0026]** Figure 2F is a cross-sectional view of another embodiment of a cellulose acetate filament illustrating a co-extensive orientation of the proximal ends.

25 **[0027]** Figure 2G is a cross-sectional view of a comparative embodiment of a cellulose acetate filament illustrating the lack of any co-extensive orientation of the proximal ends.

**[0028]** Figure 2H is a cross-sectional view of comparative embodiment of a cellulose acetate filament illustrating the lack of any co-extensive orientation of the proximal ends.

30 **[0029]** Figure 2I is a cross-sectional view of another embodiment of a cellulose acetate filament illustrating the co-extensive orientation of the proximal ends.

**[0030]** Figure 3 is a photomicrograph of a closed C shape cross-section of the filaments produced in Example 1.

**[0031]** Figure 4 is a photomicrograph of a closed C shape cross-section of the filaments produced in Example 2.

5 **[0032]** Figure 5 is a photomicrograph of a cross-section of the filaments produced in Comparative Example 1.

**[0033]** Figure 6 is a photomicrograph of a cross-section of the filaments produced in Comparative Example 2.

10 **[0034]** Figure 7 is a photomicrograph of a closed C shape cross-section of the filaments produced in Example 3.

**[0035]** Figure 8 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 3.

15 **[0036]** Figure 9 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 4.

**[0037]** Figure 10 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 5.

20 **[0038]** Figure 11 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 6.

25 **[0039]** Figure 12 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 7.

**[0040]** Figure 13 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 8.

30 **[0041]** Figure 14 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 4.

**[0042]** Figure 15 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 9.

5 **[0043]** Figure 16 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 10.

**[0044]** Figure 17 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 11.

10 **[0045]** Figures 18 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 5.

**[0046]** Figures 19 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 12.

15 **[0047]** Figure 20 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 13.

**[0048]** Figure 21 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 14.

20 **[0049]** Figure 22 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 6.

25 **[0050]** Figure 23 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 7.

30 **[0051]** Figure 24 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 8.

[0052] Figure 25 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 9.

5 [0053] Figure 26 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 15.

[0054] Figure 27 is a graph of rod tow weight, in milligrams, versus pressure drop, in millimeters of water, for filter rods prepared from filaments having a closed C cross-section, a Y cross-section, and a R cross-section.

10 [0055] Figure 28 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 11.

[0056] Figure 29 shows an optical microscope image taken at a magnification of 500x of the cross-section of the closed C shape acetate filaments produced for Example 12.

[0057] Figure 30 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 18.

15 [0058] Figure 31 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example 19.

## DETAILED DESCRIPTION OF THE INVENTION

25 [0059] The following embodiments of the present invention will be described in greater detail with reference to the accompanying drawings. While the drawings illustrate varied embodiments of the current invention, such figures and description are by way of example only. There is no intent to limit the principles and scope of the invention to the particularly described embodiment but instead is to limited by the scope of the claims that follow.

30 [0060] As used herein, any relational term, such as “first”, “second”, “top” or “upper”, “bottom” or “lower”, and the like, is used for clarity and convenience in understanding the disclosure and accompanying drawings and does not

connote or depend on any specific preference, orientation, or order, except where the context clearly indicates otherwise.

**[0061]** As used herein, the terms “about” and “substantially” in reference to a given parameter, property, or condition means that the given parameter, property, or condition is met with a small degree of variance such as within acceptable measuring and/or manufacturing tolerances and generally includes a variability of up to 5% of the designated value. For example, a term of “about 1.0” would include a variable range of from 0.95 to 1.05.

**[0062]** All numbers or percentages relating to amounts of a substance within this description are given in weight percentages (wt. %), unless clearly defined to the contrary or otherwise clear from the context.

**[0063]** Any number in a sequence of numbers includes the adjective preceding or following the sequence. For example, at least 1, 2, 3 dpf includes at least 1 dpf, or at least 2 dpf, or at least 3 dpf.

**[0064]** As used herein, a closed C shaped filament means cellulose acetate filaments that have a closed C shape as further defined below.

**[0065]** The term “filaments”, as used herein, refers to thin flexible threadlike objects that are spun from a dope extruded through a spinneret. A “fiber” can be either a filament or a staple. While reference is made throughout to filaments, every mention of filaments in an article can also be replaced with and provides support for “fiber” or “staple” since staple fibers are cut from filaments and articles can contain either filaments or staple fibers, or both. Filaments extruded in a generally longitudinally aligned manner and ultimately form a filament yarn, or its resulting staple fiber, may be of any suitable size. For example, each filament (or staple) may have a linear denier per filament (weight in g of 9000 m filament length, dpf) of at least any of at least 0.5, 0.8 1, 1.5, 2, 2.5, 3, 4, or 5 dpf. In addition or in the alternative, the dpf is not more than any of: 200, 100, 75, 50, 35, 30, 25, 20, 15, 14, 13, 12, 11, 10, 9, 8, 7, 6, 5, 4.5, 4, 3.5 3, or 2.75 dpf. Examples of suitable dpf ranges include from 0.8 to 30, or from 1 to 25, or from 1.5 to 20, or from 1.5 to 15, or from 1 to less than 4, or from 4 to 30.

**[0066]** In one embodiment or in combination with any mentioned embodiments, the fibers, including filaments and staple fibers, are desirably monofilaments.

5 **[0067]** In one embodiment or in combination with any mentioned embodiments, the fibers comprise at least 80 wt.%, or at least 90 wt.%, or at least 95 wt%, or at least 97 wt.%, or 100 wt.% cellulose acetate polymer based on the weight of all polymers in the fiber, excluding plasticizers. In one embodiment, the fibers are not bicomponent fibers and are not the result of processing bicomponent fibers.

10 **[0068]** The size of the individual filaments is not particularly limiting. The size can be given in terms of effective diameter, and in one aspect, the effective diameter of the filaments and staple fibers can range, for example, from 0.1  $\mu\text{m}$  to 1000  $\mu\text{m}$ , 1  $\mu\text{m}$  to 500  $\mu\text{m}$ , 1  $\mu\text{m}$  to 100  $\mu\text{m}$ , 1  $\mu\text{m}$  to 30  $\mu\text{m}$ , 10  $\mu\text{m}$  to 1000  $\mu\text{m}$ , 10  $\mu\text{m}$  to 500  $\mu\text{m}$ , 10  $\mu\text{m}$  to 100  $\mu\text{m}$ , or 10  $\mu\text{m}$  to 30  $\mu\text{m}$ .

15 **[0069]** In one embodiment or in combination with any mentioned embodiments, the cellulose ester filaments have a substantially C-shape cross-sectional configuration. Referring to Figure 1, the filament is prepared using a dry-spinning process involving extruding a cellulose ester solution through a spinneret 5 having a plurality of spinning holes or orifices 10. Each spinning  
20 orifice 10 has a "D" shape as shown in a magnified orifice 11 in Figure 1. By contrast, wedge shaped orifices, while in a shape that resembles an orifice that could produce a closed C shaped filament, do not produce a high population density or population of closed C shaped fibers, nor does it allow for a reliable process that consistently makes a closed C shaped filament.

25 **[0070]** Referring to Figure 1, the D-shape orifice 10 includes a substantially circular annular open portion 15. The annular opening portion 15 is defined the space within the inner circumference of the circular annular portion 15 and the chord 20. A chord 20 intersects the annular open portion 15 at two points, a first point 24 and a second point 28. The length of the cord 20, identified as "d", is  
30 less than the diameter "D" of the orifice 15. The cord length "d" is defined by the proximity of the cord 20 to the orifice 15 imaginary center "C". Center "C" is the center of an imaginary circle drawn along the circular annular portion 15

and continuing until a complete circle is drawn. The imaginary center C is a fixed imaginary point regardless of the angle  $\theta$ . For ease of discussion, the cord length "d" defines an angle  $\theta$ , which is determined by two radii,  $R_1$  and  $R_2$  of equal length extending from the center C and intersecting the first point 24 and the second point 28, respectively. The chord 20 is a structural element of the spinneret 10 that defines the shape of the annular open portion or orifice 15 through which the spinning dope flows. The closer the cord 20 is to the center C, i.e., the longer the cord 20, the greater the theta angle  $\theta$  which results in a more pronounced D-shape opening 15.

**[0071]** Accordingly, the length of the chord 20 may be described by specifying the  $\theta$  angle. In accordance with the present invention, it has surprisingly been discovered that the  $\theta$  angle should be greater than 90 degrees. In one embodiment or in combination of any mentioned embodiments, the  $\theta$  angle is at least 100°, or at least 105°, or at least 110°, or at least 115°, or at least 120°, or at least 125°, or at least 130°, and in addition or in the alternative, up to less than 180°, or up to 175°, or up to 170°, or up to 165°, or up to 160°, or up to 155°, or up to 150°, or up to 145°. Examples or ranges include 90° to less than 180°, or 90° to 175°, or 90° to 170°, or 90° to 165°, or 90° to 160°, or 90° to 155°, or 90° to 150°, or 90° to 145°, or 100° to less than 180°, or 100° to 175°, or 100° to 170°, or 100° to 165°, or 100° to 160°, or 100° to 155°, or 100° to 150°, or 100° to 145°, or 105° to less than 180°, or 105° to 175°, or 105° to 170°, or 105° to 165°, or 105° to 160°, or 105° to 155°, or 105° to 150°, or 105° to 145°, or 110° to less than 180°, or 110° to 175°, or 110° to 170°, or 110° to 165°, or 110° to 160°, or 110° to 155°, or 110° to 150°, or 110° to 145°, or 115° to less than 180°, or 115° to 175°, or 115° to 170°, or 115° to 165°, or 115° to 160°, or 115° to 155°, or 115° to 150°, or 115° to 145°, or 120° to less than 180°, or 120° to 175°, or 120° to 170°, or 120° to 165°, or 120° to 160°, or 120° to 155°, or 120° to 150°, or 120° to 145°.

**[0072]** In one embodiment or in combination of any mentioned embodiments, the  $\theta$  angle is at least 100°, or at least 105°, or at least 110°, or at least 115°, or at least 120°, or at least 125°, or at least 130°, and, in addition

or in the alternative, up to 155°, or up to 153°, or up to 152°, or up to 151°, or up to 150°, or up to 149°, or up to 148°.

**[0073]** In one embodiment or in combination of any mentioned embodiments, the filaments are made by spinning through a D shaped hole, and there is provided a spinneret hole configuration that has a D shaped hole, in which at least 60%, or at least 70%, or at least 80%, or at least 90%, or at least 95%, or at least 98% of the chord element 20 is in a straight line. The chord element 20 can also be in a horizontal line, and optionally each end bisects the inner circumference of the hole. In each case, the straight line can be measured end to end less 10% away from each end, meaning starting at a distance that is 10% away from point 24 in Figure 1 to a point that is 10% away from point 28. Thus, the entire distance measured would 80% of the chord. In another embodiment, the straight line is measured end to end less 5%, or less 2%, or less 1% away from each end, or is a straight line from end to end. In one embodiment, the chord is not “V” shaped, or is not “U” shaped, or is not “C” shaped, or is not irregularly shaped. The spinneret die head can have at least 1 D shaped hole with any of the mentioned features, or at least 10%, or at least 25%, or at least 50%, or at least 75%, or at least 90%, or 100%, of the holes on a spinneret die head can be D shaped.

**[0074]** The method for forming the spinneret 10 having a D-shape opening 15 is not particularly limited. It can be a structural insert, a part of a casting of the die plate, or added to the die plate after casting.

**[0075]** Referring to Figure 2, a diagram is provided of a filament 50 having a cross-sectional configuration of a closed C-shape that is prepared by extruding a cellulose acetate dope or solution through the above-described spinneret orifice. As defined herein, a filament or fiber exhibiting a closed C-shaped has a cross-section in the shape of a “C” having a first proximal end 65, a second proximal end 70, and a hollow core 60. As used throughout, a “closed C”, “closed C filament”, and “closed C shaped cross section” means a filament that is in the shape of a “C” having a first proximal end and a second proximal end and satisfies at least one condition A(i) or A(ii) and at least one condition B(i) or B(ii):

- A. at least a portion of a first proximal end is either:
- i. oriented toward at least a portion of a second proximal end; or
  - ii. contacting a portion of the second proximal end; and
- B. the first and second proximal ends form either:
- i. a channel defined by a gap or separation between the first proximal end and the second proximal end of the “C” shape and having a transverse distance  $D1$ , wherein the channel leads from an outer surface of the filament to a hollow core, wherein the hollow core is defined by an inner filament surface and having a diameter  $D2$ , and wherein  $D2/D1 > 1$ , or
  - ii. no channel resulting from at least a portion the first proximal end contacting at least a portion of the opposing second proximal end.

**[0076]** As illustrated in Figure 2, the filament 50 is in the shape of a “C” having a first proximal end 65 and a second proximal end 70 and satisfies at least one condition A(i) or A(ii) and at least one condition B(i) or B(ii):

- A. at least a portion of a first proximal end 65 is either:
- i. oriented toward at least a portion of the second proximal end 70; or
  - ii. contacting a portion of the second proximal end 70, and as shown in Figure 2, there is no contact; and
- B. the first and second proximal ends 65 and 70 form either:
- i. a channel defined by a gap or separation between the first proximal end 65 and the second proximal end 70 of the “C” shape and having a transverse distance  $D1$ , wherein the channel leads from an outer surface 55a of the filament 50 to a hollow core 60, wherein the hollow core 60 is defined by an inner filament surface 55b and having a diameter  $D2$ , and wherein  $D2/D1 > 1$ , or
  - ii. no channel or passageway resulting from at least a portion the first proximal end 65 contacting at least a portion of the

opposing second proximal end 70, and as shown, the proximal ends in this case do not contact each other.

**[0077]** In one embodiment or in combination with any of the mentioned embodiments, the channel opens into an annulus that has a diameter D2 that is greater than the smallest channel diameter D1. The distance D1 of the channel is taken as the smallest distance within the channel defined by the gap between the first proximal end 65 and the second proximal end 70 of the “C” shaped filament, and the diameter D2 is taken as the largest diameter within the hollow core 60. The ratio of D2/D1 in the same units may be greater than 1:1, or at least 1.1:1, or at least 1.2:1, or at least 1.3:1, or at least 1.4:1, or at least 1.5:1, or at least 1.6:1, or at least 1.7:1, or at least 1.8:1, or at least 1.9:1, or at least 2:1, or at least 2.1:1, or at least 2.3:1, or at least 2.5:1, or at least 2.8:1, or at least 3:1, or at least 3.5:1, or at least 4:1 or at least 4.5:1 or at least 5:1 or at least 5.5:1, or at least 6:1.

**[0078]** Referring to Figures 2A and 2C-2F, the delineation and definition of a proximal end of the C-cross section shape is more particularly defined. As shown in Figure 2F, the filament 50 may include an outer periphery 55a forming an outer arc and an inner periphery 55b forming an inner arc circumscribing and forming a hollow core 60. Moreover, as depicted in Figures 2A, 2C, and 2D, the C shaped filament has proximal ends 65 and 70 having planar tips x and y, respectively. In determining if the filament 50 is a closed C as defined herein, the orientation of the proximal ends 65 and 70 is determined. Accordingly, as shown in Figures 2A, 2C, and 2D, the orientation of end 65 is the direction of an imaginary line 65a or 65b extending away from and perpendicular to the plane x of proximal end 65. Similarly, as shown in Figures 2A, 2C, and 2D, the orientation of end 70 is the direction of an imaginary line 70a and 70b extending away from and is perpendicular to the plane y of proximal end 70. Lines 65a or 65b can be drawn anywhere along and perpendicular to the x plane, and even though some lines may not intersect the opposing plane y of opposing proximal end 70, if any line along and perpendicular to the x axis intersects the opposing plane y of opposing proximal end 70, the filament is a closed C shape. Likewise, lines 70a or 70b can be

drawn anywhere along and perpendicular to the y plane, and even though some lines may not intersect the opposing plane x of proximal end 70, if any line along and perpendicular to the y plane intersects the opposing plane of the opposing proximal end 70, the filament is a closed C shape. As illustrated, this configuration would be a closed C shaped filament since the orientation of proximal end 65 is toward the proximal end 70 as shown by imaginary orientation line 65a intersecting the plane y of proximal end 70 even though imaginary line 65b does not. Likewise, this configuration would be a closed C shaped filament since the orientation of proximal end 70 is toward the proximal end 65 as shown by imaginary orientation line 70a intersecting the plane x of proximal end 65 even though imaginary line 70b does not. Either one of these conditions would satisfy the requirement that the proximal ends are oriented toward each other.

**[0079]** Referring to Figure 2C, another embodiment of the closed C filament is illustrated having mutually opposed proximal ends. First proximal end 65 is oriented toward the second proximal end 70 as shown by the direction of line 65a intersecting the opposing plane y of second proximal end 70 even though lines 70a and 65b do not intersect their respective opposing planes. Correspondingly, second proximal end 70 is oriented toward the first proximal end 65 as shown by the intersection of a line 70b intersecting the opposing plane x of proximal end 65. Either one of these conditions would satisfy the requirement that the proximal ends are oriented toward each other. It is to be understood that any imaginary line between proximal ends 65 and 70 within the region between the region defined by 65a and 70b, respectively, would also demonstrate that the orientation of the proximal ends 65 and 70 are mutually opposed and oriented toward each other.

**[0080]** As shown in Figure 2D, this type of closed C shape would be considered a “single opposed” C shape since only one proximal end is oriented toward the opposing proximal end, i.e., the orientation of proximal end 70 shown by a line 70a does not intersect the plane x of proximal end 65 because no matter where the imaginary line 70a is drawn along and perpendicular to the

plane y, the orientation of end 70 does not intersect any portion of the proximal end 65 along its plane x.

**[0081]** Accordingly, when a portion of only one proximal end, 65 or 70, is oriented toward at least a portion of the other proximal end, this is considered to be a single opposed. Additionally, when at least a portion of each proximal end, 65 and 70, are oriented toward at least a portion of the other proximal end, the ends 65 and 70 are considered to be mutually opposed.

**[0082]** In one embodiment or in combination with any of the mentioned embodiments, at least 30%, or at least 40%, or at least 50%, or at least 60%, or at least 65%, or at least 70%, or at least 75% of the closed C shaped fibers are mutually opposed.

**[0083]** In the case where the end or tip is rounded, the proximal end would be an imaginary line connecting a point on the inner periphery and a point on the outer periphery, the points obtained when no substantial angular changes are seen as the point travels along the periphery. The orientation of the proximal end is an imaginary line that is perpendicular to the planar portion of the proximal end, or perpendicular to the imaginary line connecting the two mentioned points on a rounded tip configuration.

**[0084]** Where a proximal end has multiple planes, any one of the planes can be taken as a proximal end provided that the plane selected is at least 50% of the length of the longest plane on the proximal end. For example, referring to Figure 2E, proximal end 70 has non-coplanar surfaces s1 and s2. Planar surface s1 has a length that is not at least 50% of the length of s2, and therefore, only plane s2 qualifies as a plane for determining orientation. However, if the length of surface s1 is at least 50% of the length of s2, then either surface can be selected to determine the orientation of proximal end 70 and the target plane for the orientation of proximal end 65. If the s1 length is less than 50% of the s2 length, then only the s2 surface qualifies as the basis on which to determine the orientation of proximal end 70 and the target plane for the orientation of plane 65. As illustrated, the plane of surface s2 would intersect proximal end 65, and therefore the proximal end 70 is oriented toward the proximal end 70. Planar surface s1 would not qualify as a candidate for determining the

orientation of proximal end 70 because it is less than 50% of the length of planar surface s2. However, if it were at least 50% of the length of planar surface s2, proximal end 70 would nevertheless be oriented toward proximal end 65 because planar surface s2 can be selected for determining the orientation of proximal end 70.

**[0085]** Referring to Figure 2I the filament illustrated is considered to have a closed "C" shape. This is because the x plane of proximal end 65 is contacting a portion of proximal end 70 along the y axis of proximal end 70.

**[0086]** Conversely, Figures 2B, 2G, and 2H illustrate a filament 50 having an inner core 60 that is not considered to be a closed C shaped filament of the present invention. In Figure 2B, the orientation of proximal end 65 is not toward proximal end 70 because no imaginary line perpendicular to and along the plane x of proximal end 65, whether as orientation line 65a or 65b, can intersect proximal end 70 along its plane y, and proximal end 70 is not oriented toward proximal end 65 as shown by imaginary orientation lines 70a and 70b not intersecting proximal end 65 along its plane x.

**[0087]** Referring to Figure 2G, a filament 50 having an inner core 60 that is not considered to have a closed "C" shape is illustrated. Because neither of the proximal ends 65 nor 70 are oriented toward each other as shown in that any line perpendicular to either plane x or plane y does not intersect the opposing plane.

**[0088]** In another case, Figure 2H illustrates a filament 50 having an inner core 60 that is not considered to have a closed "C" shape because neither proximal end 65 nor 70 are oriented toward each other as shown in that any line perpendicular to either plane x or plane y does not intersect the opposing plane.

**[0089]** Referring again to Figure 2A, the core 60 of filament 50 is hollow, providing the filament 50 with a hollow cross-section. The term "core" as used herein is not limited to a perfect circle or one in which the circumference is completely closed. For example, a hollow core can be oval or irregular or distorted shaped, and the proximal ends 65 and 70 can be touching or in contact or can be distal such that an arcuate loop is provided in which a part of the

circumference is opened. The first proximal end 65 and second end 70 can be spaced apart a distance of less than 1.0 radian, or less than 0.8 radian, or less than 0.5 radian, or less than 0.3 radian, or less than 0.1 radian and still be characterized as a closed C configuration, and in each instance not touching, or be spaced apart by at least 0.01 radian, or at least 0.05 radian.

**[0090]** The filament can have a closed C configuration in which the proximal ends 65 and 70 are touching. The proximal ends 65 and 70 can be touching but not bonded or fused. Alternatively, the closed C configuration can be one in which the proximal ends 65 and 70 are not touching as noted above. In one embodiment or in combination with any other mentioned embodiments, the hollow core 60 has a diameter in its largest dimension that is larger than the largest distance between the proximal ends 65 and 70. The hollow core 60 can even have a diameter in its smallest dimension that is larger than the largest distance between the proximal ends 65 and 70 hollow core.

**[0091]** Advantageously, the area of the hollow core is large relative to a total cross-sectional area of the filament. For example, the hollow core 60 can have a cross section area that is at least 20% of the cross-section area of the filament 50. The cross-section shape can be calculated by drawing a closure in the gap between proximal ends 65 and 70 on the hollow core 60 and on the outer periphery 55 and determining the cross section of the filament and hollow core 60 hollow core. The hollow core 60 can have a cross-section area that is at least 25%, or at least 30%, or at least 35%, or at least 40%, or at least 45%, or at least 50%, or at least 60%, or at least 65%, or at least 70%, or at least 75%, or at least 80%, or at least 85%, and up to 90%, or up to 80%, or up to 70%, or up to 60% of the cross-section area of the filament 50.

**[0092]** In one embodiment or in combination with any mentioned embodiment, the hollow core can have an aspect ratio (width:height) of not more than 2:1, or not more than 1.9:1, or not more than 1.8:1, or not more than 1.7:1, or not more than 1.6:1, or not more than 1.5:1, or not more than 1.4:1, or not more than 1.3:1, or not more than 1.2:1, and at least 0.5:1, or at least 0.6:1, or at least 0.7:1, or at least 0.8:1, or at least 0.9:1, or at least 1:1. The width is longest diameter of the hollow core portion 60 that is substantially perpendicular

to the plane of the channel D1 between the proximal ends of the C shaped filament (channel plane) and the height is longest portion of a distance that is substantially in line with the channel plane and commences at the first entry into hollow core to the inner periphery 55b.

5 **[0093]** Reference can be made to Figure 2F to illustrate the concept. C shaped filament 50 having an outer periphery 55a and an inner hollow core or annulus 60 has an aspect ratio that can be represented by a “w” that is the longest width or diameter of the hollow core 60, and a line “h” that represents the height that intersects the first plane of the channel entry between proximal  
10 ends 65 and 70 into the hollow core 60 as shown by line 1. Although line 2 also represents an entry plane into the hollow core 60, it is the second plane of entry and the longest height would commence at the plane of line 1. The ratio of the length of line w to the length of line h is the aspect ratio of the hollow core 60 in the filament 50.

15 **[0094]** In one embodiment or in combination with any mentioned embodiment, at least 70% of distance of the inner periphery 55b is continuous based on a distance of a completed inner periphery. A completed inner periphery is the inner periphery 55b in which an imaginary straight line is drawn connecting both proximal ends, starting from the end of the inner periphery of  
20 one proximal end and extending to the end of the inner periphery of the opposing proximal end. The periphery along the proximal ends forming the channel is not part of the inner periphery. At least 75%, or at least 80%, or at least 85%, or at least 90%, or at least 95%, or at least 96%, or at least 97%, or at least 98%, or at least 99% of distance of the inner periphery 55b is continuous  
25 based on a distance of a completed inner periphery.

**[0095]** In one embodiment or in combination with any mentioned embodiment, there is provided a high population density of at least 30%, or at least 40%, or at least 50%, or at least 60%, or at least 65%, or at least 70%, or at least 75% or at least 80%, or at least 90% of Closed C shaped cellulose acetate fibers per 150 fibers are mutually opposed.  
30

**[0096]** In one embodiment or in combination with any mentioned embodiment, proximal ends 65 and 70 of the filament are co-extensively

oriented to each other in an amount of at least 5%, or at least 10%, or at least 15%, or at least 20%, or at least 25%, or at least 30%, or at least 35%, or at least 40%, or at least 50%, or at least 60%, or at least 70%, or at least 80%, or at least 85%, or at least 90%. The percentage can be determined by the following equation:

$$\%O = \frac{A_o}{A_t} \times 100$$

where %O is the percent of co-extensive orientation; and

$A_o$  is the area of overlap; and

$A_t$  is the entire area defined by  $A_o$  plus the area of the 4 triangles that have a side contiguous to a boundary defining the overlap  $A_o$ .

**[0097]** Overlapping rectangles are determined by the parallel continuations of the tip orientations as described above, and drawing imaginary lines that are at right angles to the orientation of the proximal ends. The entire area of the largest area where all lines intersect is the  $A_t$ . To illustrate this concept, reference can be made to Figure 2A, reference imaginary drawn lines 70a, 70b, 65a, and 65b are as discussed above. The imaginary lines drawn as x and y are at right angles, or perpendicular to, the directional orientation of their respective proximal ends. Reference letters A, B, C, and D are the 4 triangles having sides contiguous to a border on the co-extensive area F. The  $A_t$  is the combined areas F + A + B + C + D.

**[0098]** In one embodiment or in combination of any mentioned embodiments, a filament contains only one hollow core.

**[0099]** In one embodiment or in combination of any mentioned embodiments, the filaments form a hollow core around which is a filament having a continuous outer periphery. In one embodiment or in combination of any mentioned embodiments, the filaments form a hollow core around which is a filament having a continuous outer periphery and at least a portion of each proximal end contacts the other. This is an example of an entirely closed C shaped filament having a zero-radian gap between the proximal ends. For example, at least 0.5%, or at least 1%, or at least 2%, or at least 3%, and/or up

to 10% of the filaments per 40 filament count can have a continuous outer periphery where the proximal ends are touching each other.

**[0100]** There is now also provided a method for consistently producing a high population density of closed C filaments. In producing the acetate filament, a spinning dope containing cellulose acetate and a solvent is prepared in advance and it is dry-spun under conditions known to those skilled in the art. The spinning solution temperature is elevated and is maintained by passing it through a heated candle filter. The candle filter temperature is maintained by passing hot water through its internal channels. Although the actual temperature of the spinning solution is a few degrees below the candle filter water temperature, the candle filter water temperature serves as a good proxy for providing a spinning dope solution temperature. We have found that the candle filter water temperature should be maintained at a temperature within 40°C to 62°C, or from 40°C to less than 62°C, or from 45°C to 60°C. When the candle filter water temperature is much lower than 40°C, the solvent in the spinning solution is not fully evaporated and filament breakage is observed. Although not to be bound by any theory, it is believed that filament breakage can be attributed to the spinning step where a plurality of single filaments are in close proximity or closely attached to each other, causing a breakage of the yarn. When the temperature is much higher than 62°C, the solvent evaporation rate is rapid and the contour of the cross-section is no longer uniform, and the desired C-shape cross-section configuration is no longer obtained. Accordingly, while the temperature of the spinning solution discharged from the spinneret can be less than 62°C, or less than 61°C, or from 40°C to 60°C, or not more than 60°C, or less than 60°C, or from 40° to 58°C, or 40° to 56°C, or 45° to 56°C, or 50° to 62°C, or 45° to 60°C, or 50°C to 60°C, inclusive. Desirably, the candle filter water temperature is not, under the given process conditions, above the boiling point of the solvent used in the spinning solution, such as acetone. Each of these ranges includes the end points, and also provided support for ranges between these values such that that the endpoints are excluded (e.g., more than and/or less than).

**[0101]** We have discovered that when the temperature is much higher than 62°C the solvent evaporation rate is rapid and the contour of the cross-section is no longer uniform, so the desired C-shape cross-sectional form is not typically obtained. Within a stated candle filter water temperature range, the particular temperature selected is influenced by the dpf of the filament spun through the die hole. As the dpf of the filament decreases, the candle filter water temperature should also be lowered to ensure an excess of solvent is not evaporated prematurely. On the other hand, the candle filter water temperature should be increased to spin filaments having a higher dpf. At the spinneret, the solvent dope can be extruded through a plurality of holes to form continuous cellulose acetate filaments. The filaments may be gathered together to form bundles of several hundred, or even thousand, individual filaments. Each of these bundles, or bands, may include at least 100, 150, 200, 250, 300, 350, or 400 and/or not more than 1000, 900, 850, 800, 750, or 700 filaments. The spinnerets may be operated at any speed suitable to produce filaments and bundles having desired size and shape.

**[0102]** The spinning dope composition contains cellulose acetate and a solvent in suitable amounts. The terms, "cellulose acetate tow", "acetate tow", or "acetate tow band" as used herein, refers to a continuous, crimped filament band comprising of cellulose acetate filaments. The term "cellulose acetate", as used herein, refers to an acetate ester of cellulose wherein the hydrogen in the hydroxyl groups of the cellulose glucose unit is replaced by acetyl groups through an acetylation reaction. The cellulose acetate can have a degree of substitution ranging from 2.2 to less than 3. As used herein, the term "degree of substitution" or "DS" refers to the average number of acyl substituents per anhydroglucose ring of the cellulose polymer, wherein the maximum degree of substitution is 3.0. In some embodiments, suitable cellulose acetates may have a degree of substitution less than 3 acetyl groups per glucose unit, preferably an average in the range of 2.2 to 2.8, or in the range of 2.4 to 2.7. In some cases, at least 90, 91, 92, 93, 94, 95, 96, 97, 98, or 99 percent of the cellulose acetate has a DS of greater than 2.2, or 2.25. In some cases, at least 90 percent of the cellulose acetate can have a DS of greater than 2.2, 2.25, 2.3,

or 2.35. Typically, acetyl groups can make up at least 1, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 percent and/or not more than 99, 95, 90, 85, 80, 75, or 70 percent of the total acyl substituents.

**[0103]** The amount of cellulose acetate in the spinning dope composition can be from 22 weight % to 32 weight %, or more than 22 weight % to 32 weight %, or more than 24 weight % to 32 weight %, or from 26 weight % to 30 weight % and from 28 weight % to 30 weight %, based on the total weight of the dope solution. The amount of solvent present in the dope composition is from 65 weight % to 78 weight %, and desirably from 68 weight % to 71 weight %, based on the total weight of the dope solution. The inherent viscosity of the cellulose acetate in the spinning solution can be from 1.35 to 1.60, or from 1.45 to 1.58.

**[0104]** The solids content (i.e., solids added) of the spinning solution is generally between 22 and 32 weight percent. At the higher solids content, there is less amount of solvent that needs to be recovered, but at much above 32 weight percent, the spinning solution viscosity can be too high for good extrusion through the small spinneret holes. At a solids content from 22 weight percent or less, the flow rate of the dope solution through the spinneret is difficult to control, an excess of solvent has to be evaporated from the filament and a consistent filament closed C shape is difficult to control, and the amount of acetone recovery is high. Additionally, spinning solutions containing low solids when spun into filaments tend to cause the filaments to stick to the outside surface of the metal face of the spinnerets and are, therefore, difficult to pull the filaments into a bundle or tow band.

**[0105]** The spinning dope solution may also contain minor amounts of a delusterant such as  $\text{TiO}_2$ , and minor amount of water, and minor amounts of a plasticizer. The dope solution according to the present invention generally has minor amounts of titanium dioxide added and minor amounts of water. The amount of  $\text{TiO}_2$  in the total spinning solution is generally below 1 wt.%, or not more than 0.5 wt.%, or not more than 0.3 wt.%, or no added  $\text{TiO}_2$ . A minor amount of  $\text{TiO}_2$  can be added to increase the whiteness of the resulting filter. Excessively high amounts of  $\text{TiO}_2$  can plug the fine spinneret holes.

**[0106]** If present, the amounts of delusterant, plasticizer, and water can each be 5 wt.% or less, or not more than 5 wt.% cumulatively. In one embodiment, aside from water, delusterant, cellulose acetate, and plasticizer, the remainder of the spinning dope solution is solvent, such as acetone. In one  
5 embodiment or in combination with any of the mentioned embodiments, the amount of acetone is at least 60 wt.%, or at least 70 wt.%, or at least 80 wt.%, or at least 90 wt.%, or at least 95 wt.%, or at least 98 wt.%, or 100 wt.%, based on the weight of all solvents other than water. Other solvents having an evaporation rate profile different than acetone will affect the shape of the  
10 filament under the process conditions mentioned, and therefore, their amount should be minimized.

**[0107]** In one embodiment or in combination with any of the mentioned embodiments, the spinning dope solution contains not more than 4.5 wt.%, or not more than 4 wt.%, or not more than 3 wt.%, or not more than 2 wt.%, or not  
15 more than 1 wt.%, or not more than 0.5 wt.% plasticizer based on the total weight of the cellulose acetate and plasticizer in the dope, or based on the weight spinning dope solution, or based on the total weight of the cellulose acetate and plasticizer in the fiber, or based on the weight of the fiber, or no plasticizer is added to the solution, or no plasticizer is present in the solution. If  
20 a plasticizer is employed, the plasticizer can be a compound as opposed to a polymer, and can have a molecular weight of not more than 400 g/mole, or not more than 300 g/mole, or not more than 250 g/mole. The filament, upon exit from the spinneret, may also contain the same percentages of plasticizer, or lack of plasticizer, and any process for making the closed C shaped filaments  
25 can include dry spinning without plasticizer added to the dope solution or, if added, is within the limits described.

**[0108]** The amount of water present in the spinning solution of the present invention is generally less than 5 weight percent, or not more than 3 weight percent, or not more than 1 to 2 weight percent, based on the weight of the  
30 dope solution or the filament. Amounts of water much above 3 weight percent tend to slow the drying time of the resulting filaments whereas amounts of water

much below about 1 weight percent are difficult to obtain since the acetone is recycled from water by distillation and ambient air is humid.

**[0109]** We have also discovered that the draft ratio influences the ability to consistently form a closed C shaped filament. The “draft ratio” is the ratio of the filament take up speed by the godet rolls to the filament extrusion speed out of the spinneret. The draft ratio in the dry spinning process can be at least 0.9 or at least 0.95, or at least 1, or at least 1.01, or at least 1.05, or at least 1.1, or at least 1.2 and in addition or in the alternative, up to 1.6, or up to 1.5, or up to 1.4, or up to 1.3, or up to 1.2, or up to 1.15. Suitable ranges include draft ratios in the range of from 0.9 to 1.6, or from 0.95 to 1.5, or from 1.0 to 1.4, or from 1.05 to 1.4, or from 1.08 to 1.6, or from 1.08 to 1.4, or from 1.08 to 1.3, or from 1.08 to 1.2, or from 1.08 to 1.15, or from 1.05 to 1.6, or from 1.1 to 1.6, or from 1.2 to 1.6, or from 1.3 to 1.6, or from 1.4 to 1.6.

**[0110]** We have also discovered that at a given spinnerette hole diameter and theta angle, the dpf of the filament can be increased as the draft ratio is decreased, or conversely, the dpf of the filament can be decreased as the draft ratio is increased, while surprisingly successfully obtaining a filament having a closed C shape cross section. Thus, in one embodiment or in combination with any mentioned embodiments, there is provided a process in which the dpf of a closed C shaped filament spun through a D shaped spinneret hole at a fixed theta angle is either decreased with an increase in the draft ratio or is increased with a decrease in the draft ratio. In one embodiment or in combination with any mentioned embodiments, there is provided a process in which the dpf of a closed C shaped filament spun through a D shaped spinneret hole at a fixed theta angle is changed in an amount of 0.35 to 0.7 units for every 0.1 unit change in draft ratio. Optionally, the dpf can be adjusted within +/-3 full integers for a given D shaped hole. This provides a significant advantage for a manufacturer to create a window of dpf ranges at a given D shaped hole size without having to change the die having different spinneret hole sizes for every change in dpf, while continuing to successfully and consistently make a closed C shaped filament.

**[0111]** In one embodiment or in combination with any mentioned embodiments, there is provided a closed C shaped filament and a process for making a closed C shaped filament having a dpf of at least 5, or at least 6, or at least 7, in each case having a draft ratio of not more than 1.4, or not more than 1.3, or not more than 1.2, or not more than 1.15, or not more than 1.1, or not more than 1.05, or not more than 1.0. In one embodiment or in combination with any of the mentioned embodiments, there is provided a filament and a process for making a filament having a dpf of less than 5, or not more than 4, or not more than 3.5, or not more than 3, or not more than 2.5, or not more than 2, or not more than 1.8, or not more than 1.5, or not more than 1.3, or not more than 1.1, or not more than 1, in each case made at a draft ratio of more than 1.4, or at least 1.45, or at least 1.5, or at least 1.55.

**[0112]** We have also discovered that the ratio of the dpf to the theta angle of the D shaped hole influences the population density values of closed C cross section filament shapes, and further, that the candle filter dope temperature also has an influence, yet none of these factors alone account for obtaining high densities of closed C shaped fibers. Even some combinations of these factors do not account for obtaining a high population density of closed C shaped fibers. For example, a high population density and a low population density of closed C shaped fibers can be obtained at the same candle filter dope temperature, or high population density and a low density of closed C shaped fibers can be obtained at the same candle filter dope temperature and the same dpf, or high population density and a low population density of closed C shaped fibers can be obtained at the same dpf and same theta angle. We have now discovered that consistent and reliable closed C shaped fibers at a high population density can be obtained, or in other words, there is provided a high population density of filaments or staple fibers (in any amounts mentioned herein at or above 50% per 150 C shaped filaments), and there is provided a process for making closed C shaped filaments and staples, by spinning a cellulose acetate dope through a D shaped hole having a theta  $\theta$  angle to make a closed C shaped filament having a dpf, wherein:

- a) the  $\theta$  angle is at least  $90^\circ$ , or optionally any of the minimum  $\theta$  angles mentioned below, and
- b) the ratio of  $\theta$  angle to the dpf of the spun filament is less than 33:1, and
- c) the cellulose acetate dope temperature is not more than  $62^\circ\text{C}$ , provided that
- a. if the ratio of  $\theta$ :dpf is at or lower than 19:1 (e.g., not more than 18:1, or not more than 17:1, or not more than 16:1, etc.), the dope temperature is less than  $62^\circ\text{C}$ ; and
- b. if the ratio of  $\theta$ :dpf is more than 19:1 (e.g., 19.5:1, 20:1, 27:1 etc.), the dope temperature is less than  $60^\circ\text{C}$ ; and
- c. if the dpf of the filament is less than 6, or not more than 5, or not more than 4.5, or not more than 4, the dope temperature is less than  $60^\circ\text{C}$ .

**[0113]** The  $\theta$ :dpf ratio can be at least 9:1, or at least 10:1, or at least 11:1, or at least 12:1, or at least 13:1, or at least 15:1, or at least 16:1, or at least 17:1, or at least 18:1, or at least 19:1, or at least 19.5:1, or at least 20:1, or at least 21:1, or at least 22:1, or at least 23:1, or at least 24:1, and in addition or in the alternative, less than 33:1, or up to 32:1, or up to 31:1, or up to 30:1, or up to 29:1, or up to 28:1, or up to 24:1, or up to 22:1, or up to 21:1, or up to 20:1, or up to 19.5:1, or up to 19:1, or up to 18.5:1, or up to 18:1. Examples of  $\theta$ :dpf ranges include 13:1 to less than 33:1, or 13:1 to 32:1, or 15:1 to 19:1, or 16:1 to 18.5:1, or 13:1 to 18:1, or 15:1 to 18:1, or more than 19:1 to 32:1, or 19.5:1 to less than 33:1, or 20:1 to 32:1, or 21:1 to 32:1, or 20:1 to 28:1. In each case the cellulose acetate dope temperature is not more than  $62^\circ\text{C}$ , or less than  $62^\circ\text{C}$ , or not more than  $61^\circ\text{C}$ , or not more than  $60^\circ\text{C}$ , or less than  $60^\circ\text{C}$ , or not more than  $59^\circ\text{C}$ , or not more than  $58^\circ\text{C}$ , or not more than  $56^\circ\text{C}$ , and optionally at least  $40^\circ\text{C}$ , or at least  $45^\circ\text{C}$ , or at least  $48^\circ\text{C}$ , or at least  $50^\circ\text{C}$ , or at least  $53^\circ\text{C}$ .

**[0114]** As noted above, the  $\theta$  angle is at least  $90^\circ$ , and suitably from  $100^\circ$  to less than  $180^\circ$ , or from  $100^\circ$  to  $170^\circ$ , or from  $100^\circ$  to  $160^\circ$ , or from  $100^\circ$  to  $148^\circ$ , or  $105^\circ$  to  $148^\circ$ , or  $120^\circ$  to  $148^\circ$ , or  $130^\circ$  to  $148^\circ$ .

**[0115]** In all cases, the ratio of  $\theta$  angle to the dpf of the spun filament is less than 33:1, but can also be not more than 32:1, or not more than 30:1, or not more than 28:1.

5 **[0116]** In one embodiment or in combination with any of the mentioned embodiment, the theta angle increases with an increase in the dpf of the fiber. For example, at a dpf of greater than 10, the  $\theta$  angle can be greater than 150°, and at dpf between 6 and 10, the  $\theta$  angle can be between 120° and 150°, and at a dpf of less than 6 or less than 5, the  $\theta$  angle can be less than 120° and at least 90°.

10 **[0117]** The cellulose acetate dope temperature is not more than 62°C, although there are instances in which the dope temperature is less than 60°C, and we have also discovered the dependence of the theta angle and dpf to the dope temperature. If the ratio of  $\theta$ :dpf is at or lower than 19:1, the dope temperature is less than 62°C. We have found that higher dope temperatures, for example at 65°C, 70°C, etc., fail to produce a high population density of closed C shaped fibers, even across a variety of  $\theta$ :dpf ratios. We have also found, however, that if the ratio of  $\theta$ :dpf is more than 19:1 (e.g., 19:5:1, 20:1, 27:1, etc.), dope temperatures of must remain below 60°C to effectively generate a high population density of closed C shaped fibers. We have further found that another instance of when the dope temperature is set to a temperature that is less than 60°C is when the dpf of the filament is less than 6, or not more than 5.5, or not more than 5, or not more than 4.5, regardless of the  $\theta$ :dpf ratio. Desirably, the dope temperature when the ratio of  $\theta$ :dpf is more than 19:1, or when the dpf is less than 6, are within a range of 45°C to 58°C, or 50° to 58°C, or 52°C to 56°C, and these temperatures as well as 45°C to 61°C, or 48°C to 61°C, or 50°C to 60°C, or 50°C to 58°C, or 50°C to 56°C, are also suitable ranges when the ratio of  $\theta$ :dpf is less than 19:1 since they are all below 62°C.

20 **[0118]** The take-up rate of the filaments is desirably at least 200 m/minute, or at least 250 m/minute, or at least 300 m/minute to avoid a high build-up of volatiles, although this rate can be lowered if suitable equipment is installed to rapidly remove vapors. Suitable take up rates can range of from 200 to 900

m/minute, or from 250 to 900 m/minute, or from 300 to 900 m/minute, or from 350 to 900 meters per minute, or from 400 to 900 m/min, or from 200 to 700 m/min, or from 250 to 700 m/minute, from 300 to 700 m/minute. These rates are on a continuous basis over at least 8 hours, or at least 24 hours, or at least 1 week, or at least 1 month.

**[0119]** The filaments leave the spinneret and enter a drying cabinet. The drying cabinets have a top and a bottom. The top has a first air temperature and a first air flow, and the bottom has a second air temperature and a second air flow. In one embodiment, the top and bottom air temperature are different. In another embodiment, the top and bottom air flow are different. Solvent wet filaments enter the apparatus at the top and are subjected to drying air temperatures and flow rates from the top and bottom. The dry filaments are then removed from the bottom of the drying cabinet and collected for further processing.

**[0120]** The top air flow can be at least 10 cubic feet per minute (cfm), or at least 40 cfm, or at least 60 cfm, or at least 90 cfm, or at least 100 cfm, or at least 105 cfm. Examples of ranges include 10 to 125 cfm, or from 90 to 125 cfm, or from 100 to 125 cfm, or from 105 to 120 cfm. The top air temperature can be at least 55°C, or at least 60°C, or at least 65°C, or at least 70°C, or least 80°C, or more than 80°C, or at least 82°C, or at least 85°C, and in addition or in the alternative, up to 120°C, or up to 115°C, or up to 110°C, or up to 100°C, or up to 95°C, or up to 93°C. Example of suitable ranges include from 55°C to 120°C, or from 55°C to 115°C, or from 55°C to 110°C, or from 55°C to 105°C, or from 55°C to 100°C, or from 60°C to 120°C, or from 60°C to 115°C, or from 60°C to 110°C, or from 60°C to 105°C, or from 60°C to 100°C, or from 80°C to 120°C, or from 80°C to 115°C, or from 80°C to 110°C, or from 80°C to 105°C, or from 80°C to 100°C, or from 85°C to 120°C, or from 85°C to 115°C, or from 85°C to 110°C, or from 85°C to 100°C, or from 85°C to 95°C, or from 82°C to less than 100°C, or from 85°C to 95°C, or from more than 88°C to 95°C, or from 90°C to 95°C, or from 65°C to less than 95°C, or from 65°C to 93°C.

**[0121]** The bottom air flow can be from at least 10 cubic feet per minute (cfm), or at least 40 cfm, or at least 60 cfm, or at least 90 cfm, or at least 100

cfm, or at least 105 cfm. Examples of ranges include 10 to 125 cfm, or from 90 to 125 cfm, or from 100 to 125 cfm, or from 105 to 120 cfm, or from 12 to 120 cfm, or from 25 to 115 cfm. The bottom air temperature is at least 75°C, or at least 78°C, or at least 80°C, or at least 85°C, and in addition or in the alternative, up to 120°C, or up to 115°C, or up to 110°C, or up to 105°C, or up to 100°C, or up to 95°C. Examples of suitable bottom air temperatures range from 75°C to 120°C, or from 75°C to 115°C, or from 75°C to 110°C, or from 75°C to 105°C, or from 75°C to 100°C, or from 75°C to 95°C, or from 78°C to 120°C, or from 78°C to 115°C, or from 78°C to 110°C, or from 78°C to 105°C, or from 78°C to 100°C, or from 78°C to 95°C, or from 80°C to 120°C, or from 80°C to 115°C, or from 80°C to 110°C, or from 80°C to 105°C, or from 80°C to 100°C, or from 80°C to 95°C, or from 85°C to 120°C, or from 85°C to 115°C, or from 85°C to 110°C, or from 85°C to 105°C, or from 85°C to 100°C, or from 85°C to 95°C.

**[0122]** The bottom air flow can be at least 1 cfm lower than the top air flow, or at least 1.5 cfm lower, or at least 2 cfm lower, or at least 5 cfm lower, or at least 10 cfm lower, or at least 15 cfm lower, or at least 20 cfm lower, or at least 25 cfm lower than the top air flow, and up to about 60 cfm, or up to 55 cfm lower than the top air flow. Suitable ranges of the bottom air flow include 1 to 60 cfm, or 1 to 55 cfm or 5 to 55 cfm, or 10 to 55 cfm, or 20 to 55 cfm lower than the top air flow.

**[0123]** The bottom air temperature can be at least 1°C lower than the top air temperature, or at least 2°C lower, or at least 3°C lower, or at least 5°C lower, or at least 7°C lower than the top air temperature, and up to 20°C lower than the top air temperature, or up to 15°C lower, or up to 10°C lower, or up to 8°C lower than the top air temperature. Suitable ranges of the bottom air temperature include 1 to 20°C, or 1 to 15°C, or 1 to 10°C, or 3 to 7°C lower than the top air temperature.

**[0124]** The solvent level in the filaments exiting the cabinets prior to entering a dryer can be 4-18 wt.%, or 6-10 wt.% solvent (e.g., acetone).

**[0125]** Filament processing includes passing the filaments through a crimping zone where a patterned wavelike shape is imparted to at least a portion, or substantially all, of the individual filaments as well as processing of

filaments into staple fibers. The crimping zone includes at least one crimping device for mechanically crimping the filament yarn. An example of a mechanical crimper includes a “stuffing box” or “stuffer box” crimper that uses a plurality of rollers to generate friction, which causes the filaments to buckle and form crimps inside the box. Other types of crimpers may also be used. Examples of equipment suitable for imparting crimp to a filament yarn are described in, for example, U.S. Patent Nos. 9,179,709; 2,346,258; 3,353,239; 3,571,870; 3,813,740; 4,004,330; 4,095,318; 5,025,538; 7,152,288; and 7,585,442. In some cases, the crimping step may be performed at a rate of at least 50, 75, 100, 125, 150, 175, 200, 225, or 250 meters per minute (m/min) and/or not more than 750, 600, 550, 500, 475, 450, 425, 400, 375, 350, 325, or 300 m/min.

**[0126]** Crimping can be performed such that filaments, and the staple fibers made therefrom, have a crimp frequency of at least 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, or 14 and/or not more than 30, 29, 28, 27, 26, 25, 24, 23, 22, 21, 20, 19, 18, 17, 16, 15, 14, 13, 12, 10, 9, 8, 7, or 6 crimps per inch (CPI), measured according to ASTM D3937. The particular CPI employed depends on the application, with some applications requiring uncrimped and other light or frequent crimping. The crimp amplitude of the filaments or filaments may vary and can be, for example, at least 0.85, 0.90, 0.91, 0.92, 0.93, 0.94, 0.95, 0.96, 0.97, 0.98, 0.99, 1.00, 1.01, 1.02, 1.03, 1.04, or 1.05 mm.

**[0127]** After crimping, the filament yarn or tow band may further be dried in a drying zone in order to reduce the moisture and/or solvent content of the filament yarn. In some cases, the drying performed in the drying zone may be sufficient to reduce the final moisture content of the filament yarn to at least 3.5, 4, 4.5, 5, 5.5, 6, 6.5, or 7 weight percent, based on the total weight of the filament yarn and/or not more than 9, 8.5, 8, 7.5, 7, or 6.5 weight percent.

**[0128]** The filaments can be formed into bundles or tow bands, each of which are multiple filaments placed adjacent to each other along their lengths such that the filaments remain untwisted or entangled, or into yarns which can be multiple filaments placed adjacent to each other along their lengths such that the filaments are twisted or entangled. Filament bands are often formed to

allow for effective crimping of the filaments and can be cut into a staple fiber or processed as a continuous band, depending on the end use. As used herein, the term “staple fiber” refers to a filament cut from a filament yarn or tow band that has a discrete length, which is typically less than 150 mm, or less than 120 mm, or up to 100 mm, or up to 80 mm, or up to 65 mm, or up to 60 mm, or up to 55 mm. In some embodiments, the staple fibers may be cut to a length of at least: 1, 1.5, 2, 3, 4, 5, 6, 8, 10, 12, 15, 17, 20, 22, 25, 27, 30, 32, or 35 mm and up to 130 mm, or up to 120 mm, or up to 100mm, or up to 80 mm, or up to 65 mm. Any suitable type of cutting device may be used that is capable of cutting the filaments to a desired length without excessively damaging the fibers. Examples of cutting devices can include, but are not limited to, rotary cutters, guillotines, stretch breaking devices, reciprocating blades, and combinations thereof. Once cut, the staple fibers may be baled or otherwise bagged or packaged for subsequent transportation, storage, and/or use. The cut length of the staple fibers may be measured according to ASTM D-5103.

**[0129]** Filament bands and yarns are typically woven or knitted into a fabric article unless first converted into staple to form a thread. Filaments can also be in the form of yarn. The term “yarn”, as used herein, refers to multiple filaments placed adjacent to each other along their lengths, often twisted or entangled together to improve filament cohesiveness and performance, and typically forming a substantially rounded cross section. Yarn can be processed as continuous strands or cut into smaller lengths, depending on the end use. Multiple filaments and bundles may be assembled into a filament yarn, such as a crimped or uncrimped tow band. As used herein, a “filament yarn” or “tow yarn” refers to a yarn formed from a plurality of continuous, untwisted individual filaments. The filament yarn may be of any suitable size and, in some embodiments, may have a total denier of at least 20,000, 25,000, 30,000, 35,000, 40,000, 45,000, 50,000, 75,000, 100,000, 150,000, 200,000, 250,000, 300,000, 350,000, 400,000, 450,000, or 500,000. Alternatively, or in addition, the total denier of the filament yarn can be no more than 5,000,000, 4,500,000, 4,000,000, 3,500,00, 3,000,000, 2,500,000, 2,000,000, 1,500,000, 1,000,000, 900,000, 800,000, 700,000, 600,00, 500,000, 400,000, 350,000, 300,000,

250,000, 200,000, 150,000, 100,000, 95,000, 90,000, 85,000, 80,000, 75,000,  
or 70,000. In one embodiment or in combination of any mentioned  
embodiments, the filament yarns may have a total denier of at least 50,000,  
55,000, 60,000, 65,000, 70,000, 75,000, 80,000, 85,000, 90,000, 95,000,  
5 100,000, 150,000, 200,000, 250,000, 300,000, 350,000, 400,000, 450,000, or  
500,000.

**[0130]** In another aspect of the present invention, a plurality of filaments are  
gathered to form a bundle, which are then gathered to form a band, such as a  
tow band, having from 5 to 500,000 individual filaments in accordance with the  
10 present invention. Alternatively, the band can have from 10 to 50,000; 10 to  
40,000; 10 to 30,000; 10 to 20,000; 10 to 10,000; 10 to 1000; 100 to 50,000;  
100 to 40,000; 100 to 30,000; 100 to 20,000; 100 to 10,000; 100 to 1000; 200  
to 50,000; 200 to 40,000; 200 to 30,000; 200 to 20,000; 200 to 10,000; 200 to  
1000; 1000 to 50,000; 1000 to 40,000; 1000 to 30,000; 1000 to 20,000; 1000  
15 to 10,000; 5000 to 50,000; 5000 to 40,000; 5000 to 30,000; 5000 to 20,000;  
5000 to 10,000; 10,000 to 50,000; 10,000 to 40,000; 10,000 to 30,000; or  
10,000 to 20,000 filaments of the present invention. In certain embodiments,  
the filter tow weight, i.e., the weight of the filaments only, is greater than 1.7  
mg/mm, or greater than 2.0 mg/mm, or 2.2 to 2.5 mg/mm.

**[0131]** Another aspect of the present invention is an article of manufacture  
incorporating at least a portion of the above-described filaments. Articles are  
units produced from standard filaments, yarn, and/or a filament band, including  
other components and additives needed to meet the functional requirements of  
the intended use. Non-limiting examples include, fabrics, textile products, non-  
25 wovens, absorbent products, a pre-wetted sheet, an article of clothing,  
insulation, medical applications such as surgical face masks and gowns, and a  
bandage, medical packaging, filters for organic and inorganic fluidic and  
aerosolized materials (such as automotive, water treatment, sprayed and  
vaporized liquids such as pesticides and herbicides for home and agricultural  
30 applications), filter rods, cigarette filters for both combustible and heat not burn  
applications, electronic tips, vaping devices, and filter, threads and yarns in  
liquid storage reservoirs.

**[0132]** Non-limiting examples of an absorbent sheet includes bedding covers, bath towels, paper towels and wipes, handkerchiefs, napkins, and spill containment tubes. Non-limiting examples of a personal hygiene absorbent article include a sanitary napkins, tampons, bandages, and incontinent containment articles. With respect to the sanitary napkin and bandages, the article may further include a removable adhesive on at least one surface to adhere the article proximal to the wearer or a wearer's garment.

**[0133]** The pre-wetted sheet may further include at least one compound selected from the group consisting of: a fragrance, a cleansing agent, a disinfectant, a skin moisturizing agent, an emulsifier, and a preservative.

**[0134]** Non-limiting examples of a filter medium include an acetate tow band, a smoking device filter plug, a smoking device filter element or yarn or filament bundle located other than at the mouthpiece, a liquid-vaporizing device filter, a water purification filter, an automotive filter, an air purification filter, surgical face masks and gowns, and a blood purification filter. The closed C shaped filaments can cool hot vapors or gases such as would be generated in smoking devices. The closed C shaped filaments can also allow for rapid gas and vapor and liquid movement through the filament while providing physical barrier properties to air particulates, such as would be desirable for automotive filters, an air purification filter, surgical face masks and gowns, and a blood purification filter.

**[0135]** In the case the filament is utilized as a filter medium, for ease of description, a smoking article utilizing a filter plug or segment will be described. The term "smoking article" relates to all kinds of smokable products like cigarettes, cigars, cigarillos, and non-combusting products such as heat not burn, e-cigarettes, etc. In these smoking articles, the kind of smokable material used, e.g., tobacco, tobacco derivatives, expanded tobacco, reconstituted tobacco, tobacco substitutes, non-burnable products, etc., as well as mixtures thereof, is not particularly limited. These smoking articles are provided with a filter. For such applications, the fibrous tow plug may be wrapped with the plug wrap such that each end of the filter material remains exposed. The plug wrap can vary, see for example, U.S. Patent No. 4,174,719 to Martin, which is

incorporated herein by reference in its entirety. Typically, the plug wrap is selected from a porous or non-porous paper material. Suitable plug wrap materials are commercially available. Exemplary plug wrap papers ranging in porosity from 1100 CORESTA units to 26000 CORESTA units are available from Schweitzer-Mauduit International as Porowrap 17-M1, 33-M1, 45-M1, 70-M9, 95-M9, 150-M4, 150-M9, 240M9S, 260-M4 and 260-M4T; and from Miquel-y-Costas as 22HP90 and 22HP150. Non-porous plug wrap materials typically exhibit porosities of less than 40 CORESTA units, and often less than 20 CORESTA units. Exemplary non-porous plug wrap papers are available from Olsany Facility (OP Paprina) of the Czech Republic as PW646; Wattenspapier of Austria as FY/33060; Miquel-y-Costas of Spain as 646; and Schweitzer-Mauduit International as MR650 and 180. Plug wrap paper can be coated, particularly on the surface that faces the mixed fibrous bundle, with a layer of a film-forming material. Such a coating can be provided using a suitable polymeric film-forming agent (e.g., ethylcellulose, ethylcellulose mixed with calcium carbonate, nitrocellulose, nitrocellulose mixed with calcium carbonate, or a so-called lip release coating composition of the type commonly employed for cigarette manufacture). Alternatively, a plastic film (e.g., a polypropylene film) can be used as a plug wrap material. For example, non-porous polypropylene materials that are available as ZNA-20 and ZNA-25 from Treofan Germany GmbH & Co. KG can be employed as plug wrap materials. In various embodiments, the plug wrap can be a rapidly disintegrating material such that the biodegradation of the filter element is improved.

**[0136]** If desired, "non-wrapped" filter segments may also be produced. Such segments are produced using the types of techniques generally set forth herein. However, rather than employing a plug wrap that circumscribes the longitudinally extending periphery of the filter material, a somewhat rigid rod is provided, for example, by applying steam to the shaped mixed fibrous bundle. Techniques for commercially manufacturing non-wrapped filter rods are possessed by Essentra PLC, Milton Keys, UK.

**[0137]** Cigarette making operations used in combination with a filter preparation process may be conducted using a conventional automated

cigarette rod making machines. Exemplary cigarette rod making machines are of the type commercially available from Molins PLC or Hauni-Werke Korber & Co. KG. For example, cigarette rod making machines of the type known as MkX (commercially available from Molins PLC) or PROTOS (commercially available from Hauni-Werke Korber & Co. KG) can be employed.

**[0138]** A filter utilizing the closed C shaped filaments may contain one or more particulate additive(s) which are not particularly limited and are those additives normally used in filters for smoking articles. The additives can be in powder (particle diameter of 50 to 150  $\mu\text{m}$ ) or granular form (particle diameter of 150 to 1000  $\mu\text{m}$ ). Examples of suitable particulate additives include flavorants or sorbents, e.g., a menthol solution, activated carbon/charcoal, zeolite, ion exchange resin, magnesium silicate like sepiolite, silica gel, alumina, molecular sieves, carbonaceous polymer resins and diatomaceous earths, or combinations thereof. Also, other additives, such as humectants, can be used. According to certain aspects, the particulate additive comprises or is charcoal/activated carbon.

**[0139]** In preparing the article of manufacture, the article may include natural and synthetic filaments. Desirably, the article includes greater than 5 weight %, or at least 10 weight %, or at least 15 weight %, or at least 20 weight %, or at least 25 weight %, or at least 30 weight % depending on the application, even greater than 50 weight %, or greater than 75 weight %, or at least 80 weight %, or at least 90 weight % of closed C shaped cellulose acetate filaments, based on the weight of the synthetic filaments. In preparing an article of manufacture, the article may include greater than 5 weight %, or at least 10 weight %, or at least 15 weight %, or at least 20 weight %, or at least 25 weight %, or at least 30 weight % or depending on the application, even greater than 50 weight %, or greater than 75 weight %, or at least 80 wt.%, or at least 90 wt.% of closed C shaped cellulose filaments, based on the weight of the natural and synthetic filaments.

**[0140]** In one embodiment or in combination of any mentioned embodiments, any of the above-mentioned articles of manufacture can have filaments with any of the above-mentioned characteristics, including those

embodiments relating to the shape, orientation, and dimensions of the closed C shaped filaments.

**[0141]** The process allows one to consistently produce a high population density of closed C shaped filaments. Thus, there is now also provided a continuous dry spinning process for making cellulose acetate monofilaments in which at least 50%, or at least 60%, or at least 70%, or at least 75%, or at least 80%, or at least 85%, or at least 90% or at least 95% of the cellulose acetate monofilaments produced over an 8 hour period, or over a 24 hour period, or over a 48 hour period, or over a 1 week period, or over a 1 month period, have a closed C shape. The closed C shaped filaments can have any of the additional attributes or features mentioned throughout. In a process of making a filament, bundle, tow band or yarn, the percentage consistency of closed C shaped can be determined by taking a random sample of at least 150 filaments (whether from a tow band, bundle or from a bobbin) each hour over the 8 hour period with each successive sample represented by the production from each successive hour, and observing them under an optical microscope at a 500X magnification, where the fiber sample cross sections for viewing are prepared and viewed under any one of the following methods:

**[0142]** Method A: A small skein of hand tight untwisted filaments (not mechanically compressed and some gaps between the filament cross sections should be present when the cross sections are viewed at 500x) is dipped into a solvent suitable of removing any oily substance or lubricant on the fiber surfaces then set aside to dry. The dried skein is then coated with an epoxy or non-volatile wax. The coated skein is then sliced with a microtome to expose the cross-section of the fibers. The slices are then placed on slide and inserted into the microscope to make the photomicrograph.

**[0143]** Method B: a skein of hand tight untwisted filaments (not mechanically compressed and some gaps between the filament cross sections should be present when the cross sections are viewed at 500x) is inserted through a 1 mm x 2 mm hole of a slide holder and secured, following which the fiber bundle is cut to make it flush to a first surface of a slide holder, the holder is turned over and the strands are cut flush to the second surface, leaving small lengths of the

fiber bundle about as thick as the holder, cut on each end to reveal the cross sections. The slide is then inserted on the optical microscope stage to produce the photomicrograph.

5 **[0144]** If staple fibers are evaluated, Method B should be employed, and a bundle of staple fibers is secured in the holder for cutting on both sides.

**[0145]** The samples under either Method A or B, and all other samples, are viewed under a 500x field of view on an optical photomicrograph. Two screenshots may need to be taken to count for examining the 150 filaments.

10 **[0146]** The process is considered to consistently produce closed C shaped filaments if the target percentage (minimum of 50%) of closed C shaped cross sections in each consecutive hourly sample over an 8 hour period is satisfied.

15 **[0147]** There is also provided any of the above-mentioned articles such as fabrics, textiles, woven fabrics or textiles, non-woven webs or fabrics or textiles, and filters, also including filaments, bundles, tow bands, bales, staple fibers, slivers, bobbins, rovings, yarns and any filaments or staple fibers used to make the articles, with any one of them having a minimum population density of closed C shaped fibers, optionally with any one or more of the above-mentioned additional characteristics, of at least 50% per 150 fibers of cellulose acetate. With respect to articles other than filaments in production, bundles, tow bands  
20 and yarn, it is not practical to observe and measure every fiber in an article, and therefore, the minimum population density of closed C shaped fibers per 150 cellulose acetate fibers can be measured by obtaining 2 random samples of cellulose acetate fibers from these articles, with each sample containing at least 150 cellulose acetate fibers (whether filaments or staple), counting the  
25 number of closed C shaped fibers under 500x magnification, and obtaining the percentage of closed C shaped fibers from each sample, adding the number of closed C shaped fibers from the two samples, and dividing by the total of 300 fibers, or merely taking an average of the percentages from each sample. In the event that the article contains mixed fibers, such as from a fabric or apparel,  
30 the fibers counted in the denominator to obtain the minimum population density of closed C shaped fibers are only cellulose acetate fibers. This may require multiple photomicrographs across different areas of a sample depending on

how sparsely populated the sample is with cellulose acetate C shaped fibers and is completed for a sample when a count of 150 cellulose acetate fibers having a C shape is obtained. If the article has two or more different layers or segments of fabrics, then the two samples should be taken from each layer or segment. In this embodiment, the percentage is measured against cellulose acetate fibers instead of all fiber types. In the event that the article contains mixed polymer types of fibers (e.g., polyester and cellulose acetate), the fibers counted in the denominator to obtain the minimum population density of closed C shaped fibers are only the cellulose acetate fibers. If none of the samples has any C shaped fibers, the article is not deemed to contain C shaped fibers. There is also provided any of the above-mentioned articles such as fabrics, textiles, woven fabrics or textiles, non-woven webs or fabrics or textiles, and filters, also including filaments, bundles, tow bands, bales, staple fibers, slivers, bobbins, rovings, yarns and any filaments or staple fibers used to make the articles, with any one of them having a minimum population density of closed C shaped fibers, optionally with any one or more of the above-mentioned additional characteristics, of at least 50% per 150 fibers of C shaped cellulose acetate, using the same sampling method described above. In this embodiment, the percentage is measured against cellulose acetate fibers having a C shaped instead of all cellulose acetate fibers. In the event that the article contains mixed polymer types of fibers (e.g., polyester and cellulose acetate), or has some cellulose acetate fibers that are C shaped and some that are round, crenulated, Y, or a different shape, the fibers counted in the denominator to obtain the minimum population density of closed C shaped fibers are only the C shaped cellulose acetate fibers.

**[0148]** In one embodiment or in combination of any mentioned embodiments, and whether the percentage is taken against all cellulose acetate fibers or against C shaped fibers, there is provided any of the above mentioned articles, also including bundles and tow bands and yarns and any fibers used to make the articles, having a minimum population density of closed C shaped fiber, optionally with any one or more of the above mentioned additional characteristics, of at least 55%, or at least 60%, or at least 65%, or at least

68%, or at least 70%, or at least 72%, or at least 74%, or at least 75%, or at least 77%, or at least 80%, or at least 82%, or at least 85%, or at least 87%, or at least 90%, or at least 92%, or at least 95%, or at least 97%, in each case per 150 fibers (or per 150 cellulose acetate fibers or 150 C shaped cellulose acetate fibers). As against a population of C shaped fibers, not all C shaped fibers are closed C shaped. A C shaped fiber may not be "closed" as defined above even though it is a fiber spun from the same spinneret orifice as a closed C shaped fiber.

**[0149]** Each of these population density values can be associated with the process of making filaments, any of the characteristics of the fibers mentioned above (e.g., dpf, length, singly opposed, mutually opposed, D2/D1 ratio, aspect ratio, process condition, D shaped spinneret, theta angle, etc.), and articles containing the closed C shaped fibers.

**[0150]** In any of these cases, the fibers can be uncrimped, and may even be crimped and have any of the above-mentioned closed C shaped density values.

**[0151]** While mention was made that the density values of the closed C shaped fibers can be on the loose filaments or staple fibers, or on filaments or staple used to make article, or on a tow band, the density values mentioned can also be obtained from filaments or staple fibers drawn from the article. Alternatively, the population density values mentioned can also include the filaments or staple fibers as present in the article. Such filaments or staple fibers may have been carded, drawn, spun, chopped, compressed, wound, bonded, dry or wet laid, etc. Their form is not limited.

**[0152]** In one embodiment or in combination of any mentioned embodiments, there is provided a smoking device filter, or a cigarette filter, or a filter plug, or a filter element contained in a smoking device, that either:

1. at least 50% of the fibers in a filter cross section are comprised of closed C shaped filaments, or
2. at least 50% of the cellulose acetate fibers in a filter cross section are comprised of closed C shaped filaments, or
3. at least 50% of the C shaped fibers in a filter cross section are comprised of closed C shaped filaments.

**[0153]** In embodiment #3, the measurement is based only on filaments having a C shaped, and other shapes would not be counted toward determining the percentage of closed C shape filaments. For example, in a filter plug having a core of C shaped surrounded by Y shaped filaments, random samples are taken only from the core to determine how many have a closed C shape.

**[0154]** In one embodiment or in combination with any mentioned embodiments, the filter has a pressure drop lower than the same filter having Y-shaped filaments per unit of weight and at the same dpf. Thus, the closed C shaped filaments have the advantage of providing a lower pressure drop per unit weight even though they have a hollow core.

**[0155]** The filaments may further be subjected to secondary treatments as dictated by the filaments end use. Such secondary treatments include filament processing and the addition of photodegradation agents, biodegradation agents, decomposition accelerating agents, and various types of other additives.

**[0156]** Photodegradation agents include pigments including coated or uncoated anatase or rutile titanium dioxide, which may be present alone or in combination with one or more of the augmenting components such as, for example, various types of metals. Other examples of photodegradation agents include benzoin, benzoin alkyl ethers, benzophenone and its derivatives, acetophenone and its derivatives, quinones, thioxanthenes, phthalocyanine and other photosensitizers, ethylene-carbon monoxide copolymer, aromatic ketone-metal salt sensitizers, and combinations thereof.

**[0157]** Biodegradation agents and/or decomposition accelerating agents include salts of oxygen acid of phosphorus, esters of oxygen acid of phosphorus or salts thereof, carbonic acids or salts thereof, oxygen acids of phosphorus, oxygen acids of sulfur, oxygen acids of nitrogen, partial esters or hydrogen salts of these oxygen acids, carbonic acid and its hydrogen salt, sulfonic acids, carboxylic acids, organic acid selected from the group consisting of oxo acids having 2 to 6 carbon atoms per molecule, saturated dicarboxylic acids having 2 to 6 carbon atoms per molecule, and lower alkyl esters of the oxo acids or the saturated dicarboxylic acids with alcohols having from 1 to 4

carbon atoms. Biodegradation agents may also comprise enzymes such as, for example, a lipase, a cellulase, an esterase, and combinations thereof. Other types of biodegradation and decomposition agents can include cellulose phosphate, starch phosphate, calcium secondary phosphate, calcium tertiary phosphate, calcium phosphate hydroxide, glycolic acid, lactic acid, citric acid, tartaric acid, malic acid, oxalic acid, malonic acid, succinic acid, succinic anhydride, glutaric acid, acetic acid, and combinations thereof.

**[0158]** The filaments, and the staple fibers made therefrom, may include a top-coat finish added after crimping to impart certain properties or characteristics to the filaments. Such top-coat finish materials include phosphate salts, sulfate salts, ammonium salts, and combinations thereof. Minor amounts of other components, such as surfactants, anti-static agents, wetting agent, antioxidants, biocides, anti-corrosion agents, pH control agents, emulsifiers, and combinations thereof, may also be present in order to enhance the stability and/or processability of the finish. Any suitable method of applying the finish to the filaments may be used and can include, for example, spraying, wick application, dipping, or use of squeeze, lick, or kiss rollers.

**[0159]** The top-coat finish may be added at one or more points during the formation of the filaments, including, for example, after the crimper, before the cutter, or after the cutter.

**[0160]** In one embodiment or in combination with any mentioned embodiment, there is also provided a process for making a closed C shaped filament includes the steps of: a) providing a cellulose acetate dope; b) extruding the dope through at least one spinneret having at least one D-shaped orifice to form a wet filament; and c) drying the wet filament in an apparatus adapted for removing volatiles from the wet filament to form the closed C shaped filament.

**[0161]** In making the D-shaped spinneret, a spinneret having a substantially circular orifice has a portion or segment that is occluded or blocked by a cord intersecting two circumferential points on the orifice; forming an angle  $\theta$  defined by the angle between two radii,  $R_1$  and  $R_2$ , extending from the center to the two

intersectional points on the circumference. The angle  $\theta$  desirably is greater than 90°, or any of the values described above.

**[0162]** The drying step is performed in a drying apparatus having a top, where the wet filaments enters the apparatus, and a bottom, where the filament is removed, wherein the drying apparatus has a top air flow of from 35 cubic feet per minute (cfm) to 135 cfm and having a top air temperature of from 45°C to 85°C, or 50°C to 80°C, or 65°C to 75°C, optionally also a bottom air flow of from 35 cfm to 135 cfm, or at least 5 cfm less than the top air flow, and a bottom air temperature of from 55°C to 120°C, or 65°C to 115°C, or from 70°C to 100°C, or at least 2°C less than the top air temperature.

**[0163]** In one embodiment or in combination with any of the mentioned embodiments, there is provided a dry spinning process for obtaining consistent or a high population density of closed C shaped filaments having a denier per filament from 1 to 30 by:

1. passing a dope solution through a filter maintained at a temperature from 40° to 62°C to obtain a heated dope solution;
2. extruding the heated dope solution through a D shaped spinneret to produce a filament;
3. drying the filament at a temperature with an air flow of from 35 cfm to 135 cfm and at temperature of from 45°C to less than 100°C, and
4. drawing the filament at a draft ratio of from 40 meters per minute to 700 meters per minute.

**[0164]** In one embodiment or in combination with any of the mentioned embodiments, there is provided a dry spinning process for obtaining consistent or a high population density of closed C shaped filaments having a denier per filament from 1 to 30 by:

1. passing a dope solution through a filter maintained at a temperature from 40° to 62°C, or 45°C to 60°C, to obtain a heated dope solution;
2. extruding the heated dope solution through a D shaped spinneret to produce a filament;

3. drying the filament at a top air flow, a top air temperature, a bottom air flow, and a bottom air temperature, where the bottom air flow is at least 1 cfm lower than the top air flow and the bottom air temperature is at least 1°C lower than the top air temperature; and
4. drawing the filament at a draft ratio of at least 1.05, optionally at a take up rate from 200 meters per minute to 900 meters per minute.

**[0165]** In one embodiment or in combination with any of the mentioned embodiments, there is provided a dry spinning process for obtaining consistent closed C shaped filaments having a denier per filament from 1 to 30 by:

1. passing a dope solution through a filter maintained at a temperature from 40° to 62°C, or 45°C to 60°C, to obtain a heated dope solution;
2. extruding the heated dope solution through a D shaped spinneret to produce a filament;
3. drying the filament at a top air flow from 100 to 125 cfm, a top air temperature from more than 85° to 100°C, a bottom air flow that is at least 1 cfm lower than the top air flow, where the bottom air flow is at least 1 cfm lower than the top air flow and the bottom air temperature is at least 1°C lower than the top air temperature; and
4. drawing the filament at a draft ratio of at least 1.01, or at least 1.05, or at least 1.1, or at least 1.2, optionally at a take up rate from 400 meters per minute to 900 meters per minute.

In each of the above process embodiments, the theta angle can be in any of the ranges expressed above.

## EXPERIMENTAL

**[0166]** In preparing the filaments from the examples below, the filaments were washed with ether solvent to remove the spin finish. The filaments were then stretched across a frame and epoxied together to form a rigid rod of encapsulated filaments. Twenty-five grams of Electron Microscopy Sciences RTM Epo-Fix low viscosity resin with 3 g of hardener were mixed together. To the mixture was added 0.5 mL of dye mixture (14 g of ORCO.RTM. Orcocil Red B dye in 760 mL of ethanol). The mixture was stirred slowly until it was

homogeneous. A 1.5-mL micro centrifugation tube was filled to 3/4 capacity with the epoxy mixture. A 10 mm thick specimen from a filter rod was cut and placed on top of the epoxy. The filter was allowed to absorb the epoxy and the tube was placed in a tray and left in a controlled laboratory environment for up to 12 hours to allow the epoxy mixture to harden and embed the filter rod specimen. The specimen was removed from the tube by pitching the bottom of the tube with pliers.

**[0167]** The epoxied filaments were cut perpendicular to the filament axis to form a sample having a thickness of 3 microns. The sample was placed endwise on a microscope slide with cover plate and observed and photographed. Image analysis of the specimen was generated by the following technique: the specimen was placed on an Olympus MZ-130x85 motorized microscope stage. Either the 5x or 10x magnification setting was activated. BX61 STREAM Motion system software was opened. The "Define MIA scanning area with stage" function in the software's "Process Manager" was used to identify the top left and bottom right corners of the specimen. Each frame was focused as indicated by the software, the image collection process was run, and the data was saved. The software can be used to produce a single stitched image of the full filter rod cross-section.

#### **Calculations (Test Method ECCF-A-FE-G-MIC-2-1):**

##### **Specific Surface Area:**

**[0168]** Specific surface area is calculated using the formula:

$$\text{Specific Surface Area} = \text{perimeter} / (\text{area} \times \text{density})$$

where perimeter is in microns, area is in square microns and density is in grams/cubic centimeter.

##### **Circumscribed Radius:**

**[0169]** Circumscribed radius is calculated using the formula:

Circumscribed Radius =  $(\text{breadth}^2 + (\text{length}/2)^2) / (2 \times \text{breadth}) - 1$ ; wherein for a rectangle, the longest feret would be length; and perpendicular to the longest

feret would be breadth. Wherein "feret" refers to a measure in a specific direction of an object.

**Shape Factor:**

5 [0170] Shape factor is calculated by the following formula:  
Shape factor =  $\text{perimeter}^2 / (\text{equivalent circumference}^2) = \text{perimeter}^2 / (4 \times \pi \times \text{area})$ ;

10 where the perimeter is in microns and area is in square microns. The equivalent circumference is the circumference of a circle with the measured filament cross sectional area.

**Denier:**

15 [0171] Denier is a measure of a filament's weight versus length in units of grams per 9000 meters measured according to ASTM D1577-01 using the FAVIMAT vibroscope procedure.

**EXAMPLES 1 AND 2**

20 [0172] Filaments were prepared having a substantially closed-C cross-section configuration. A homogenous cellulose acetate spinning dope solution was prepared containing cellulose acetate having an average degree of substitution of 2.5 - 28.5 wt%; water - 0.5%; titanium dioxide (TiO<sub>2</sub>) - 0.4%; and the remainder being acetone as a solvent. The dope was dry spun under the following conditions:

[0173]

Table 1

Example No.	1	2
Filament dpf	6.7	6.7
Spinneret hole segment angle	132°	140°
Hole diameter equivalent, mm	0.06	0.06
Dope temperature, °C	60	60
Top air flow, cfm	115	115
Top air temperature, °C	92	92
Bottom air flow, cfm	114	114
Bottom air temperature, °C	88	88
Draft ratio	1.09	1.09
Spinning Speed, m/minute	600	600

[0174] One parameter evaluated in this work was the extruded dope temperature. The actual dope temperature can be estimated by monitoring the temperature of the water warming the dope as it flows through the final filtration just before the spinnerets. This temperature is called the candle filter water temperature (CFWT), and the extruded dope is understood to be warmed to this temperature.

[0175] The spinneret hole segment angle is defined as a part of a circle corded off of a straight line, the ends of the cord having circumferential intersection, wherein the angle “ $\theta$ ” is measured from the center of the circle outward to each circumferential intersection. This is more particularly illustrated in Fig. 1. As used herein, the term “spinneret hole segment angle” may be used interchangeably with theta (“ $\theta$ ”) angle, the parameters and characteristics of which are described above. The resulting filaments had a denier per filament (dpf) of 6.7. Figure 3 shows an optical microscope Method A image taken at a magnification of 500x of the cross section of the acetate filaments produced in Example 1, and Figure 4 shows an optical microscope Method A image taken at a magnification of 500x of the cross section of the acetate filaments produced

in Example 2. As can be seen from some of the disfigured filament cross sectional shapes in Figure 4, the increase in the theta relative to the dpf ( $\theta$ :dpf ratio of 21:1) at 60°C starts to affect the density of closed C shaped fibers. While the dope temperature of Example 2 is acceptable at its  $\theta$ :dpf ratio of more than 19:1 at 60°C, further closed C shaped density improvements can be made by lowering the temperature or lowering the  $\theta$ :dpf ratio at the higher end of the temperature range.

### COMPARATIVE EXAMPLES 1 AND 2

[0176] Filaments were made using spinnerets having different segment angles. The homogenous cellulose acetate spinning dope solution of Example 1 was dry spun under the conditions presented in Table 2. All resulting filaments had a dpf of 6.7. The dope was dry spun under the conditions shown in Table 2 below.

Table 2

Comparative Example No.	C1	C2
Filament dpf	6.7	6.7
Spinneret hole segment angle, degree	115	123
Hole diameter equivalent, mm	0.06	0.06
Dope temperature, °C	60	60
Top air flow, cfm	115	115
Top air temperature, °C	92	92
Bottom air flow, cfm	114	114
Bottom air temperature, °C	88	88
Draft ratio	1.09	1.09
Spinning Speed, m/minute	600	600

[0177] Figures 5 and 6 show optical microscope images taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Examples C1 and C2, respectively. It is clear from these images that the spinneret hole segment angle impacts filament cross section formation.

**EXAMPLE 3**

[0178] Filaments were prepared having a substantially closed-C cross-section configuration. A homogenous cellulose acetate spinning dope solution was prepared containing 28.5 weight % cellulose acetate having an average degree of substitution of 2.5, 0.5% water, and the remainder of the spinning solution was acetone as a solvent. The dope was dry spun under the following conditions:

**Table 3**

<b>Example No.</b>	<b>3</b>
Filament dpf	4
Spinneret hole segment angle	105°
Hole diameter equivalent, mm	0.052
Dope temperature, °C	55
Top air flow, cfm	15
Top air temperature, °C	63
Bottom air flow, cfm	12
Bottom air temperature, °C	80
Draft ratio	1.09
Spinning Speed, m/minute	618

[0179] Figure 7 shows an optical microscope image by Method B taken at a magnification of 500x of a high population density population of closed C cross-section shapes of the acetate filaments produced in Example 3.

**COMPARATIVE EXAMPLES C3 AND C4**

[0180] The homogenous cellulose acetate spinning dope solution of Example 3 was used and spun dried under the conditions presented in Table 4.

[0181]

**Table 4**

<b>Comparative Example No.</b>	<b>C3</b>	<b>C4</b>
Filament dpf	4	4
Spinneret hole segment angle	132°	151°
Hole diameter equivalent, mm	0.052	0.052
Dope temperature, °C	55	55
Top air flow, cfm	12	12
Top air temperature, °C	63	63
Bottom air flow, cfm	12	12
Bottom air temperature, °C	80	80
Draft ratio	1.09	1.09
Spinning Speed, m/minute	724	724

[0182] Figure 8 shows an optical microscope image by Method B taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example C3.

[0183] Figure 9 shows an optical microscope image by Method B taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Example C4.

[0184] The images of Comparative Examples C3 and C4 demonstrate that, even at low dope temperatures and temperatures that were the same as Example 3 at 55°C, none of these filaments exhibited the substantially closed, hollow configuration of Example 3. Further, Comparative Example C3 and Example 1 both utilized the same spinneret hole segment angle and yet, at the different dpf values and dope temperatures, yielded different filament cross section shapes as shown in their respective images.

#### **COMPARATIVE EXAMPLES C5-C7**

[0185] Using the spinneret of Example 3, the temperature of the extruded dope was evaluated as a spinning parameter. Filament samples were made from the homogenous cellulose acetate spinning dope solution of Example 3 and dry spun with dope temperatures of 60, 65, 70, and 75 °C and the parameters shown in Table 5.

Table 5

<b>Comparative Example</b>	<b>C5</b>	<b>C6</b>	<b>C7</b>	<b>C8</b>
Filament dpf	4	4	4	4
Spinneret hole segment angle	105°	105°	105°	105°
Hole diameter equivalent, mm	0.052	0.052	0.052	0.052
Dope temperature, °C	60	65	70	75
Top air flow, cfm	15	15	15	15
Top air temperature, °C	63	63	63	63
Bottom air flow, cfm	12	12	12	12
Bottom air temperature, °C	80	80	80	80
Draft ratio	1.09	1.09	1.09	1.09
Spinning Speed, m/minute	618	618	618	618

**[0186]** Figures 10-13 show optical microscope images by Method B taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Examples C5-C8, respectively. As can be seen from Examples 3 and C5-C7, lower dpf values require dope temperatures of less than 60°C to create filament cross-sections that exhibit substantially closed, hollow configuration.

#### **EXAMPLE 4**

**[0187]** Filaments were prepared having a substantially closed-C cross-section configuration. Filament samples were made from the homogenous cellulose acetate spinning dope solution of Example 3 and dry spun with the processing conditions shown in Table 6.

[0188]

**Table 6**

<b>Example No.</b>	<b>4</b>
Filament dpf	4
Spinneret hole segment angle	105°
Hole diameter equivalent, mm	0.052
Dope temperature, °C	50
Top air flow, cfm	15
Top air temperature, °C	63
Bottom air flow, cfm	25
Bottom air temperature, °C	80
Draft ratio	1.09
Spinning Speed, m/minute	618

Figure 14 shows an optical microscope image by Method B taken at a magnification of 500x of high population density population of closed C cross-section shapes of the acetate filaments produced in Example 4.

**COMPARATIVE EXAMPLES C9-C11**

[0189] Using the spinneret of Example 4, the temperature of the extruded dope was evaluated as a spinning parameter. Filament samples were made from the homogenous cellulose acetate spinning dope solution of Example 4 and dry spun with dope temperatures of 60, 65, and 70 °C and the parameters shown in Table 7.

**Table 7**

<b>Comparative Example No.</b>	<b>C9</b>	<b>C10</b>	<b>C11</b>
Filament dpf	4	4	4
Spinneret hole segment angle	105°	105°	105°
Hole diameter equivalent, mm	0.052	0.052	0.052
Dope temperature, °C	60	65	70
Top air flow, cfm	15	15	15
Top air temperature, °C	63	63	63
Bottom air flow, cfm	25	25	25
Bottom air temperature, °C	80	80	80
Draft ratio	1.09	1.09	1.09
Spinning Speed, m/minute	618	618	618

15

[0190] Figures 15-17 show optical microscope images by Method B taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Examples C9-C11, respectively. Comparing the filaments from Example 4 and Comparative Examples C9-C11, when the dpf is lower than 6, such as at 4, dope temperatures of 60°C or more do not result in a product wherein at least 50 percent of the filaments have a closed C cross-section configuration. Although Figure 15 is unsatisfactory, it can be seen that a dope temperature of 60°C is a tipping point since more closed C shaped filaments were made at this temperature than at 65 or 70 °C.

#### EXAMPLE 5

[0191] Filaments were prepared having a substantially closed-C cross-section configuration. Filament samples were made from the homogenous cellulose acetate spinning dope solution of Example 3 and dry spun with the processing conditions shown in Table 8.

**Table 8**

<b>Example No.</b>	<b>5</b>
Filament dpf	4
Spinneret hole segment angle	122°
Hole diameter equivalent, mm	0.052
Dope temperature, °C	50
Top air flow, cfm	15
Top air temperature, °C	63
Bottom air flow, cfm	25
Bottom air temperature, °C	80
Draft ratio	1.09
Spinning Speed, m/minute	618

[0192] Figure 18 shows an optical microscope image by Method B taken at a magnification of 500x of the cross-section of the acetate filaments produced in Example 5. As can be seen at least 90% of the filaments have a closed C configuration.

**COMPARATIVE EXAMPLES C12-C14**

[0193] Using the spinneret of Example 4, the temperature of the extruded dope was evaluated as a spinning parameter. Filament samples were made from the homogenous cellulose acetate spinning dope solution of Example 4 and dry spun with dope temperatures of 60, 65, and 70 °C and the parameters shown in Table 9.

**Table 9**

<b>Comparative Example No.</b>	<b>C12</b>	<b>C13</b>	<b>C14</b>
Filament dpf	4	4	4
Spinneret hole segment angle	122°	122°	122°
Hole diameter equivalent, mm	0.052	0.052	0.052
Dope temperature, °C	60	65	70
Top air flow, cfm	15	15	15
Top air temperature, °C	63	63	63
Bottom air flow, cfm	25	25	25
Bottom air temperature, °C	80	80	80
Draft ratio	1.09	1.09	1.09
Spinning Speed, m/minute	618	618	618

[0194] Figures 19-21 show optical microscope images taken at a magnification of 500x of the cross-section of the acetate filaments produced for Comparative Examples C12-C14, respectively. It is clear from a comparison of Comparative Examples C12-C14 and Example 5 that higher dope temperatures of 60°C or more for the lower dpf filaments create a high number of cross sections that do not exhibit substantially a closed C shaped configuration.

**EXAMPLES 6-9**

[0195] Filament samples were made using the spinneret of Example 1 from the homogenous cellulose acetate spinning dope solution of Example 3. The spinneret hole segment angle was the same as it was in Example 1. The processing conditions were as presented in Table 10 below.

[0196]

**Table 10**

<b>Example Nos.</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>
Filament dpf	6.7	6.7	6.7	6.7
Spinneret hole segment angle	132°	132°	132°	132°
Hole diameter equivalent, mm	0.06	0.06	0.06	0.06
Dope temperature, °C	42	47	52	57
Top air flow, cfm	116	116	116	116
Top air temperature, °C	85	85	85	85
Bottom air flow, cfm	116	116	116	116
Bottom air temperature, °C	95	95	95	95
Draft ratio	1.1	1.1	1.09	1.09
Spinning Speed, m/minute	600	600	600	600

[0197] Figures 22-25 show optical microscope images using Method A taken at a magnification of 500x of the cross section of the acetate filaments produced in Examples 6-9, respectively. The quality of the cross-sectional shape of Example 6, while acceptable, is marginal and begins to suffer as the dope temperature approaches 40°C. Improvements are seen at higher dope temperatures, up to the 60°C dope temperature of Example 1.

#### **COMPARATIVE EXAMPLE C15**

[0198] Filament samples were made using the spinneret of Example 1 from the homogenous cellulose acetate spinning dope solution of Example 3. The spinneret hole segment angle was the same for Comparative Example C15 as it was in Example 1. The processing conditions were as presented in Table 10 below. In Comparative Example C15 the dope temperatures were varied as well as the top air temperature was lower and bottom air temperature was higher from those in Example 1. The processing conditions are shown in Table 11.

[0199]

**Table 11**

<b>Comparative Example No.</b>	<b>C15</b>
Filament dpf	6.7
Spinneret hole segment angle	132°
Hole diameter equivalent, mm	0.06
Dope temperature, °C	62
Top air flow, cfm	116
Top air temperature, °C	85
Bottom air flow, cfm	116
Bottom air temperature, °C	95
Draft ratio	1.08
Spinning Speed, m/minute	600

[0200] Figure 26 shows an optical microscope image by Method A taken at a magnification of 500x of the cross section of the acetate filaments produced in Comparative Example C15. It is clear from a comparison of Comparative Example C15 and Examples 1 and 6-9 that a dope temperature of 62°C generates an unacceptable closed C shaped population density.

**EXAMPLE 10**

[0201] Filament from Example 1 was processed into a tow band and then into filter rods. The filaments were treated with a standard amount of a mineral oil-based tow lubricant and the filaments were crimped at 20 crimps per inch to make 28,000 total denier tow. The tow was converted into filter rods using a standard AF2E plug maker and had the filter rod processing parameters and characteristics shown in Table 12.

**Table 12**

<b>Example No.</b>	<b>10</b>
Triacetin plasticizer, wt. %	7
Triacetin plasticizer weight/rod, mg	43
Rod circumference, mm	24.4
Rod length, mm	102
Non-porous plug wrap thickness, mm	0.037
Paper and glue weight factor, mg	76

### COMPARATIVE EXAMPLES C16 AND C17

5 [0202] Comparative Example 16, which utilized 6.7 dpf “Y” cross-section filament made into 28,000 total denier tow and then converted into filter rods in the same manner as Example 10, had the same filter rod processing parameters and characteristics as presented in Example 10.

10 [0203] Comparative Example C17, utilized a 6.7 dpf regular or cloud cross-section filament made into 28,000 total denier tow and then converted into filter rods in the same manner as Example 10, had the same filter rod processing parameters and characteristics as presented in Example 10.

15 [0204] Filter rods of Example 10 and Comparative Examples C16 and C17 were evaluated for filtration performance as indicated by the three-point capability curve relating pressure drop to filter rod tow weight. The results are shown in Figure 27. In the graph, the filter rods comprised filaments having the cross-sections of: Y are represented by a triangle; regular or cloud shaped are represented by a circle or dot; and a closed-C are represented by a square. All the filaments had a dpf of 6.7. In this graph, it is clear that the tow filaments having the closed-C cross-section gave a lower pressure drop per unit weight that either the regular (round) shaped filaments or the Y shaped filaments.

20

### EXAMPLE 11

25 [0205] Filaments were prepared having a substantially closed-C cross-section configuration. Filament samples were made from the homogenous cellulose acetate spinning dope solution of Example 3 and dry spun with the processing conditions shown in Table 13.

[0206]

**Table 13**

<b>Example No.</b>	<b>11</b>
Filament dpf	8.0
Spinneret hole segment angle	140
Hole diameter equivalent, mm	0.06
Dope temperature, °C	52
Top air flow, cfm	20
Top air temperature, °C	85
Bottom air flow, cfm	58
Bottom air temperature, °C	95
Draft ratio	1.0
Spinning Speed, m/minute	300

[0207] Figure 28 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced in Example 11 using Method B. As can be seen at least 95%, if not at least 98% of the filaments have a closed C configuration.

**EXAMPLE 12**

[0208] Filaments were prepared having a substantially closed-C cross-section configuration. Filament samples were made from the homogeneous cellulose acetate spinning dope solution of Example 3 and dry spun with the processing conditions shown in Table 14, below.

**Table 14**

<b>Example No.</b>	<b>12</b>
Filament dpf	12
Spinneret hole segment angle	148°
Hole diameter equivalent, mm	0.08
Dope temperature, °C	52
Top air flow, cfm	20
Top air temperature, °C	85
Bottom air flow, cfm	58
Bottom air temperature, °C	95
Draft ratio	1.02
Spinning Speed, m/minute	300

[0209] Figure 29 shows an optical microscope image taken at a magnification of 500x of the cross-section of the acetate filaments produced in this Example using Method B. As can be seen in Figure 29, at least 95%, if not at least 98% of the filaments have a closed C configuration.

5

### COMPARATIVE EXAMPLES C18 AND C19

[0210] Comparative filaments were produced using the same dope, dpf, and processing conditions utilized in Example 12, except that different spinneret hole segment angles were used (i.e., 154° and 160°). The operating and processing conditions are provided in Table 15, below.

10

**Table 15**

<b>Comparative Example No.</b>	<b>C18</b>	<b>C19</b>
Filament dpf	12	12
Spinneret hole segment angle	154°	160°
Hole diameter equivalent, mm	0.08	0.08
Dope temperature, °C	52	52
Top air flow, cfm	20	20
Top air temperature, °C	85	85
Bottom air flow, cfm	58	58
Bottom air temperature, °C	95	95
Draft ratio	1.09	1.09
Spinning Speed, m/minute	300	300

[0211] Figures 30 and 31 show optical microscope images taken at a magnification of 500x of the cross-section of the comparative acetate filaments using Method B. As shown in Figures 30 and 31, the comparative filaments of C18 and C19, respectively, consistently produce only “open C” fibers. Thus, C18 and C19 demonstrate that spinneret hole segment angles (theta angles) of 154° or greater consistently fail to produce closed C filaments, as described herein.

15

[0212] Having described the invention in detail, those skilled in the art will appreciate that modifications may be made to the various aspects of the invention without departing from the scope and spirit of the invention disclosed and described herein. It is, therefore, not intended that the scope of the

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invention be limited to the specific embodiments illustrated and described but rather it is intended that the scope of the present invention be determined by the appended claims and their equivalents. Moreover, all patents, patent applications, publications, and literature references presented herein are incorporated by reference in their entirety for any disclosure pertinent to the practice of this invention.

## CLAIMS

What is claimed is:

- 5 1. A process for making closed C shaped filaments comprising a cross-sectional configuration having a first proximal end, a second proximal end, and a hollow core, the process comprising:
- 10 a. providing a cellulose acetate dope comprising cellulose acetate and solvent;
  - b. extruding the cellulose acetate dope through at least one spinneret having at least one D-shaped orifice to form wet filaments; and
  - c. drying the wet filaments in an apparatus adapted for removing solvent from the wet filaments to form the closed C shaped filaments in which -
- 15 A. at least a portion of the first proximal end is either -
- i. oriented toward at least a portion of the second proximal end, or
  - ii. contacting at least a portion of the second proximal end; and
- B. the first proximal end and the second proximal end form either -
- 20 i. a channel defined by a gap between the first proximal end and the second proximal end having a transverse distance  $D1$ , wherein the channel leads from an outer surface of the closed C shaped filaments to the hollow core defined by an inner filament surface and having a diameter  $D2$ , and wherein  $D2/D1 > 1$ , or
  - 25 ii. no channel or passageway resulting from at least a portion the first proximal end contacting at least a portion of the second proximal end.
- 30 2. The process of claim 1, wherein the cellulose acetate dope has a concentration of the cellulose acetate of from 28 weight % to 35 weight %, the cellulose acetate having an average degree of substitution of from 2.2 to 2.8, and

the cellulose acetate dope is at a temperature of 45°C to less than 62°C as measured from a candle filter water temperature (CFWT).

5 3. The process of claim 1, wherein the D-shaped orifice comprises a circular annular open portion defined as a space within an inner circumference of the circular annular portion and a chord, wherein the chord intersects a first point and a second point on the inner circumference, wherein  $\theta$  is the angle determined by and between two radii, ( $R_1$ ) and ( $R_2$ ), extending from an imaginary center and intersecting the first point and the second point, the imaginary center is defined  
10 as the center of an imaginary circle drawn along the circular annular portion and continuing until a complete circle is drawn, and wherein the  $\theta$  is from 90° to 160°.

15 4. The process of claim 1, wherein at least a portion of the first proximal end and the second proximal end are spaced apart at a distance of less than 1.0 radian and are not touching.

20 5. The process of claim 1, wherein at least a portion of the closed C shaped filaments have the first proximal end and the second proximal end in contact with each other.

6. The process of claim 1, wherein at least 30% of the closed C shaped filaments are mutually opposed.

25 7. The process of claim 1, wherein the hollow core has a cross-section area that is at least 25% of the cross-section area of an individual closed C shaped filament.

30 8. The process of claim 1, wherein the hollow core has an aspect ratio (width:height) of not more than 2:1.

9. The process of claim 1, wherein the first proximal end and the second proximal end are co-extensively oriented to each other in an amount of at least 5%.

5 10. The process of claim 1, wherein the cellulose acetate dope comprises less than 5 weight % of a delusterant, a plasticizer, and water, based on the total weight of the cellulose acetate dope.

10 11. The process of claim 1, wherein the cellulose acetate dope contains not more than 4.5 weight % of plasticizer based on the total weight of the cellulose acetate dope.

15 12. The process of claim 1, further comprising drawing at least a portion of the closed C shaped filaments at a draft ratio from 0.9 to 1.6.

13. The process of claim 12, wherein the dpf of a closed C shaped filament spun through the D-shaped orifice at a fixed  $\theta$  angle is changed in an amount of 0.35 to 0.7 units for every 0.1 unit change in draft ratio.

20 14. The process according to any one of claims 1-13, wherein the dpf can be adjusted within  $\pm 3$  full integers for a given D shaped orifice.

25 15. An article comprising cellulose acetate fibers, wherein at least 50% of the cellulose acetate fibers are closed C shaped fibers based on:

- a. 150 cellulose acetate fibers, or
- b. 150 C shaped cellulose acetate fibers.

30 16. The article of claim 15, wherein at least 65% of the cellulose acetate fibers are the closed C shaped fibers.

17. The article of claim 15, wherein the cellulose acetate fibers comprise a cellulose acetate having a degree of substitution (DS) of from 2.2 to 2.8.

5 18. The article of claim 15, wherein the closed C shaped fibers have a first proximal end and a second proximal end that either:

- 10 i. form a channel defined by a gap between the first proximal end and the second proximal end having a transverse distance  $D1$ , wherein the channel leads from an outer surface of the closed C shaped fibers to a hollow core defined by an inner fiber surface and having a diameter  $D2$ , and wherein  $D2/D1 > 1$ , or
- ii. no channel or passageway results from at least a portion the first proximal end contacting at least a portion of the second proximal end.

15 19. The article of claim 15, wherein at least 30% of the closed C shaped fibers are mutually opposed.

20 20. The article of claim 18, wherein the first proximal end and the second proximal end are co-extensively oriented to each other in an amount of at least 5%.

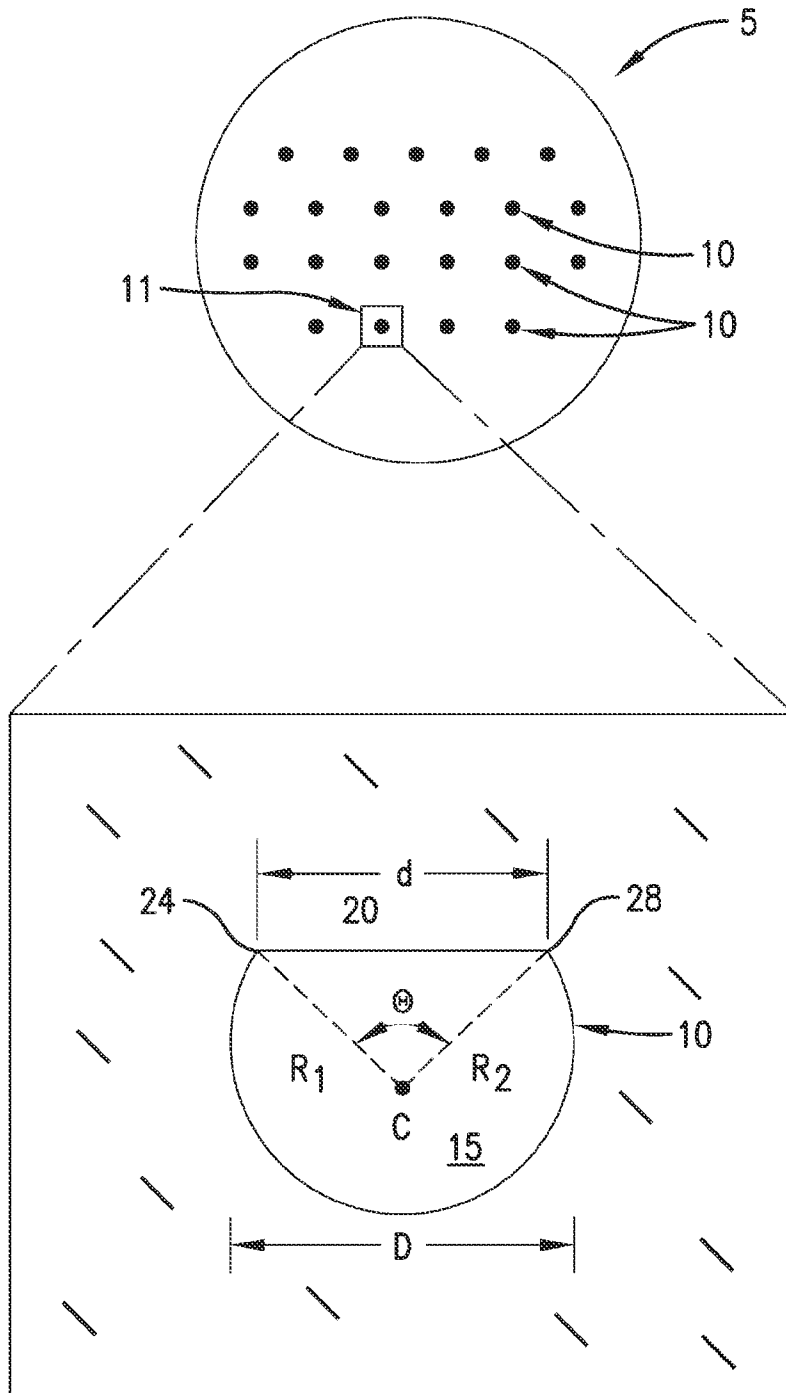
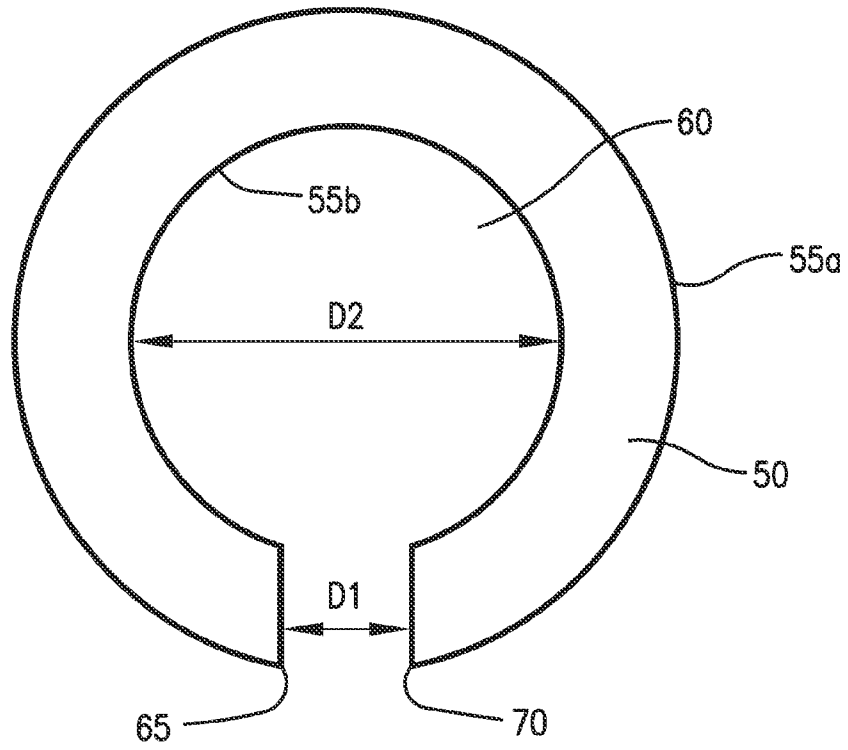
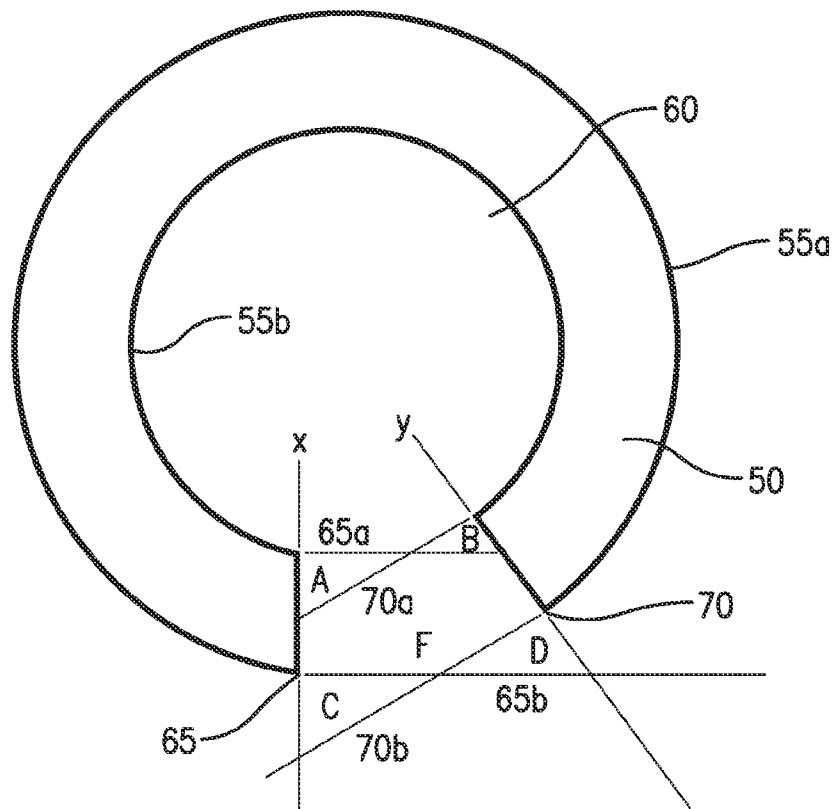


Fig. 1.

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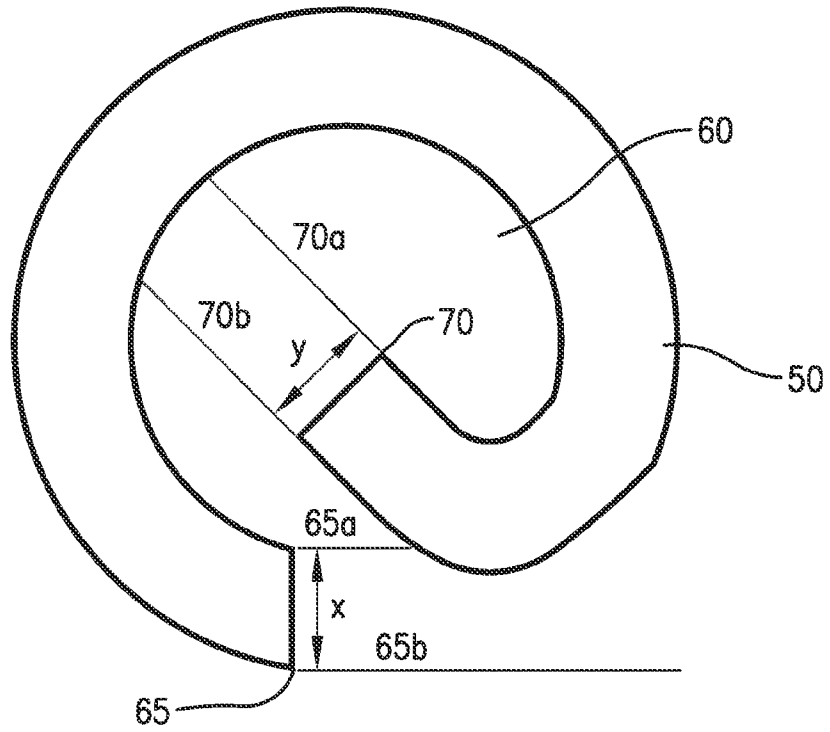


*Fig. 2.*

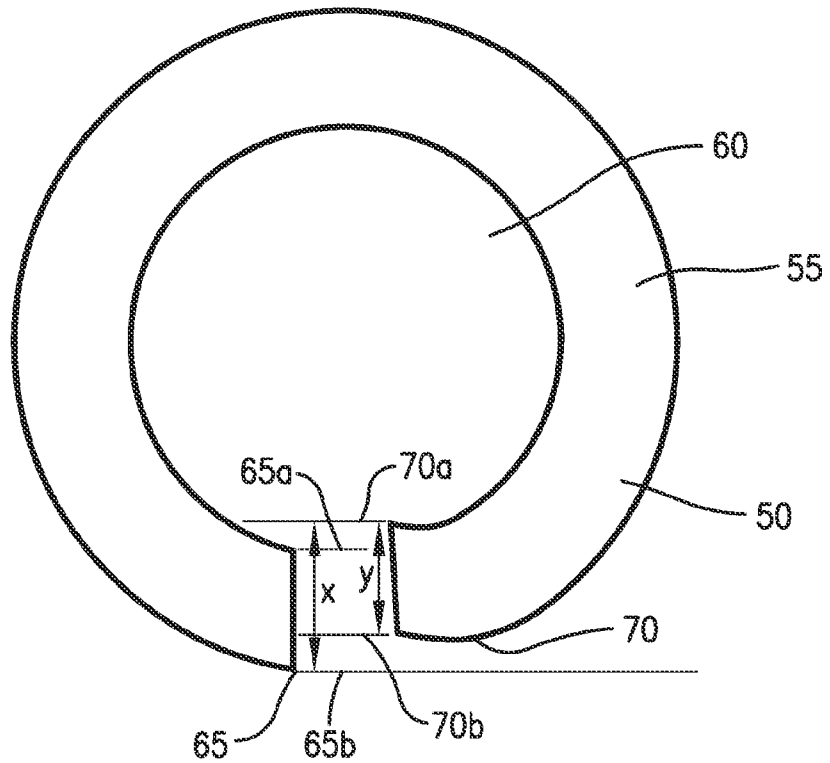


*Fig. 2A.*

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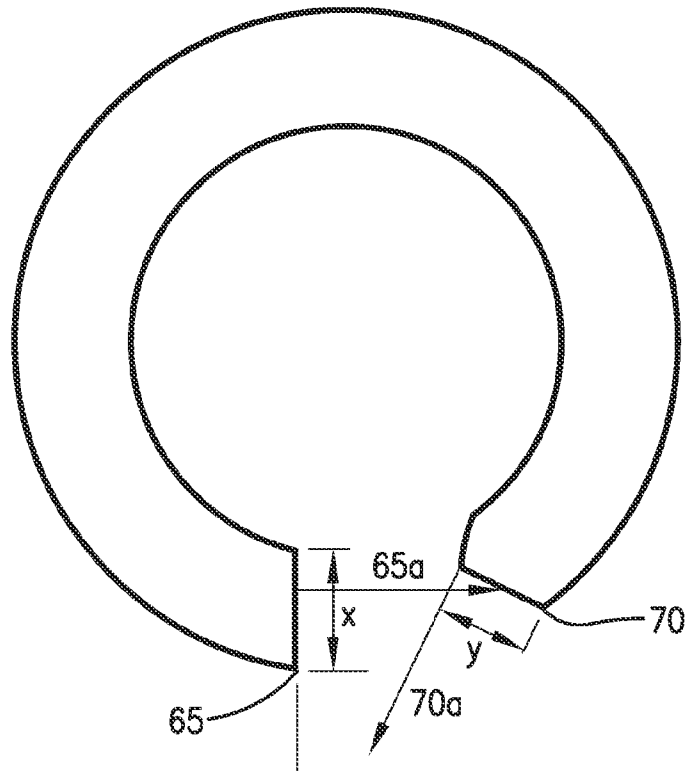


*Fig. 2B.*

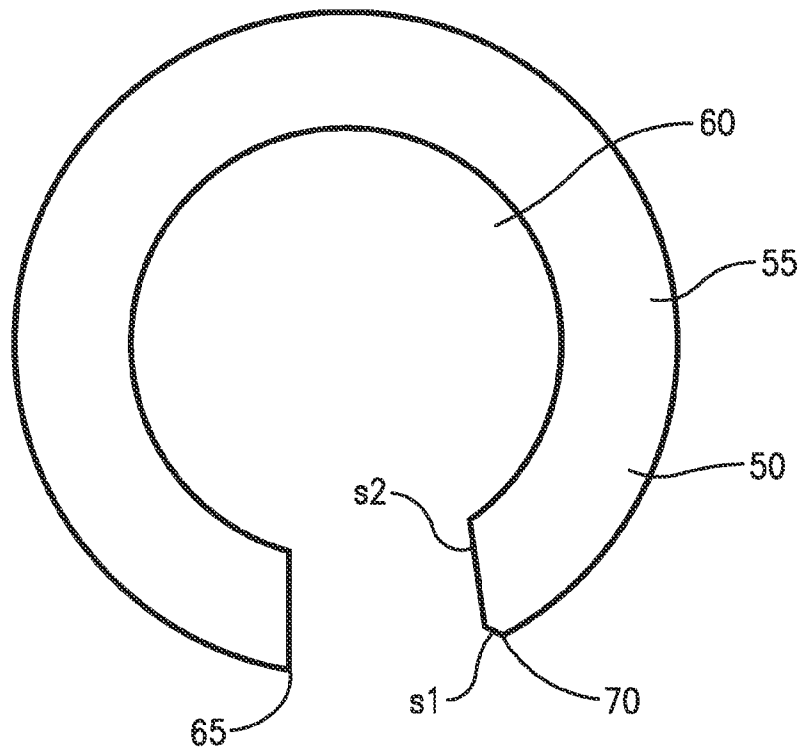


*Fig. 2C.*

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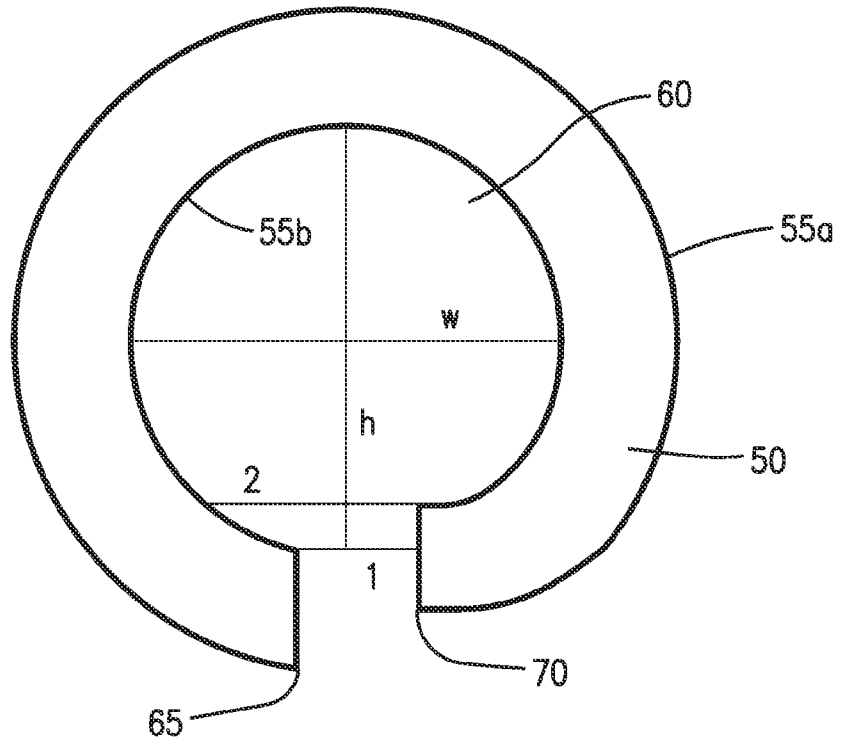


*Fig. 2D.*

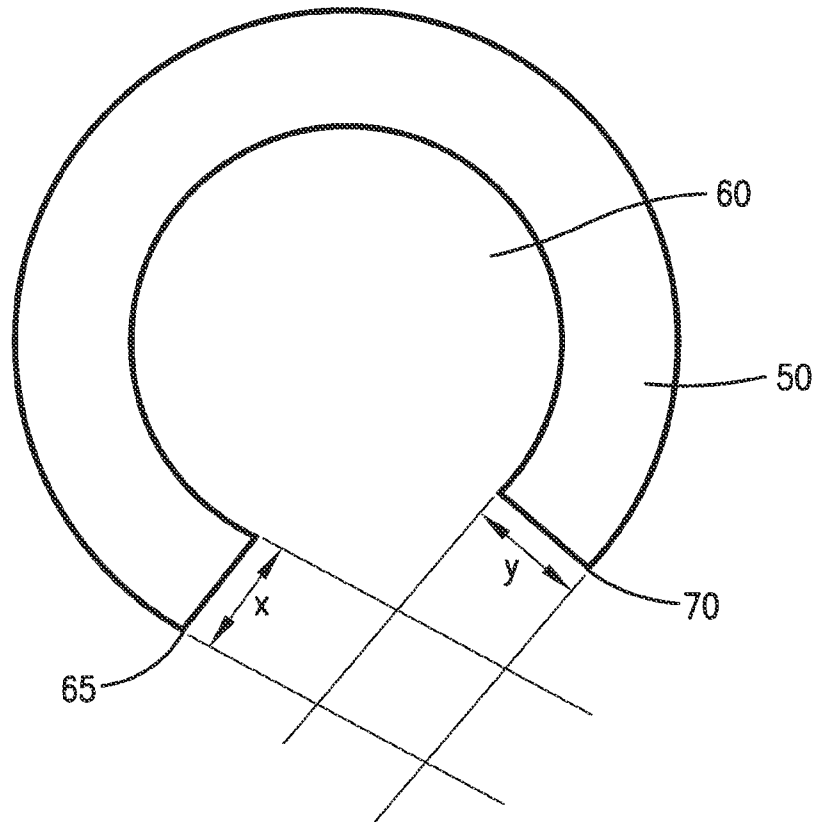


*Fig. 2E.*

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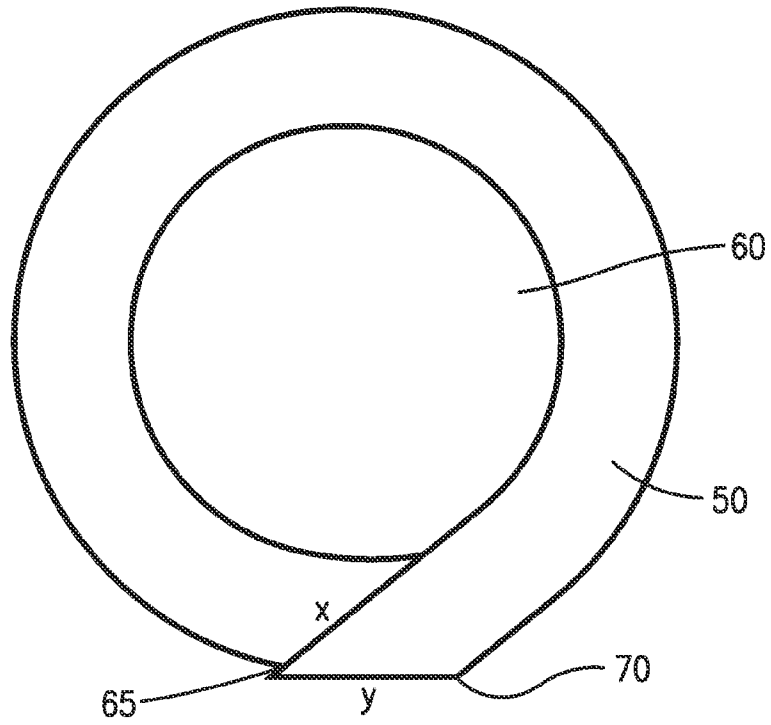


*Fig. 2F.*

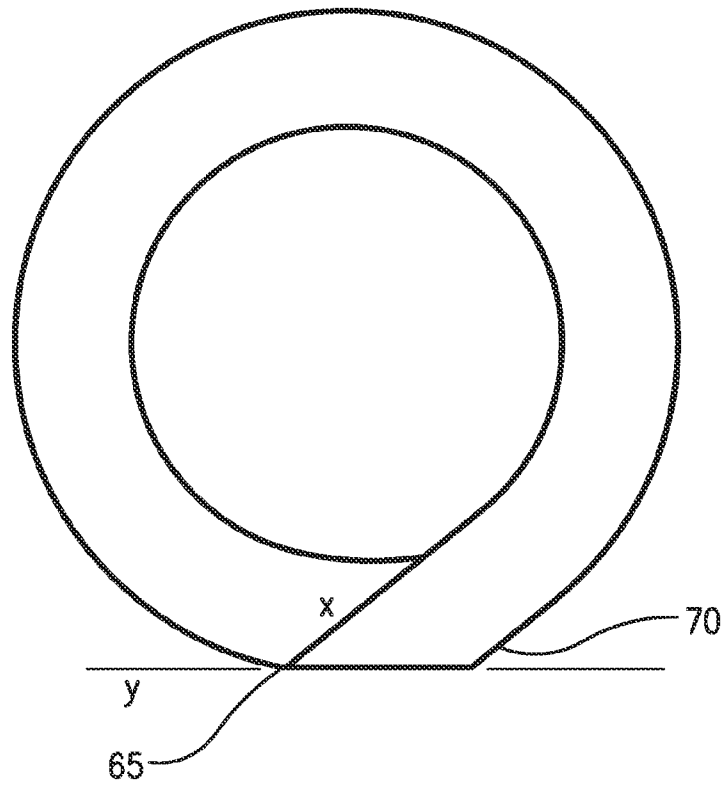


*Fig. 2G.*

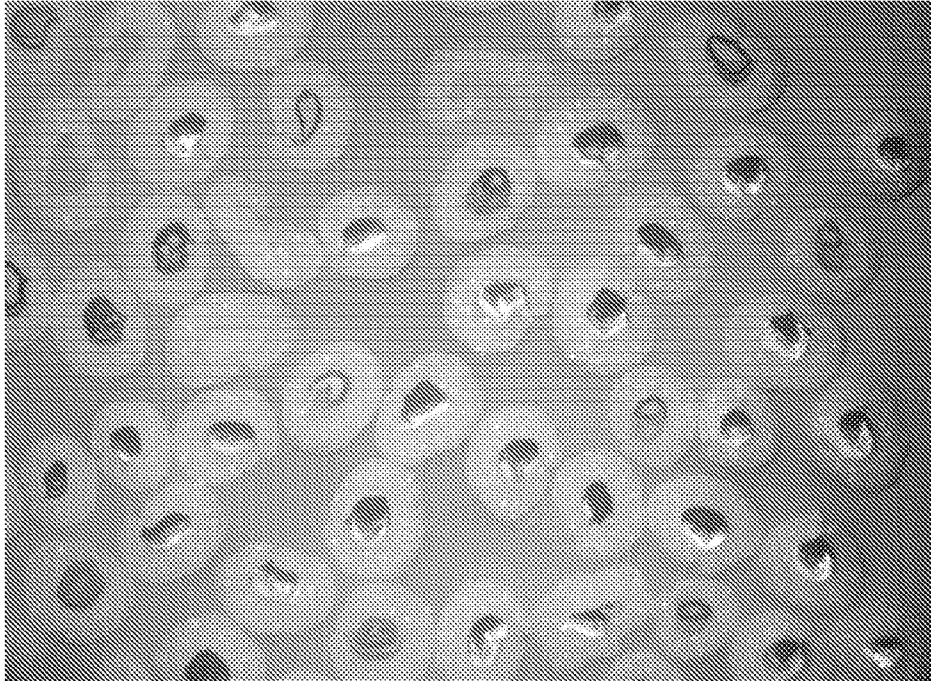
6/23



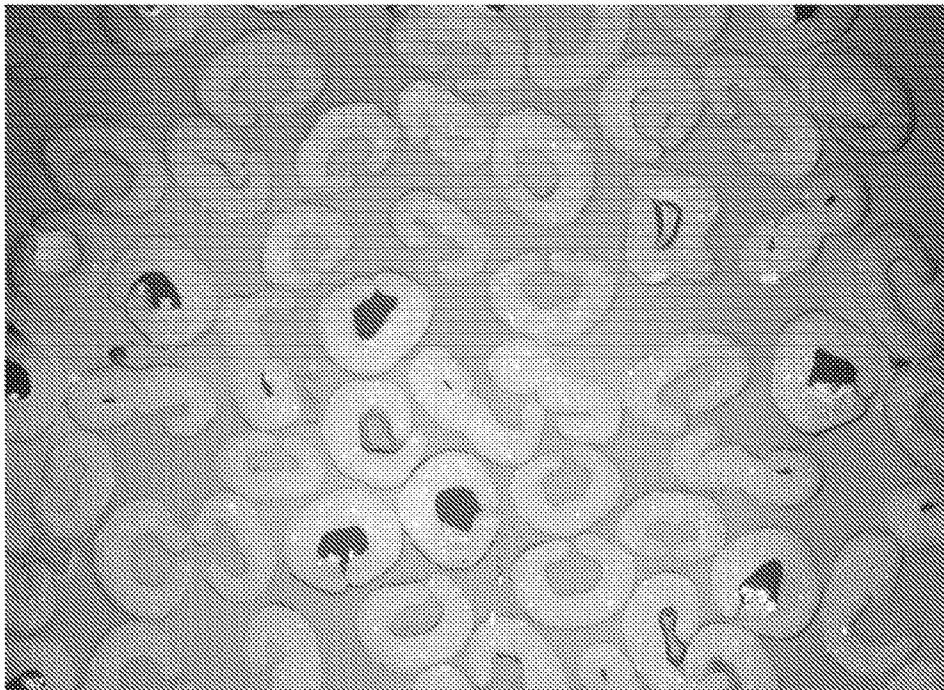
*Fig. 2H.*



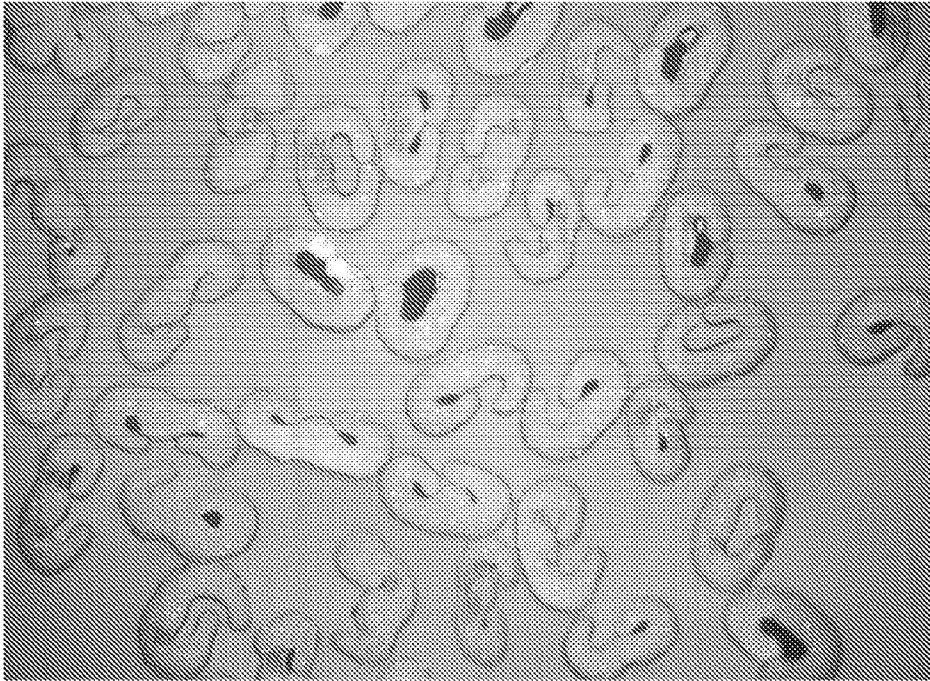
*Fig. 2I.*



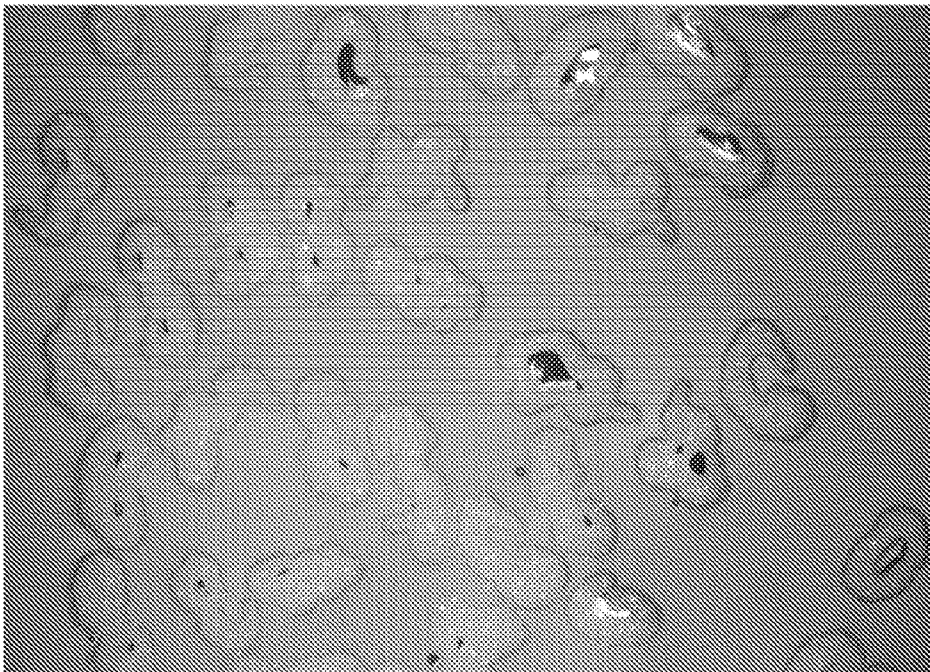
*Fig. 3*



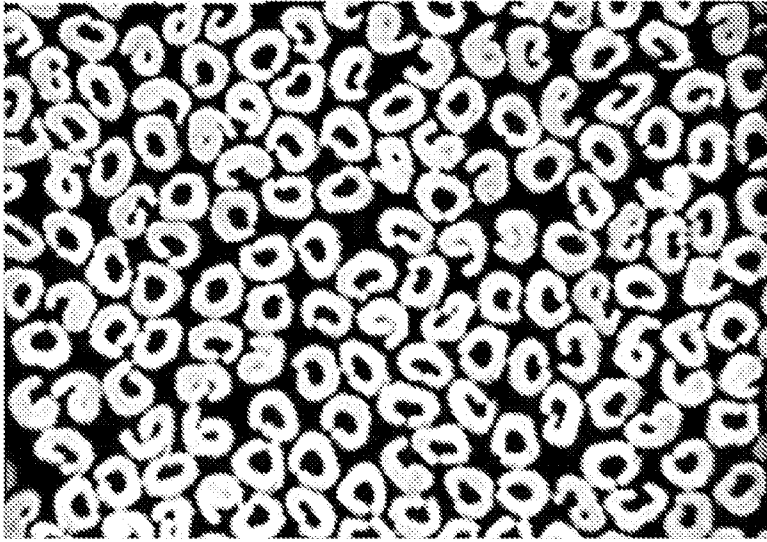
*Fig. 4*



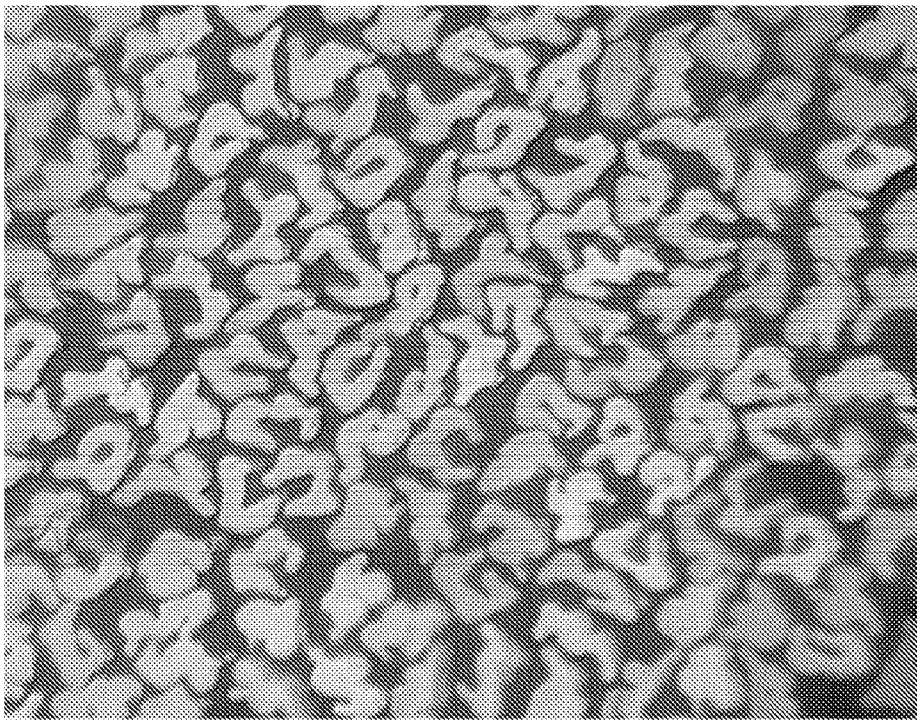
*Fig. 5*



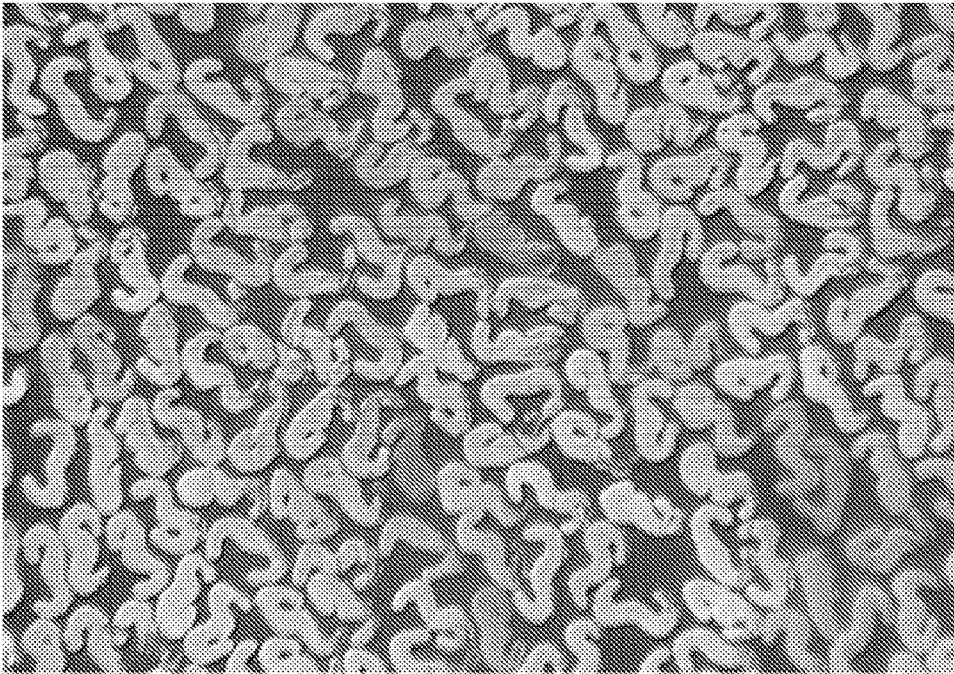
*Fig. 6*



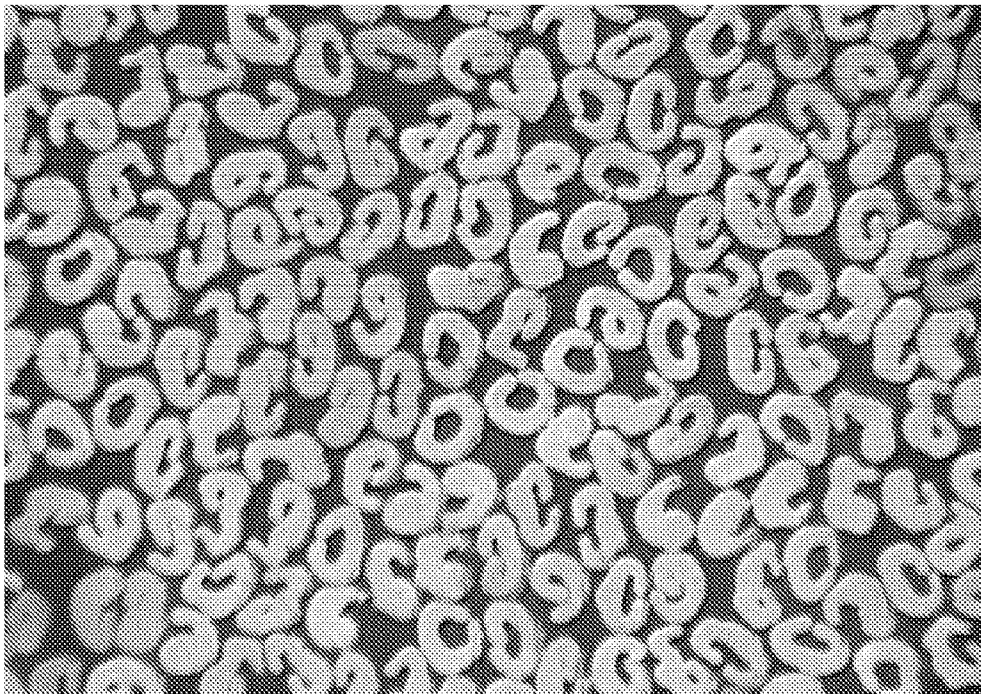
*Fig. 7*



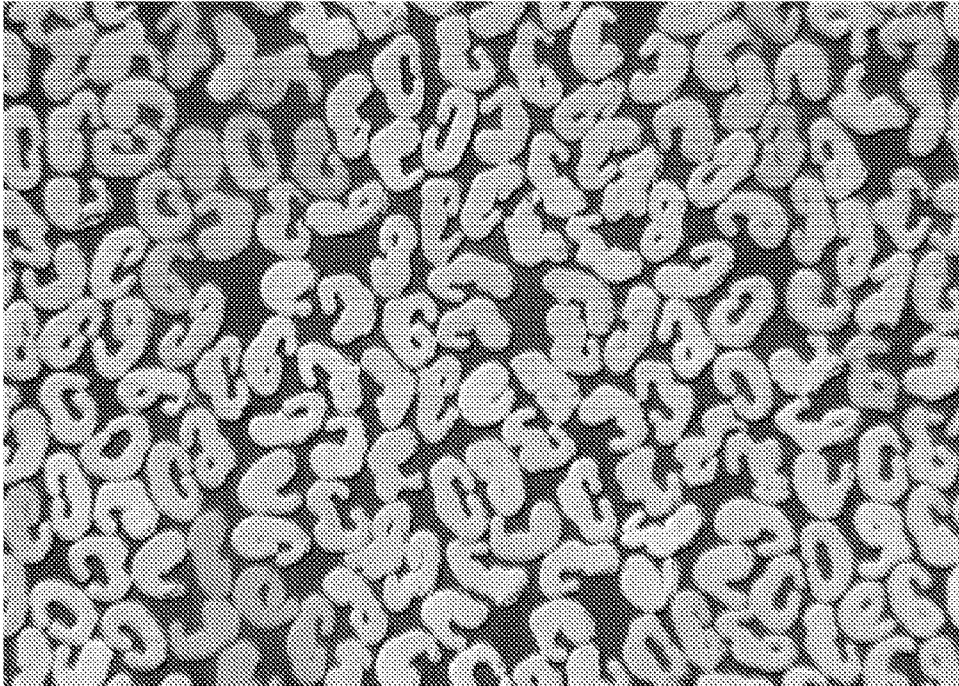
*Fig. 8*



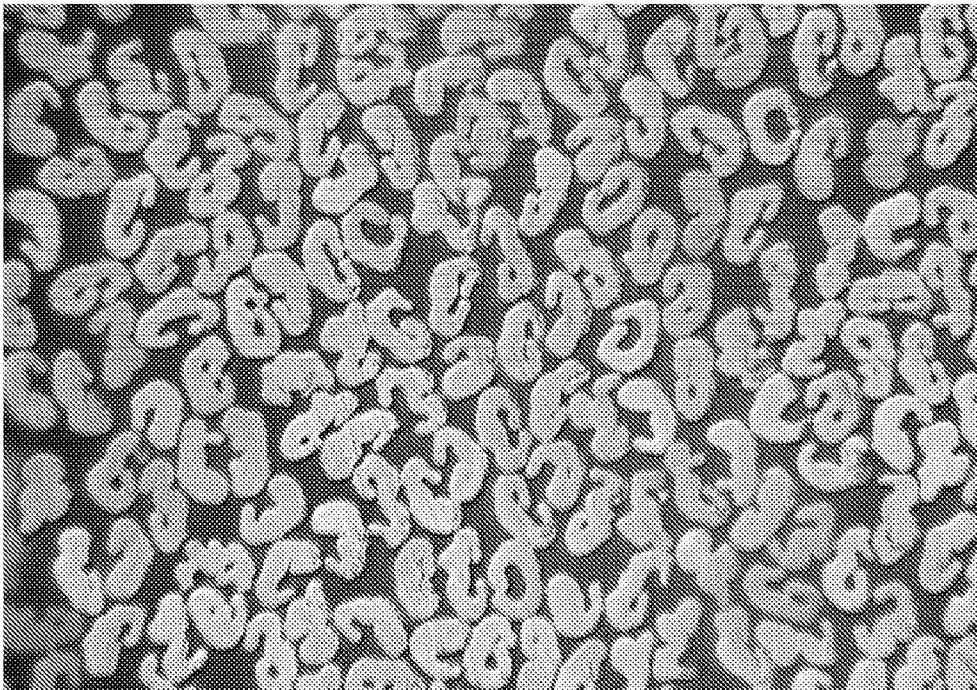
*Fig. 9*



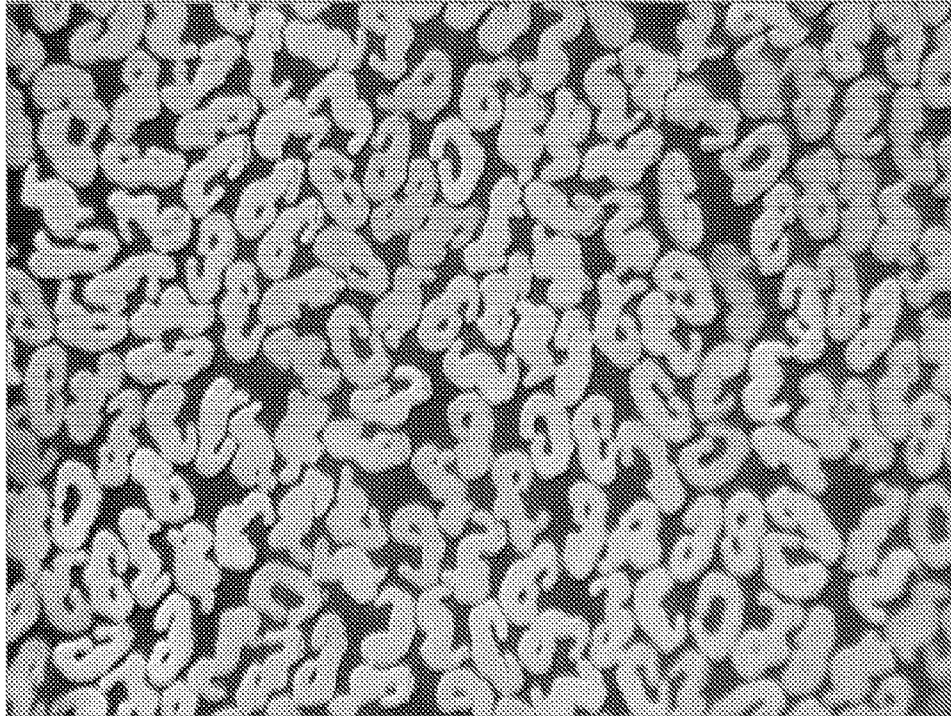
*Fig. 10*



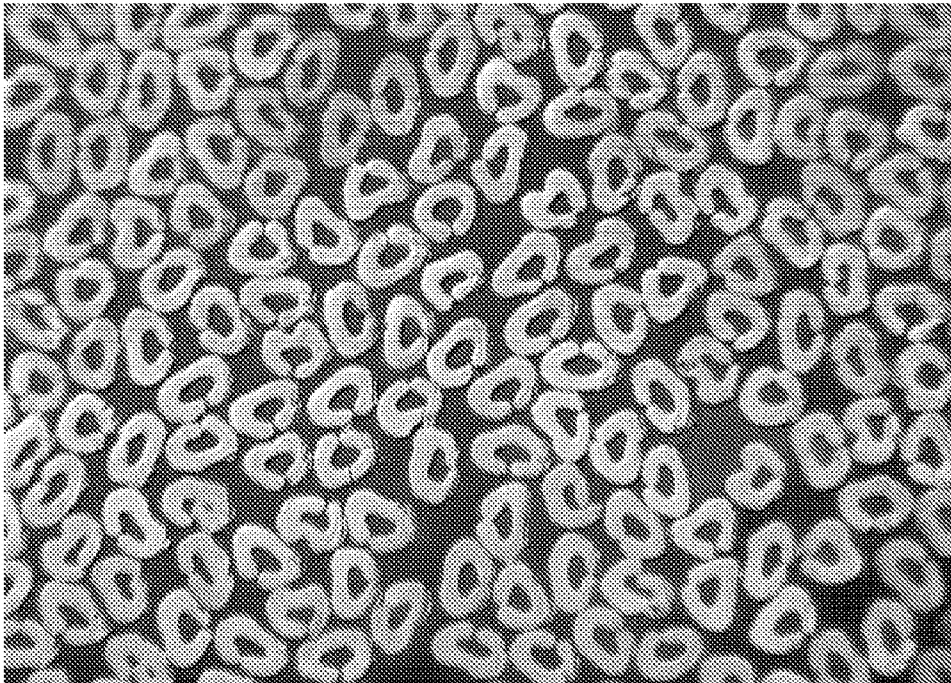
*Fig. 11*



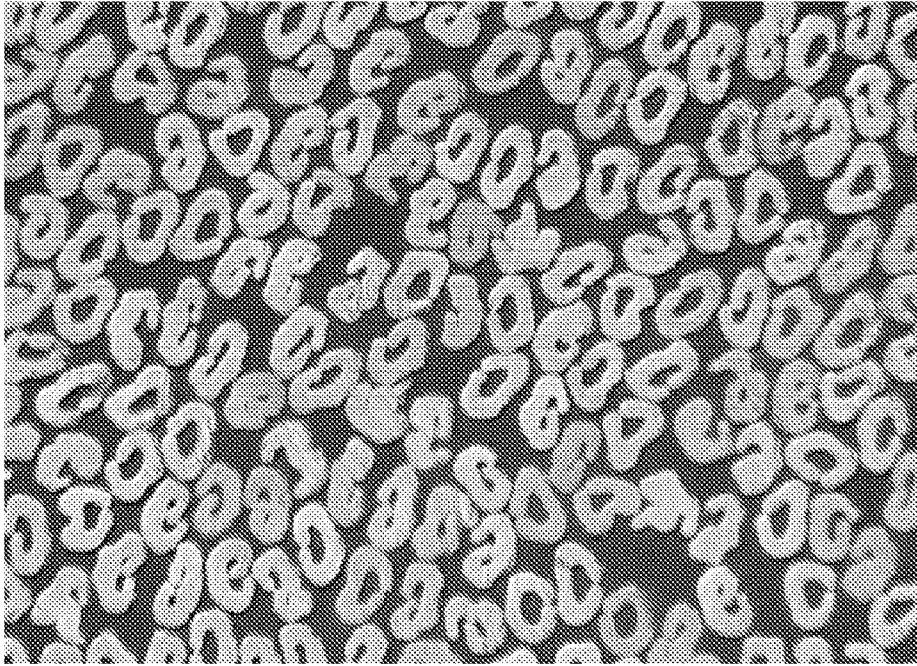
*Fig. 12*



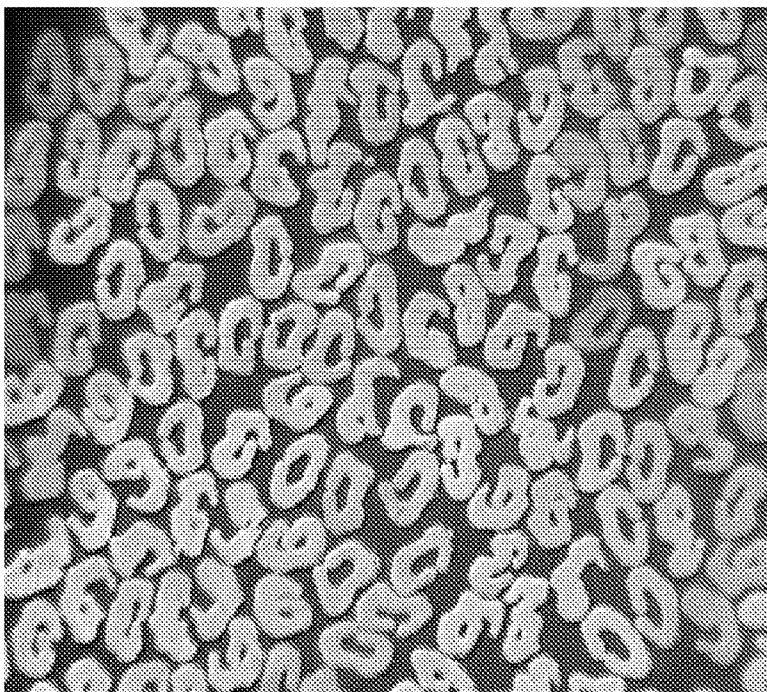
*Fig. 13*



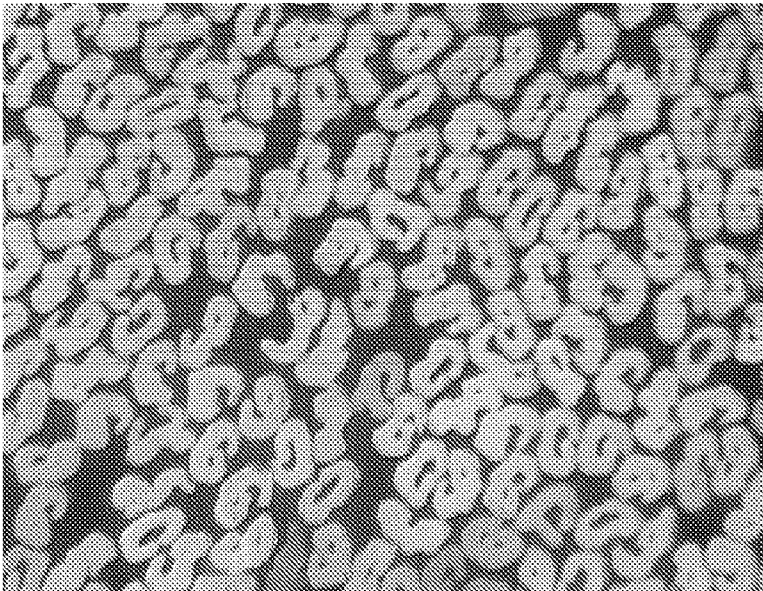
*Fig. 14*



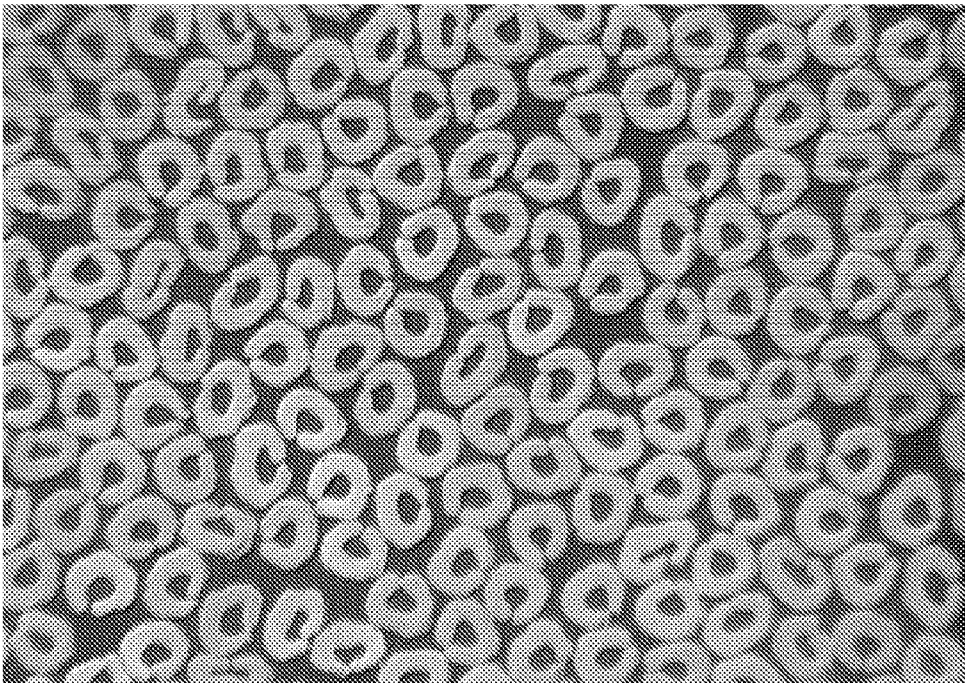
*Fig. 15*



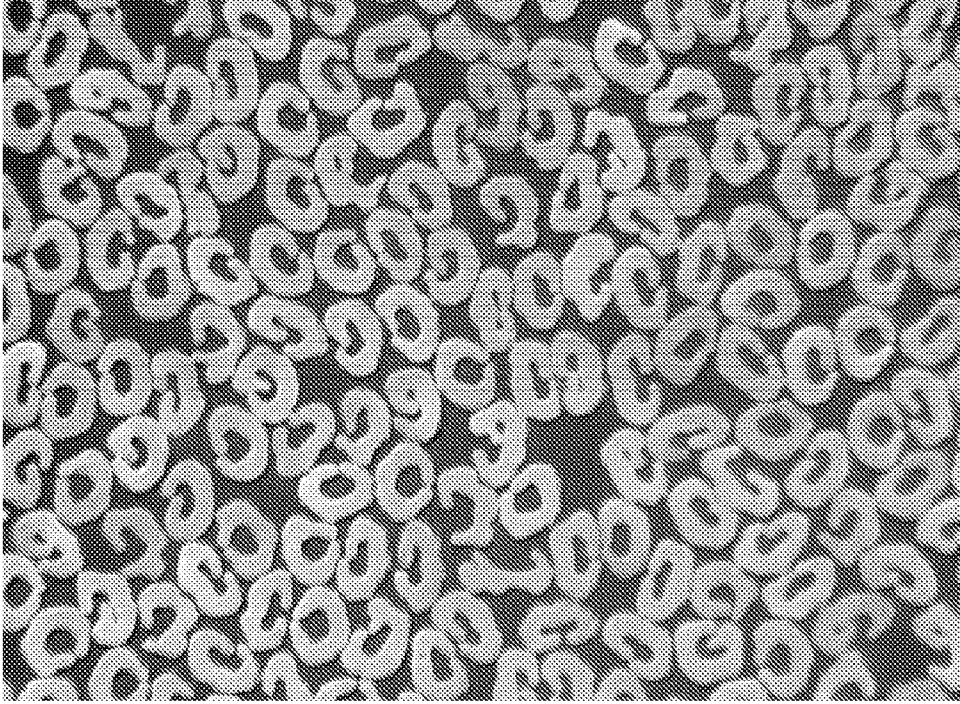
*Fig. 16*



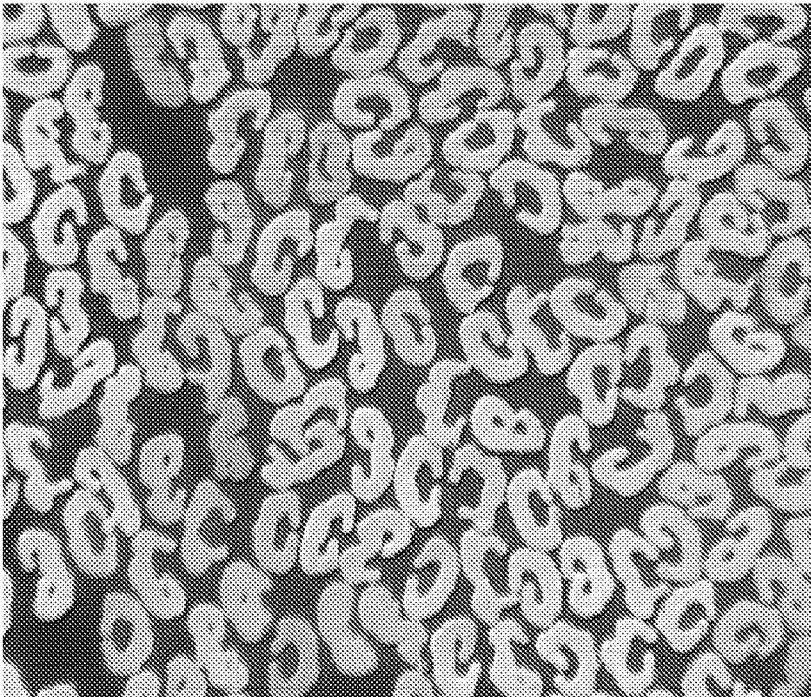
*Fig. 17*



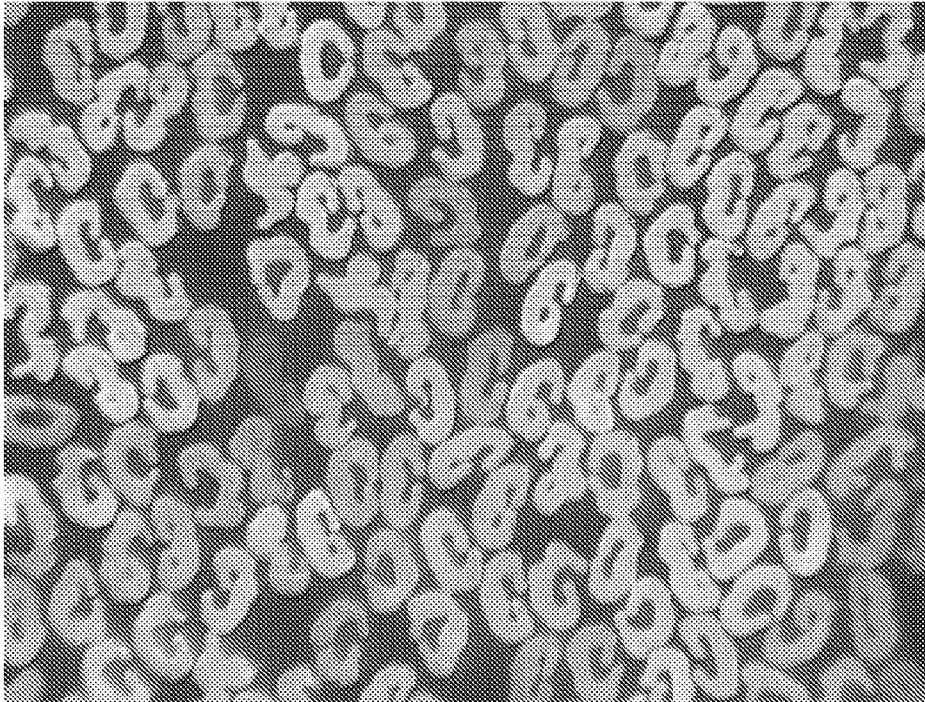
*Fig. 18*



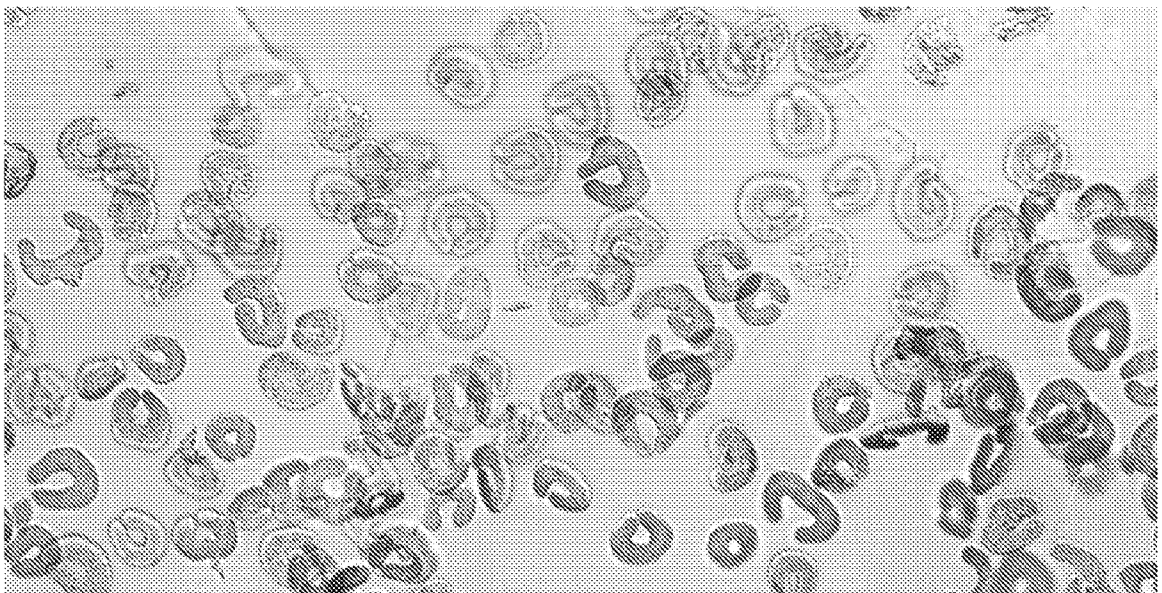
*Fig. 19*



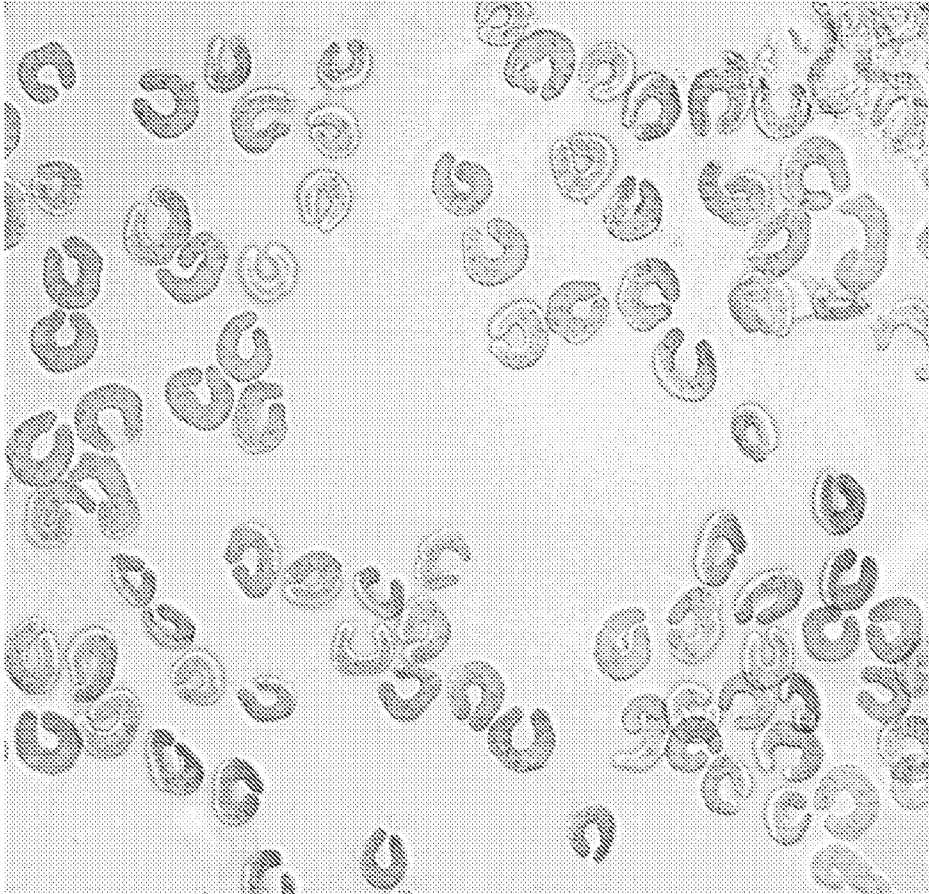
*Fig. 20*



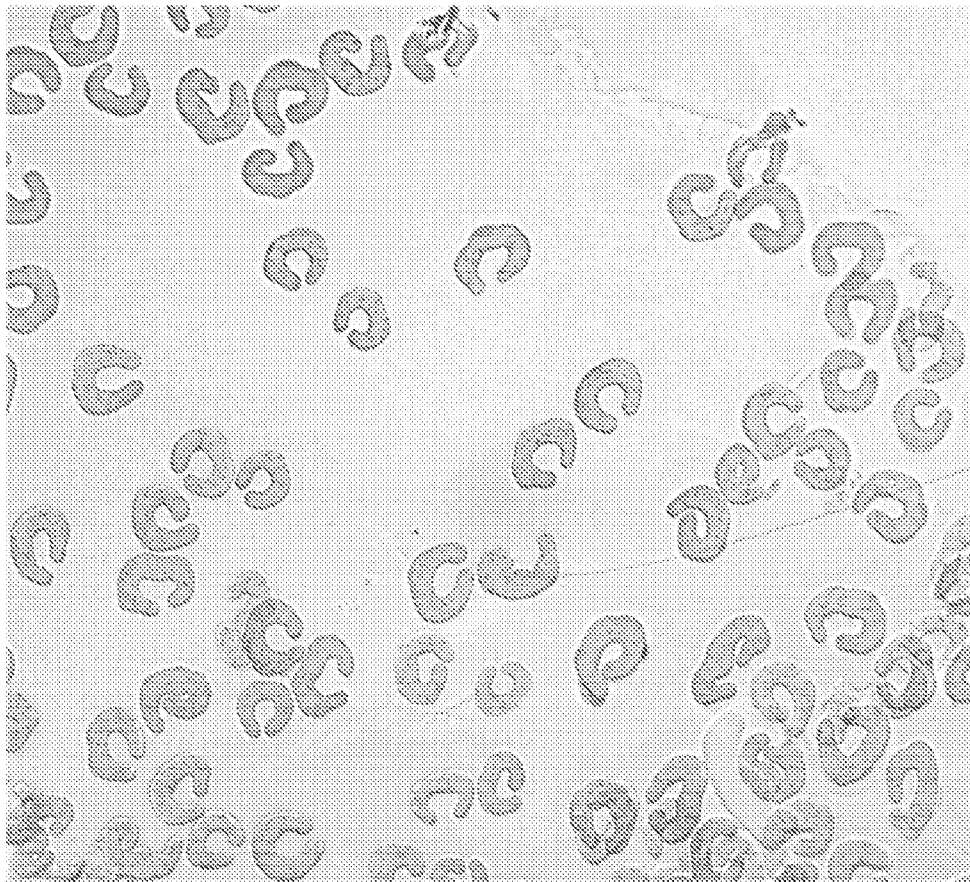
*Fig. 21*



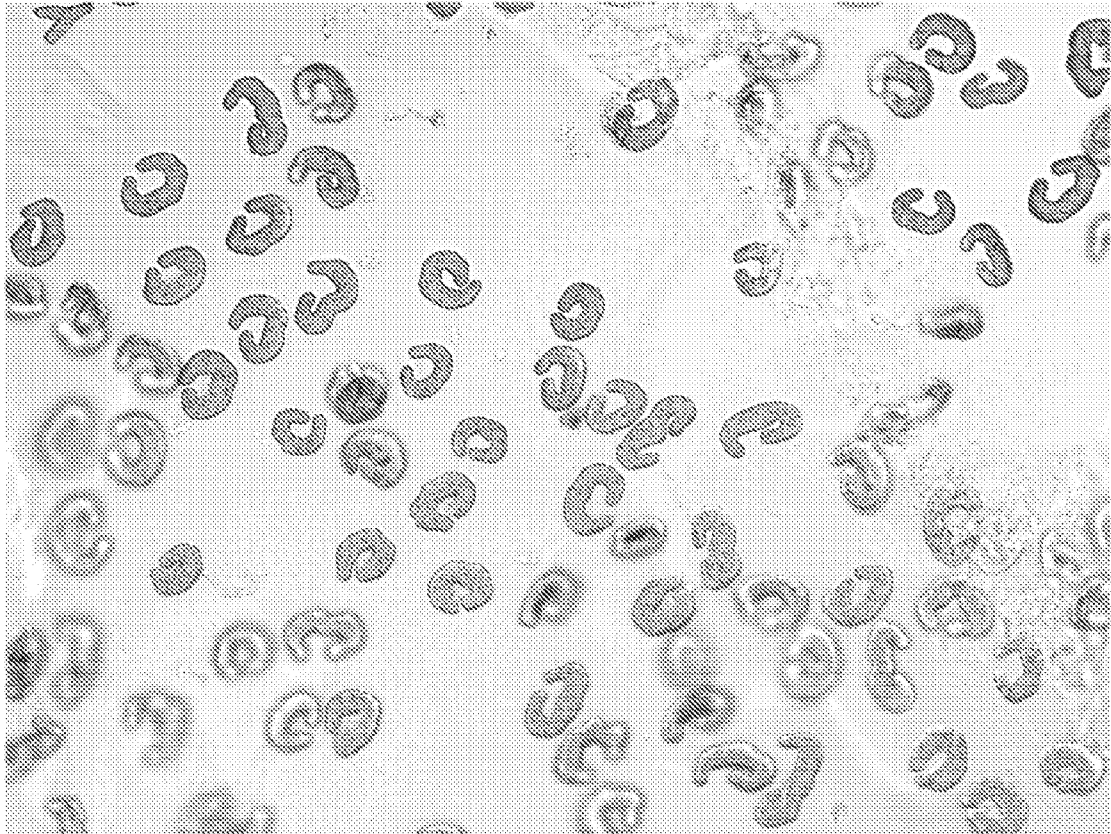
*Fig. 22*



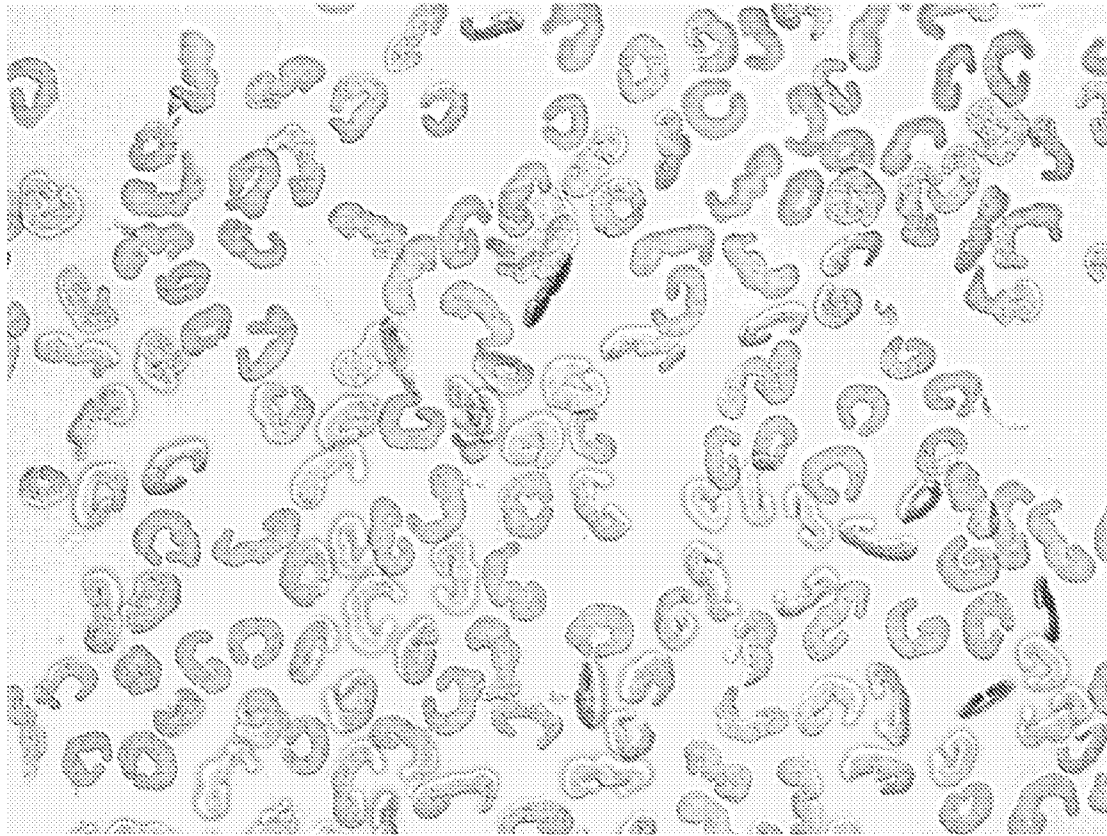
*Fig. 23*



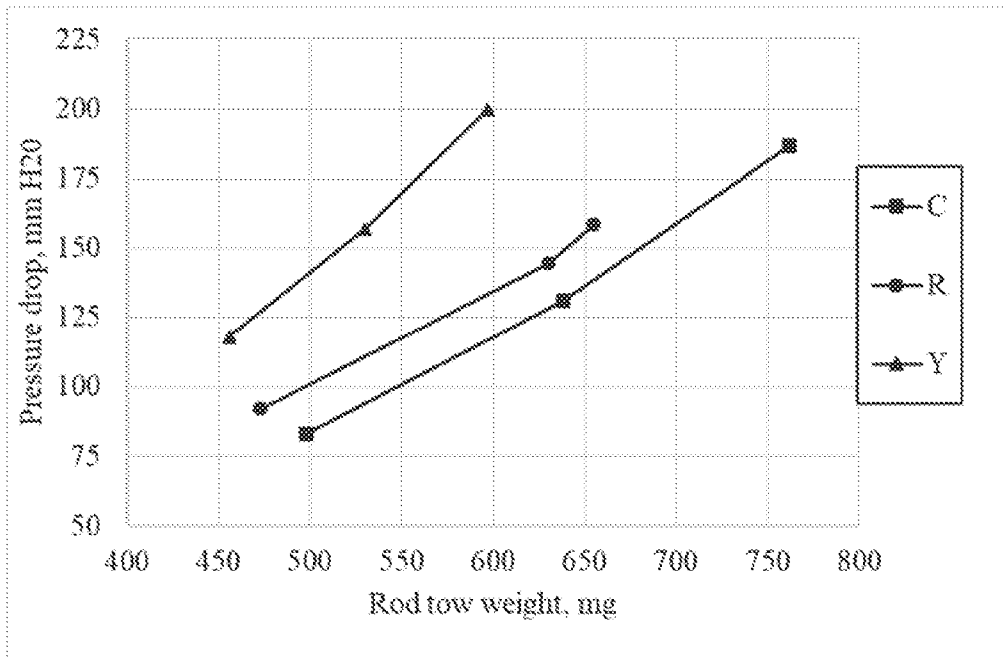
*Fig. 24*



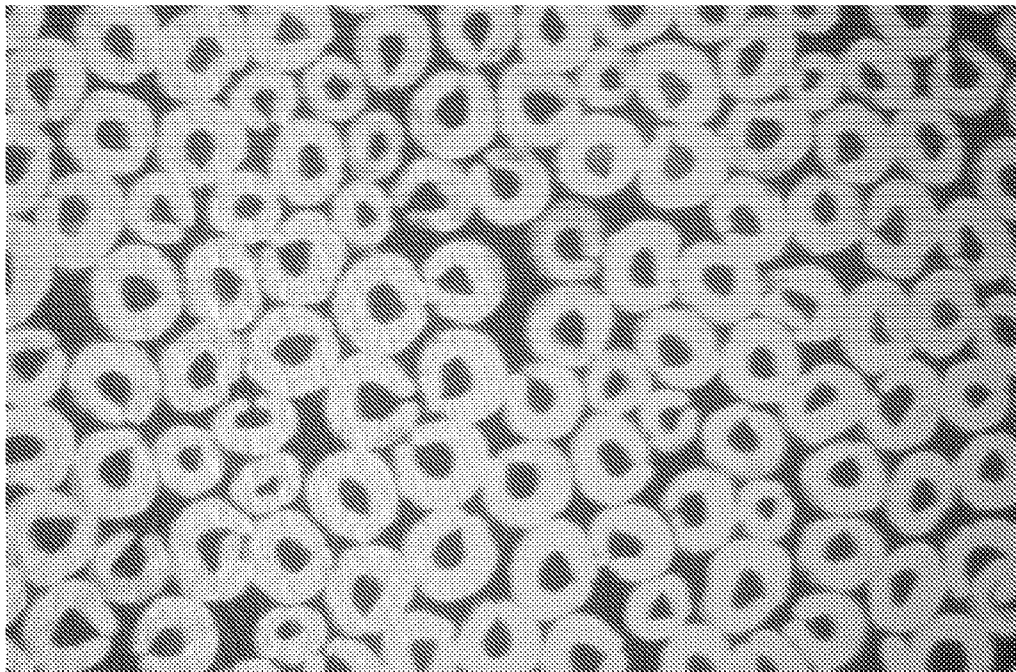
*Fig. 25*



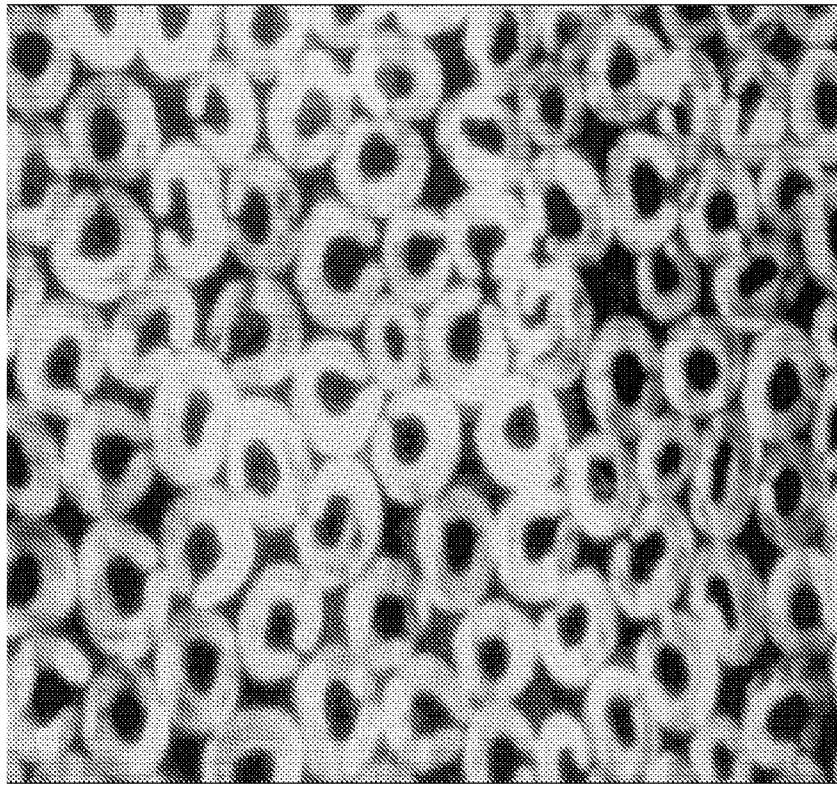
*Fig. 26*



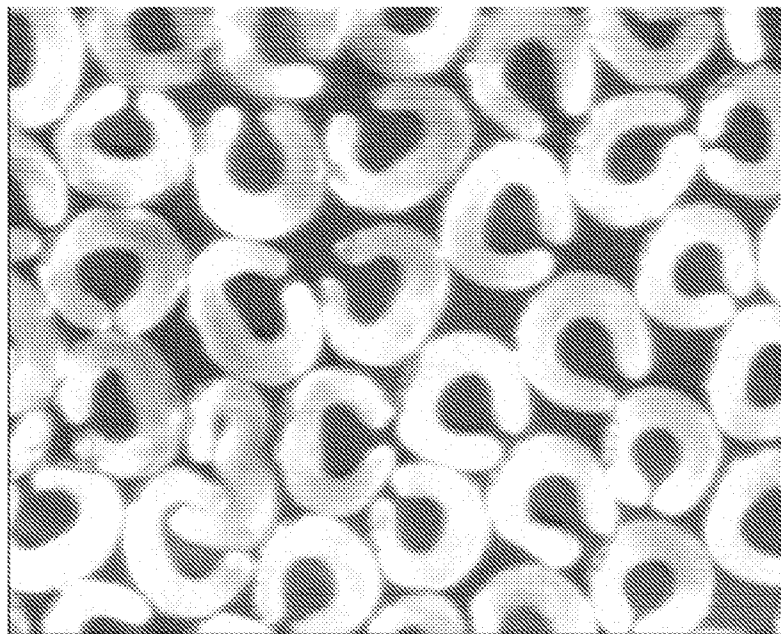
*Fig. 27*



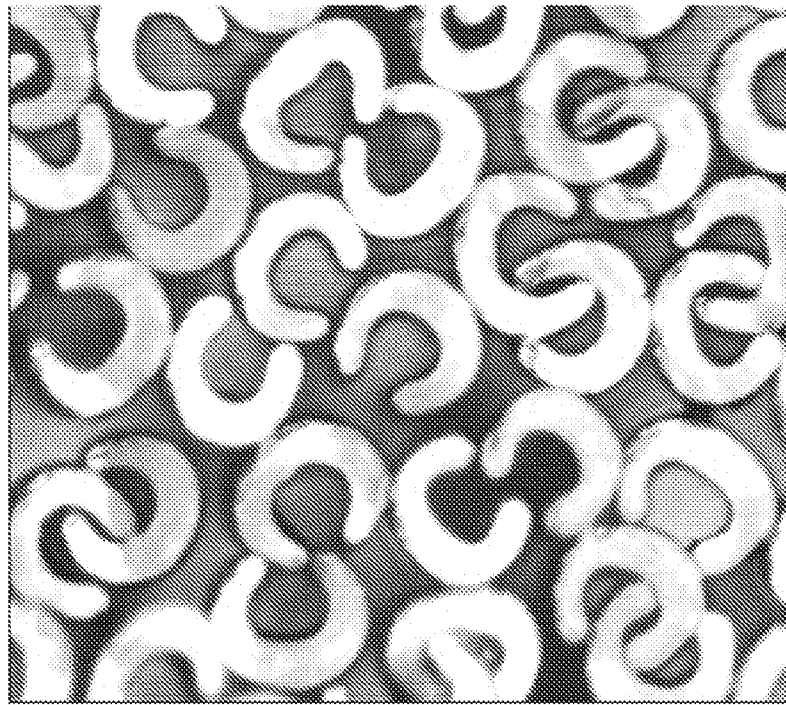
*Fig. 28*



*Fig. 29*



*Fig. 30*



*Fig. 31*

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US2022/034824

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - INV. - D01D 5/24; D01D 5/253 (2022.01)

ADD. - D01D 5/22 (2022.01)

CPC - INV. - D01D 5/24; D01D 5/253 (2022.08)

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

See Search History document

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

See Search History document

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

See Search History document

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US 2020/0002858 A1 (EASTMAN CHEMICAL COMPANY) 02 January 2020 (02.01.2020) entire document	15-17 ----- 2, 8, 11, 18-20
X --- Y	EP 3 821 733 A1 (DAICEL CORPORATION) 19 May 2021 (19.05.2021) entire document	1, 5 ----- 4, 7
Y	GB 880,688 A (EASTMAN KODAK COMPANY) 25 October 1961 (25.10.1961) entire document	1, 2, 6, 8-12
Y	US 5,707,737 A (MORI et al) 13 January 1998 (13.01.1998) entire document	1, 2, 6, 8-12, 18-20
Y	US 2016/0251777 A1 (TORAY CHEMICAL KOREA INC) 01 September 2016 (01.09.2016) entire document	4, 7
A	US 2,838,364 A (SMITH) 10 June 1958 (10.06.1958) entire document	1-20

 Further documents are listed in the continuation of Box C. See patent family annex.

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&amp;" document member of the same patent family

Date of the actual completion of the international search

19 August 2022

Date of mailing of the international search report

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