United States Patent [19]

Ferment et al.

[11] **3,883,718**

[45] **May 13, 1975**

[54] **APPARATUS FOR THERMALLY PROCESSING OF CONTINUOUS LENGTHS OF FIBROUS MATERIALS**

[75] Inventors: **George R. Ferment**, Dover; **Stuart M. French**, Chatham, both of N.J.; **Paul A. Sessa**, Charlotte, N.C.

[73] Assignee: **Celanese Corporation**, New York, N.Y.

[22] Filed: **Jan. 31, 1974**

[21] Appl. No.: **438,303**

[52] **U.S. Cl.** .... **219/388**; 28/61; 34/155; 68/5 D; 219/365; 264/290 R; 264/345

[51] **Int. Cl.** **F27b 9/06**

[58] **Field of Search** .... 28/61, 62; 219/365, 388, 219/400; 260/78.4; 264/288, 290, 345; 34/155; 57/34 HS; 68/5 D, DIG. 1

[56] **References Cited**

UNITED STATES PATENTS

2,823,292	2/1958	Kunzle	219/388 X
3,034,277	5/1962	Richter	219/388 X
3,277,228	10/1966	Killoran et al.	264/290 R
3,303,169	2/1967	Pitzl	264/290 R
3,439,391	4/1969	De Vore et al.	28/1
3,496,332	2/1970	Lunge	219/388 X
3,551,549	12/1970	Sundbeck	264/290 R

FOREIGN PATENTS OR APPLICATIONS

335,501	2/1959	Switzerland	219/388
581,097	8/1958	Italy	219/388

Primary Examiner—Volodymyr Y. Mayewsky

[57] **ABSTRACT**

An improved apparatus is provided for the thermal treatment of continuous lengths of fibrous materials (e.g. drawing, relaxing, drying, etc.) which comprises an elongated heat treatment tube enclosed in a coaxial cylindrical jacket. A heating element may be positioned within the annular space that surrounds the heat treatment tube to impart heat to a fluid introduced into the annular space at the inlet end of the apparatus. This heated fluid may then be directed both to an aspirator at the inlet end of the heat treatment tube and to a gas port which communicates with the inlet end of the apparatus. Valve means are provided for closing the gas port at the start-up of operations to permit the aspirated gas to pull the end of the fibrous material through the heat treatment tube. After start-up the gas port is reopened and the fibrous material preheated before it enters the heat treatment tube. The fibrous material also is heated by radiation from the wall of the heat treatment tube. Optionally, the exit end of the apparatus can be provided with means to recycle a portion of the fluid. The apparatus is particularly suited for the thermal treatment (e.g. hot drawing) of a continuous length of a multifilament polybenzimidazole fibrous material.

7 Claims, 10 Drawing Figures

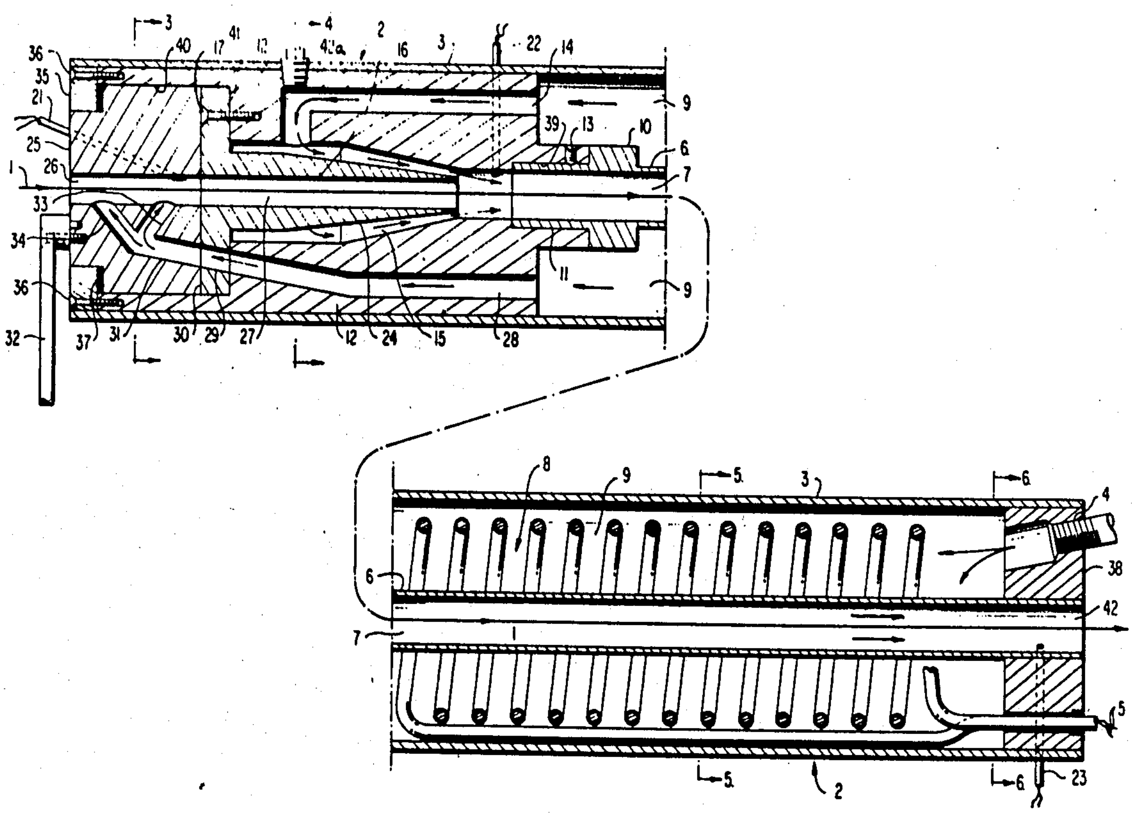


FIG 1

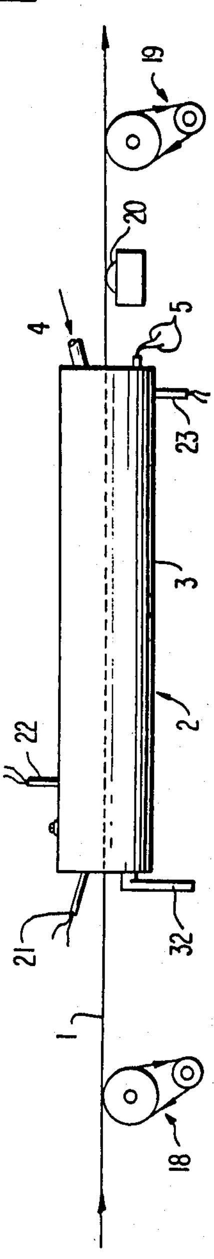


FIG 8

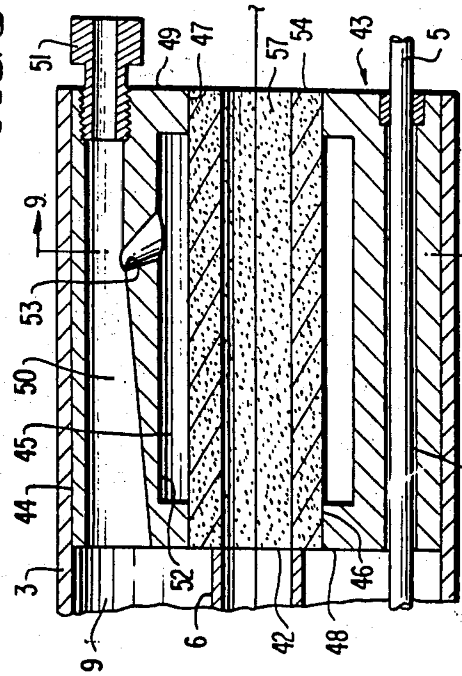


FIG 10

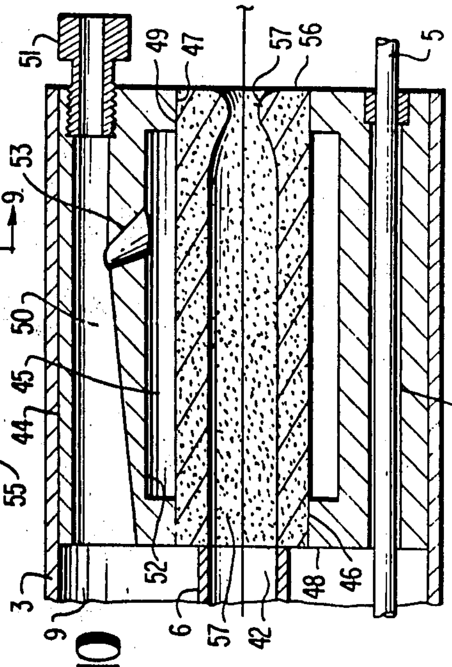


FIG 9

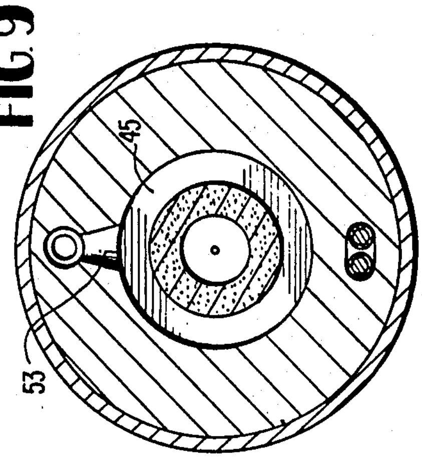


FIG 7

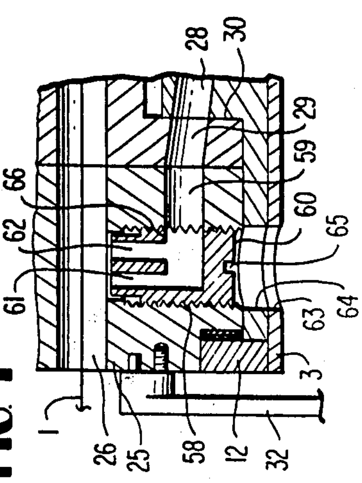


FIG 5

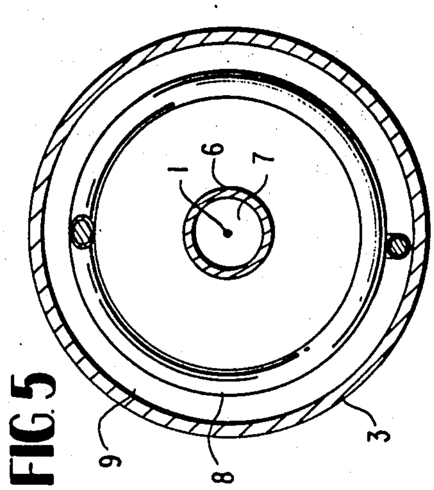
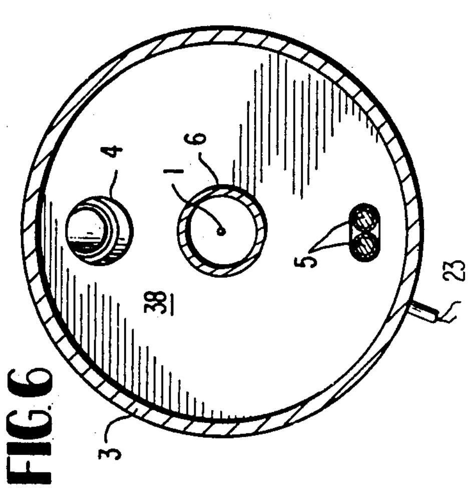


FIG 6



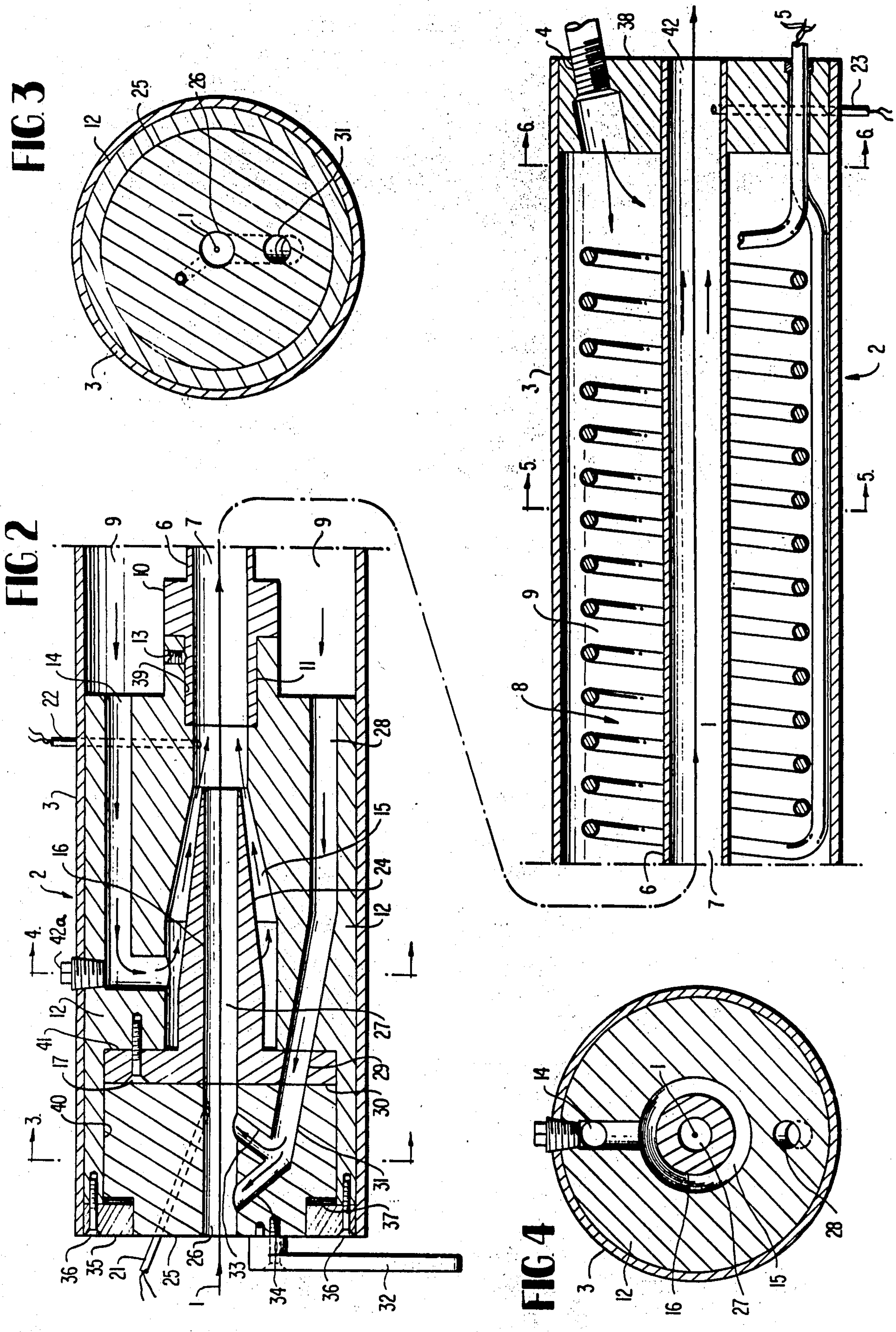


FIG 2

FIG 3

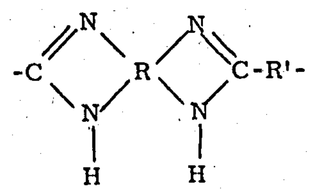
FIG 4

APPARATUS FOR THERMALLY PROCESSING OF CONTINUOUS LENGTHS OF FIBROUS MATERIALS

BACKGROUND OF THE INVENTION

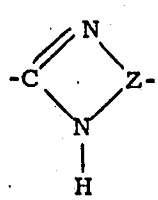
A variety of apparatus and processes have been proposed in the past for the thermal processing of a continuous length of fibrous material on a continuous basis. For instance, a continuous length of synthetic polymeric material (e.g. a polybenzimidazole fibrous material) may be drawn, relaxed, or otherwise thermally treated while passing through the heating zone of an appropriate apparatus.

Polybenzimidazoles are a known class of heterocyclic polymers. Typical polymers of this class and their preparation are more fully described in U.S. Pat. No. 2,895,948, U.S. Pat. No. Re. 26,065, and in the Journal of Polymer Science, Vol. 50, pages 511–539 (1961) which are herein incorporated by reference. The polybenzimidazoles consist essentially of recurring units of the following Formulas I and II. Formula I is:



wherein R is a tetravalent aromatic nucleus, preferably symmetrically substituted, with the nitrogen atoms forming the benzimidazole rings being paired upon adjacent carbon atoms, i.e., ortho carbon atoms, of the aromatic nucleus, and R′ is a member of the class consisting of (1) an aromatic ring, (2) an alkylene group (preferably those having four to eight carbon atoms), and (3) a heterocyclic ring from the class consisting of (a) pyridine, (b) pyrazine, (c) furan, (d) quinoline, (e) thiophene, (f) pyran.

Formula II is:



wherein Z is an aromatic nucleus having the nitrogen atoms forming the benzimidazole ring paired upon adjacent carbon atoms of the aromatic nucleus.

As set forth in U.S. Pat. No. Re. 26,065, the aromatic polybenzimidazoles having the recurring units of Formula II may be prepared by self-condensing a trifunctional aromatic compound containing only a single set of ortho disposed diamino substituents and an aromatic, preferably phenyl, carboxylate ester substituent. Exemplary of polymers of this type is poly-2,5(6)-benzimidazole prepared by the autocondensation of phenyl-3,4-diaminobenzoate.

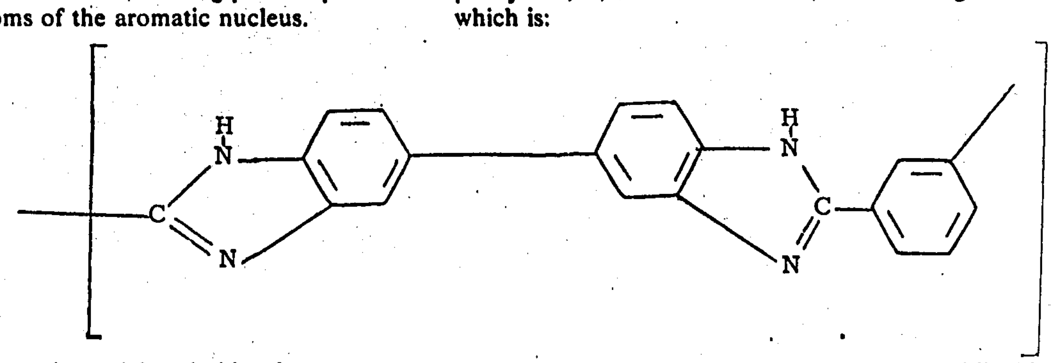
As also set forth in the above-mentioned patent, the aromatic polybenzimidazoles having the recurring units of Formula I may be prepared by condensing an aromatic tetraamine compound containing a pair of orthodiamino substituents on the aromatic nucleus with a dicarboxyl compound selected from the class consisting of (a) the diphenyl ester of an aromatic dicarboxylic acid, (b) the diphenyl ester of a heterocyclic dicarboxylic acid wherein the carboxyl groups are substituents upon a carbon in a ring compound selected from the class consisting of pyridine, pyrazine, furan, quinoline, thiophene and pyran and (c) an anhydride of an aromatic dicarboxylic acid.

Examples of polybenzimidazoles which have the recurring structure of Formula I are as follows:

poly-2,2′-(m-phenylene)-5,5′-bibenzimidazole;
poly-2,2′-(pyridylene-3″,5″)-5,5′-bibenzimidazole;
poly-2,2′-(furylene-2″,5″)-5,5′-bibenzimidazole;
poly-2,2′-(naphthalene-1″,6″)-5,5′-bibenzimidazole;
poly-2,2′-(biphenylene-4″,4″)-5,5′-bibenzimidazole;
poly-2,2′-amylene-5,5′-bibenzimidazole;
poly-2,2′-octamethylene-5,5′-bibenzimidazole;
poly-2,6-(m-phenylene)-diimidazobenzene;
poly-2,2′-cyclohexeneyl-5,5′-bibenzimidazole;
poly-2,2′-(m-phenylene)-5,5′-di(benzimidazole) ether;
poly-2,2′-(m-phenylene)-5,5′-di(benzimidazole) sulfide;
poly-2,2′-(m-phenylene)-5,5′-di(benzimidazole) sulfone;
poly-2,2′-(m-phenylene)-5,5′-di(benzimidazole) methane;
poly-2′,2″-(m-phenylene)-5′,5″-di(benzimidazole) propane-2,2; and
poly-2′,2″-(m-phenylene)-5′,5″-di(benzimidazole) ethylene-1,2

where the double bonds of the ethylene groups are intact in the final polymer.

The preferred polybenzimidazole for use in the present invention is one prepared from poly-2,2′-(m-phenylene)-5,5′-bibenzimidazole, the recurring unit of which is:



Preferably, aromatic polybenzimidazoles are selected, e.g., polymers consisting essentially of the recurring units of Formulas I and II wherein R′ is an aromatic ring of a heterocyclic ring.

Any polymerization process known to those skilled in the art may be employed to prepare the polybenzimidazole which may then be formed into a continuous length of fibrous material. Representative techniques for preparing the polybenzimidazole are disclosed in U.S. Pat. Nos. 3,509,108, 3,549,603, and 3,551,389, which are assigned to the assignee of the present invention and are herein incorporated by reference.

With respect to aromatic polybenzimidazoles, preferably equimolar quantities of the monomeric tetraamine andd dicarboxyl compound are introduced into a first stage melt polymerization reaction zone and heated therein at a temperature above about 200°C., preferably at least 250°C., and more preferably from about 270°to 300°C. The reaction is conducted in a substantially oxygen-free atmosphere, i.e., below about 20 ppm oxygen and preferably below about 8 ppm oxygen, until a foamed prepolymer is formed having an inherent viscosity, expressed as deciliters per gram, of at least 0.1, and preferably from about 0.13 to 0.3, the inherent viscosity (I.V.) as used herein being determined from a solution of 0.4 grams of the polymer in 100 ml. of 97 percent H_2SO_4 at 25°C.

After the conclusion of the first stage reaction, which normally takes at least 0.5 hour and preferably 1 to 3 hours, the foamed prepolymer is cooled and then powdered or pulverized in any convenient manner. The resulting prepolymer powder is then introduced into a second stage polymerization reaction zone wherein it is heated under substantially oxygen-free conditions as described above, to yield a polybenzimidazole polymer product, desirably having an I.V., as measured above, of at least 0.6, e.g., 0.80 to 1.1 or more.

The temperature employed in the second stage is at least 250°C., preferably at least 325°C., and more preferably from about 350°to 425°C. The second stage reaction generally takes at least 0.5 hour, and preferably from about 1 to 4 hours or more.

A particularly preferred method for preparing the polybenzimidazole is disclosed in the aforesaid U.S. Pat. No. 3,509,108. As disclosed therein aromatic polybenzimidazoles may be prepared by initially reacting the monomer in a melt phase polymerization at a temperature above about 200°C. and a pressure above 50 psi (e.g., 300 to 600 psi) and then heating the resulting reaction product in a solid state polymerization at a temperature above about 300°C. (e.g. 350°to 500°C.) to yield the final product.

As is known in the art, polybenzimidazoles are generally formed into continuous lengths of fibrous materials by solution spinning, that is, by dry or wet spinning a solution of the polymer in an appropriate solvent such as N,N-dimethylacetamide, N,N-dimethylformamide, dimethylsulfoxide or sulfuric acid (used only in wet spinning) through an opening of predetermined shape into an evaporative atmosphere for the solvent in which most of the solvent is evaporated (dry) or into a coagulation bath (wet), resulting in the polymer having the desired filamentary shape.

The polymer solutions may be prepared in accordance with known procedures. For example, sufficient polybenzimidazole may be dissolved in the solvent to yield a final solution suitable for extrusion containing from about 10 to 45 percent by weight of the polymer, based on the total weight of the solution, preferably from about 20 to 30percent by weight.

One suitable means for dissolving the polymer in the solvent is by mixing the materials at a temperature above the atmospheric boiling point of the solvent, for example 25°to 120°C. above such boiling point, and at a pressure of 2 to 15 atmospheres for a period of 1 to 5 hours.

Preferably, the polymer solutions, after suitable filtration to remove any undissolved portions, are dry spun. For example, the solutions may be extruded through a spinneret into a conventional type downdraft spinning column containing a circulating inert gas such as nitrogen, noble gases, combustion gases or superheated steam. Conveniently, the spinneret face is at a temperature of from about 100°to 170°C., the top of the column from about 120°to 220°C., the middle of the column from about 140°to 250°C., and the bottom of the column from about 160°to 320°C. After leaving the spinning column, the continuous filamentary materials are taken up, for example, at a speed within the range of about 50 to 350 meters or more per minute. If the continuous filamentary materials are to be washed while wound on bobbins, the resulting "as-spun" materials may be subjected to a slight steam drawing treatment at a draw ratio of from about 1.05:1 to 1.5:1 in order to prevent the fibers from relaxing and falling off the bobbin during the subsequent washing step. Further details with respect to a method for dry-spinning a continuous length of a polybenzimidazole fibrous material are shown in U.S. Pat. No. 3,502,576 to Bohrer et al. which is assigned to the same assignee as the present invention and is herein incorporated by reference.

The continuous length of polybenzimidazole fibrous material is next washed so as to remove at least the major portion of residual spinning solvent, e.g., so that the washed materials contain less than about 1 percent by weight solvent based on the weight of the continuous filamentary material, and preferably so as to obtain an essentially spinning solvent-free fibrous material (i.e., a fibrous material containing less than about 0.1 percent solvent by weight). Typically, a simple water wash is employed; however, if desired, other wash materials such as acetone, methanol, methylethyl ketone and similar solvent-miscible and volatile organic solvents may be used in place of or in combination with the water. The washing operation may be conducted by collecting the polybenzimidazole fibrous material on perforated rolls or bobbins, immersing the rolls in the liquid wash bath and pressure washing the fibrous material, for example, for about 2 to 48 hours or more. Alternatively, the continuous length of polybenzimidazole fibrous material may be washed on a continuous basis by passing the fibrous material in the direction of its length through one or more liquid wash baths (e.g., for 1 to 10 minutes). Any wash technique known to those skilled in the art may be selected.

The continuous length of polybenzimidazole fibrous material may next be dried to remove the liquid wash bath by any convenient technique. For instance, the drying operation for bobbins of yarn may be conducted at a temperature of about 150°to 300°C. for about 2 to 100 hours or more. Alternatively, the continuous length of polybenzimidazole fibrous material may be dried on a continuous basis by passing the fibrous material in the direction of its length through an appropriate drying zone (e.g. an oven provided at 300°to 400°C. for 1 to 2 minutes). If drying is employed, preferably the drying temperature does not exceed about 250°C. for several hours or 400°C. for more than 1 minute, as above these limits degradation of the fiber may occur. As is known to those skilled in polybenzimidazole fiber technology, the fibrous material has a propensity to pick up about 10 to 13 percent moisture by weight when exposed to ambient conditions for an appreciable period of time.

Heretofore continuous lengths of polybenzimidazole fibrous materials have been hot drawn (1) while in sliding contact with a hot surface, e.g. a hot shoe, or (2) while passing for a plurality of passes through a radiantly heated drawing zone in which the fibrous material is suspended. Difficulties have been encountered with hot shoe polybenzimidazole drawing techniques because of solid deposit formation upon the hot contact surface which requires periodic cleaning, and which may result in fiber damage and decreased line stability if allowed to accumulate. Also, precise hot shoe temperature maintenance uniformity has been of prime importance. When radiant heat has been supplied to the polybenzimidazole fibrous material in prior art techniques (e.g., the process of U.S. Pat. No. 3,622,660), it has been essential that the continuous length of fibrous material be passed through the drawing zone for a plurality of passes in order to accomplish the desired degree of drawing. Such processes additionally require a complex string-up arrangement which is impractical for large scale economic production, and commonly are accompanied by the production of broken filaments.

In commonly assigned U.S. Pat. application Ser. No. 297,511 filed Oct. 13, 1972, now U.S. Pat. No. 3,849,529, of G. R. Ferment, A. E. Prince, Jr., and P. A. Sessa there are disclosed an improved single pass process for drawing continuous lengths of polybenzimidazole filaments and an improved apparatus for carrying out the process.

Essentially, the apparatus disclosed in the copending application comprises an elongated draw tube which is surrounded by a coaxial, cylindrical heating jacket. Provision is also made for introducing a heated gas directly into the draw tube. This arrangement permits heating the filament both by radiant heat through the wall of the draw tube and by direct contact with the heated gas. The present invention is considered an advance over the subject matter of U.S. Pat. application Ser. No. 297,511. The process of U.S. Pat. application Ser. No. 297,511 is improved when the process modifications disclosed herein are adopted, and the improved apparatus disclosed herein utilized.

It is an object of the present invention to provide an improved apparatus and process for the thermal processing (e.g. drawing, drying, relaxing, etc.) of a wide variety of continuous lengths of fibrous materials on a continuous basis.

It is an object of the present invention to provide an improved apparatus and process which is particularly suited for the hot drawing of a polybenzimidazole fibrous material.

It is an object of the present invention to provide an improved apparatus and process which respresents an advance over the subject matter of commonly assigned U.S. Pat. application Ser. No. 297,511.

It is an object of the present invention to provide an improved apparatus for thermally processing continuous lengths of fibrous materials which is compact and requires no external heaters.

It is another object of the present invention to provide an improved apparatus for thermally processing continuous lengths of fibrous materials wherein heat loss is minimized and fuel conserved.

It is a further object of the present invention to provide an improved apparatus for the thermal processing of continuous lengths of fibrous materials on a continuous basis wherein the initial stringup of the fibrous material is rendered simple.

These and other objects, as well as the scope, nature, and utilization of the process will be apparent from the following detailed description and appended claims.

SUMMARY OF THE INVENTION

It has been found that in an apparatus for thermally processing continuous lengths of fibrous materials wherein the fibrous materials are passed through a heating unit to take-up means the heating unit including,

- an elongated cylindrical heat treatment tube having an inlet end and outlet end for the continuous passage therethrough of the fibrous materials to be heat treated,

an improvement comprises providing:

- an elongated cylindrical jacket coaxially surrounding the heat treatment tube for providing an annular chamber about the heat treatment tube;
- means for introducing a fluid into the annular chamber;
- means operably connected to the heating unit for heating the fluid and promoting the conduction of heat from the annular chamber through the heat treatment tube to heat continuous lengths of fibrous materials passing therethrough; and
- means connected to the inlet end of the heat treatment tube for introducing heated fluid from the annular chamber into the heat treatment tube to thereby further heat the continuous lengths of fibrous materials passing through the heat treatment tube.

It has been found that in a process for thermally treating continuous lengths of fibrous materials including the step of passing the fibrous materials through an elongated cylindrical heat treatment tube having an inlet end and an outlet end to take-up means, an improvement comprises:

- introducing fluid into an annular chamber coaxially surrounding the cylindrical heat treatment tube; and
- heating the continuous lengths of fibrous materials passing through the cylindrical heat treatment tube by,
  - heating the fluid and promoting the conduction of heat from the annular chamber through the heat treatment tube, and
  - introducing heated fluid from the annular chamber directly into the inlet end of the cylindrical heat treatment tube for passage through said heat treatment tube along with the continuous lengths of fibrous material.

DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic representation of an apparatus of the present invention showing its relationship to a continuous length of fibrous material being treated.

FIG. 2, consisting of two parts, is a longitudinal section of the entire heating unit of the present invention showing the internal structure, wherein the inlet end is shown in the upper part of FIG. 2 and exit end is shown in the lower part of FIG. 2.

FIG. 3 is a transverse section taken on lines 3—3 of FIG. 2 and shows the structure and arrangement of the preheat means.

FIG. 4 is a transverse section taken on line 4—4 of FIG. 2 and shows the location of the nozzle.

FIG. 5 is a transverse section taken on line 5—5 of FIG. 2 and shows the internal arrangement of the heat treatment tube and the heating means within the annular space about the heat treatment tube.

FIG. 6 is a transverse section taken on line 6—6 of FIG. 2 and shows the entry ports for the heating fluid and the heating means.

FIG. 7 shows an alternate embodiment of the portion of the preheat control unit represented in FIG. 3.

FIG. 8 is a longitudinal sectional view of the exit end of the apparatus showing an alternate embodiment in which means are provided for recycling a portion of the exiting fluid to the incoming fluid.

FIG. 9 is a transverse section taken on line 9—9 of FIG. 8 showing the relative location of the bypass means.

FIG. 10 is a longitudinal sectional view of the exit end of the apparatus and shows a further embodiment of means for delivering a portion of the exiting fluid into the incoming fluid.

DESCRIPTION OF PREFERRED EMBODIMENTS

The continuous length of fibrous material which is thermally processed in accordance with the present invention may be natural or synthetic in origin. Lengths of fibrous materials consisting of staple or continuous fibers may be processed. The continuous length of fibrous material preferably is formed of one or more continuous filaments, and most preferably is a multifilament fibrous material such as a yarn, strand, cable, tow, and the like.

In a particularly preferred embodiment of the process a continuous length of polybenzimidazole fibrous material is hot drawn in accordance with treatment temperatures and residence times described in commonly assigned U.S. Pat. application Ser. No. 297,511, filed Oct. 13, 1972 which is herein incorporated by reference.

The polybenzimidazole fibrous material prior to drawing or other thermal processing in accordance with the present invention preferably possesses a denier per filament of about 1 to 20, and most preferably about 3 to 16 (e.g., 3 to 6 for a multifilament tow and 8 to 16 for a multifilament yarn). Multifilament yarns selected for use in the process preferably contain about 10 to 500 filaments, and most preferably about 25 to 200 filaments. A multifilament tow selected for use in the process preferably contains about 1,000 to 300,000 filaments, or more, and most preferably about 50,000 to 150,000 filaments. when tows containing an extremely large number of filaments are drawn in accordance with the present invention, it is preferred that the tows be supplied to the heat treatment tube (described hereafter) while in a flattened ribbon-like configuration.

The fluid introduced into the apparatus of the present invention is preferably air; however, other gaseous atmospheres such as nitrogen, argon, helium, superheated steam, etc. may be selected.

The fluid provided in the heat treatment tube is preferably at a temperature of about 400°to 600°C., and most preferably at a temperature of about 430°to 530°C. when hot drawing a continuous length of polybenzimidazole fibrous material.

The continuous length of polybenzimidazole fibrous material may be drawn through the application of a longitudinal tension thereto while passing in the direction of its length for a single pass through the heat treatment tube provided with a flowing fluid as described hereafter with heat being supplied to the fibrous material both by radiation and convection. The polybenzimidazole fibrous material may be drawn at a draw ratio of about 2:1 to 5:1 while passing through the heat treatment tube, and most preferably at a draw ratio of about 2:1 to 3.5:1.

The term "draw ratio," as is well known, is a measure of the degree of stretching during the orientation of the fibrous material, expressed as the ratio of the cross-sectional area of the undrawn material to that of the drawn material. While any of the several known ways for measuring or determining draw ratio may be employed, typically, the draw ratio is found by taking the ratio of the surface speed of the take-up roll at the exit end of the heat treatment tube to the surface speed of the feed or supply roll at the entrance end of the heat treatment tube.

It is possible that the continuous length of polybenzimidazole fibrous material which is drawn in the present invention be in intimate association with a substantial quantity of water when introduced into the heat treatment tube (described hereafter), i.e., the fibrous material may be (1) in intimate association with its equilibrium moisture content of about 10 to 13 percent by weight based upon the weight of the fibrous material, or (2) be soaking wet and contain up to about 100 percent by weight of water based upon the weight of the fibrous material, e.g., often about 15 to about 70 percent by weight of water. When the fibrous material is provided in association with an appreciable quantity of water the drying may be surprisingly conducted simultaneously with drawing in the heat treatment tube (described hereafter) without foaming or sacrifice of tensile properties within the resulting drawn fibrous material. A time consuming separate drying step may accordingly be completely eliminated.

The minimum hot drawing residence time for a polybenzimidazole fibrous material, i.e., the time during which the material is heated while suspended in the heat treatment tube while under a longitudinal tension, is dependent upon the single filament denier of the fibrous material, the number of filaments in the continuous length of fibrous material, and whether the fibrous material is in a substantially anhydrous form when introduced into the heat treatment tube. These factors also determined the optimum residence times of the fibers in the heat treatment zone. Typically, when filaments of about 1 to 20 denier are present in a yarn of about 10 to 500 filaments, residence times of about 0.05 to 10 seconds, preferably 0.1 to 2 seconds, and more preferably 0.2 to 0.5 seconds are employed. When processing tows of about 50,000 to 150,000 filaments of 1 to 20 denier per filament, typically the residence times are about 0.5 to 30 seconds, preferably 1 to 15 seconds, and more preferably 3 to 10 seconds. The shorter residence times are associated primarily with anhydrous fibrous materials, smaller denier filaments, and with smaller yarns and tows.

The drawing speed commonly is at least 10 meters per minute and is influenced by the length of the heat treatment tube. The "drawing speed" is defined as the rate at which the continuous length of fibrous material is supplied to the heat treatment tube while under a longitudinal tension. The drawing speed is preferably about 10 to 30 meters per minute when drawing a tow, and preferably about 50 to 150 meters per minute when processing a multifilament yarn.

Referring briefly to FIG. 1, it is seen that the heating unit comprises essentially an elongated cylinder indicated generally by reference numeral 2, through which the continuous length of fibrous material 1 is passed in the direction of its length while axially suspended therein to take-up means. The fibrous material is placed under a longitudinal tension by two pairs of skewed rollers indicated generally by 18 and 19, respectively. The inside of the cylinder is heated by means of an electrical element, not shown in FIG. 1, but whose external connections are indicated at 5. Gas is also admitted to the inside of the cylinder through inlet port 4, as will be more fully described below.

Referring to FIG. 2, which consists of two parts, it will be seen that the fibrous material 1 is passed continuously through elongated heat treatment tube 6 which defines a heating zone 7. Surrounding the heat treatment tube 6, there is a concentric, coaxial, elongated, cylindrical jacket 3 of sufficient diameter to provide an annular chamber between the heat treatment tube and the inside surface of the cylindrical jacket to accommodate a heating element 8. This heating element is a coiled electrical resistance heating element with the ends extending from the exit end of the apparatus connected by means of leads 5 to a source of electrical energy (not shown). A heat transfer fluid, such as air, is admitted to the annular chamber 9 through gas inlet port 4 in end closure 38. Leads 5 are also admitted into the annular chamber through the same end closure 38. As can be seen by the arrows, the air inlet port is located at the exit end of the apparatus.

Referring specifically to the upper portion of FIG. 2, it will be seen that the inlet end of heat treatment tube 6 is provided with an enlarged shoulder 10 and an extension 11. Extension 11 of heat treatment tube 6 is inserted into a cylindrical opening 39 in a stationary cylindrical end plug 12 which is enclosed in an overhanging extended portion of the cylindrical jacket 3 and is secured to end plug 12 by means of set screw 13. End plug 12 is provided with a duct 14, one end of which communicates with the annular chamber 9, the other end of which connects with a conical bore 15 to form an aspirator in end plug 12. Stationary cylindrical plug 12 is provided with a cylindrical recess 40 which extends from the extreme left end, as seen in the upper portion of FIG. 2, and ends as shoulder 41. A nozzle 16 having an axial bore 27 is secured on shoulder 41 by means of screw 17. Nozzle 16 is of sufficient length to extend into conical bore 15 its entire length. The outside surface of nozzle 16 is also conical in shape and tapers inward toward the end of bore 15 to provide a conical annular space between the inside surface of the conical bore 15 and the outside conical surface 24 of nozzle 16. In a preferred form, the nozzle tapers less rapidly than the conical bore in the end plug with the result that the conical annular space between the plug and the nozzle gradually decreases in width. As a result, the velocity of the fluid passing between the two parts gradually increases to provide an aspiration zone of low pressure as a result of the Venturi effect at the exit end of the nozzle.

A solid rotatable cylindrical member 25 is inserted in the remaining space of recessed opening 40 of the stationary cylindrical end plug 12. This rotatable cylindrical member bears on surface 30 of the nozzle and also fits tightly within space 40. A central bore 26 is axially coincident with the axis of the heat treatment tube and the bore of the nozzle and is of the same diameter as the nozzle bore 27. A duct 28 is provided in the body of end plug 12 which connects the annular space 9 with the matching duct 29 through flange 30 of the nozzle. Duct 29, in turn, connects with duct 31 in the rotatable cylindrical plug member 25. Duct 31 branches into legs 33 and 34, which open into the central bore 26. Handle 32 secured in any suitable manner to member 25, is used to rotate the latter. As shown in the upper portion of FIG. 2, ducts 28, 29, 31, 33 and 34 are in such position that the hot gases in annular space 9 will be conducted to the central bore 26. The purpose of this arrangement is to preheat the fibrous material as it enters the apparatus and to minimize the reception of relatively cooler ambient air into the nozzle bore 27. During string-up, however, the handle is used to rotate duct 31 out of register with duct 29 so that no fluid can be conducted from annular space 9 to the inlet end of bore 26. By so doing, all of the gas is directed through duct 14 around the outside surface of nozzle 16. Because of the progressively decreasing diameter of the conical space of aspirator 15 the velocity of the fluid increases until it reaches a maximum at the exit end of the nozzle. The space immediately beyond the end of the nozzle thus becomes a zone of very low pressure, i.e. an aspiration zone and serves to draw the end of the fibrous material into the apparatus and through the heat treatment tube 6 during string-up. Once the end of the fibrous material has been secured to take-up means (not shown) the cylindrical member 25 is rotated so that hot gases will be permitted to pass from the annular space 9 into the inlet end of bore 26 to preheat the fibrous material as it enters the apparatus. Thus, the fibrous material passing through the heat treatment tube is heated both by the hot gases which surround them during passage through heat treatment tube 6 and also by radiation from the walls of the heat treatment tube which have been heated by the fluid present in annular chamber 9.

Rotatable plug 25 is secured in place by means of a sealing ring 35 which is secured to the stationary end plug 12 by means of screws 36. To provide the necessary sealing pressure of the rotatable plug 25 against the surface of flange 30 of nozzle 16, a spring loaded washer 37 is inserted between ring 35 and rotatable cylindrical member 25.

Temperature sensing elements 21 and 22 are inserted in bore 26 and the entrance end of heat treatment tube 6, respectively, to measure the temperatures at those points.

Should additional or auxiliary heating fluid be desired or needed, plug 42a which closes a port leading into duct 14 may be removed and replaced by an inlet tube connected to the same or some other source of fluid.

Referring to the lower portion of FIG. 2, it will be seen that the heat treatment zone of the apparatus comprises that part which extends between the nozzle and the end closure 38 at the exit end of the heat treatment tube. This heating area, as already described, may contain heating element 8 wound in a spiral around heat treatment tube 6. Air or other fluid forced in through port 4 in end closure 38 travels through the annular chamber 9 and is heated by the electrical heating element 8. At the same time, heating element 8 imparts heat to the wall of heat treatment tube 6. Thus, a fibrous material passing through the center of draw tube 6 is heated both by contact with the hot gases flowing through the tube and also by radiation from the heated walls of the tube. A temperature sensing device 23, similar to 21 and 22 is inserted near the outlet end of the draw tube to measure the temperature of the exiting gases. These temperature sensing devices connected to measuring instruments (not shown) give the operator the information necessary to control the temperature at the several critical points between the entrance and the exit ends of the apparatus. After heating is completed, the fibrous material and fluid may exit through the open end 42 of draw tube 6.

As just described, it was shown that in the embodiment described in FIG. 2, the exit gases may be exhausted to the atmosphere. However, another preferred embodiment provides for a portion of the gases to be collected and delivered into the entering gases, as described below.

Referring to FIG. 8, the cylindrical outer jacket 3, in the just-mentioned preferred embodiment, is extended beyond the exit end 42 of heat treatment tube 6 to form a hollow cylindrical chamber beyond the exit end of heat treatment tube 6. Into this chamber is inserted a chambered cylindrical plug, designated generally as 43, whose outer wall 44 is of such dimension as to fit tightly inside the opening of the extension of cylindrical jacket 3 to form an air-tight seal. This plug 43 is sealed in the opening by any suitable means. Plug 43 is provided with an inner cylindrical chamber 45 which is shorter than the length of the plug itself. End walls 48 and 49 of inner cylindrical chamber 45 have circular holes 46 and 47, respectively, of a diameter smaller than that of the inner cylindrical chamber 45. Through these holes is inserted a porous tube 54 which is the same length as chambered cylindrical plug 43. This porous tube has an inner bore 57 of the same diameter as draw tube 6. The chambered cylindrical plug 43 also has drilled in it a gas duct 50 which connects an inlet port 51 with the annular chamber 9 inside the cylindrical jacket 3. This duct is of unifrom diameter up to a point where hole 53 through wall 52 of the inner cylindrical chamber 45 connects the latter with duct 50. From that point to where the duct 50 opens into the annular space 9, the diameter constantly increases. The pressure of the exiting gases is sufficient to cause a portion of these gases to diffuse through the walls of porous tube 54 into inner cylindrical chamber 45 and from there to pass through hole 53 into duct 50. There they mingle with incoming gases forced in through port 51 and are recirculated through annular chamber 9 through the nozzle and into the heat treatment tube 6. In this manner, less of the gases are exhausted to the atmosphere, while the gases freshly blown in through port 51 are preheated. Thus, there is not only a reduction in exhaust gases in the atmosphere, but a saving in energy due to the preheating of the incoming gases.

In still another embodiment, which is an improvement over that shown in FIG. 9, even greater amounts of exhaust gases are recycled through the apparatus. Thus, referring to FIG. 10, it can be seen that the porous tube 56 has a constricted opening 57 at its exit end, i.e. an annular restriction. This constriction restricts the axial flow of fluid and causes a certain amount of back pressure which results in an additional flow of fluid through the porous walls of the tube. In this manner, even less of the fluid is exhausted to the atmosphere while an additional amount is recycled with the incoming fluid for greater economy of material and heat.

FIG. 7 discloses still a further embodiment of the present invention. In this embodiment, a preferred modification is made of the means for preheating the fibrous material at the entrance end of the apparatus.

Referring to FIG. 7, rotatable member 25, instead of being provided with duct 31 and branching ducts 33 and 34, as shown in the upper portion of FIG. 2, has instead a hole 58 drilled radially from the outer surface of the rotatable plug to the axial bore 26. This hole is then threaded to accomodate screw 60. A duct 59 connects duct 29 in the flange 30 of the nozzle with the just described radial threaded hole 58. Thus, there is again communication between annular space 9 and the interior of the axial bore 26. Screw 60 has a short duct 66 drilled at right angles to its axis which connects with cut-out portions 61 and 62 which are parallel to the longitudinal axis of the screw. Thus, as shown in FIG. 7, the cutout portions of screw 60 provide communication between the annular chamber 9 and the interior of the bore 26. The amount of fluid flowing to the interior of bore 26 is regulated by turning the screw with a screwdriver in slot 65. Access to the screw is provided by hole 63 which is drilled in the outer cylindrical jacket 3 and hole 64 which is drilled in stationary cylindrical end plug 12. As shown, the maximum amount of fluid will flow through this combination of ducts. However, the amount can be decreased by increments by turning the screw one turn clockwise for each increment of flow to be decreased. Thus, by predetermining the pitch of the thread in hole 58 and of the screw 60, the amount of axial travel can be predetermined for each turn of the screw. As in the case of the embodiment shown in the upper portion of FIG. 2, these ducts can be closed off during string-up by rotating the member 25 by means of handle 32. By means of this modification, it is possible to obtain a more precise control of the amount of preheating fluid which is directed at the incoming fibrous material.

As previously indicated, the preferred embodiments of the apparatus as described above are particularly suited for carrying out the polybenzimidazole drawing process of U.S. Pat. application Ser. No. 297,511. For this purpose the overall length of the apparatus advantageously can be about 2 to 6 feet. A suitable length to overall diameter can vary from about 4:1 to 10:1. The bore of the heat treatment tube can be about 5/16 inch inner diameter, and the walls of the porous tube can have a thickness of about ⅛ inch or less. The stream of fluid (e.g. air) can be introduced at a rate to produce a gas flow in the main bore of the heat treatment tube of about 50 to 200 cubic feet per second which exhibits a Reynold's number preferably above about 4,000, and most preferably a Reynold's number of about 5,000 to 20,000.

Although the invention has been described with preferred embodiments, it is to be understood that variations and modifications may be resorted to as will be apparent to those skilled in the art. Such variations and modifications are to be considered within the purview and scope of the claims appended hereto.

We claim:

1. In an apparatus for thermally processing continuous lengths of fibrous materials wherein said fibrous materials are passed through a heating unit to take-up means, said heating unit including,

an elongated cylindrical heat treatment tube having an inlet end and outlet end for the continuous passage therethrough of said fibrous materials to be heat treated,

wherein the improvement comprises:

an elongated cylindrical jacket coaxially surrounding said heat treatment tube for providing an annular chamber about said heat treatment tube;

end closures being positioned in the annular chamber between said cylindrical jacket and said heat treatment tube closing the annular chamber at its ends;

means for introducing a fluid into said annular chamber;

an electric resistance heating coil mounted within the interior of said cylindrical jacket for heating the fluid and promoting the conduction of heat from said annular chamber through said heat treatment tube to heat continuous lengths of fibrous materials passing therethrough;

means connected to the inlet end of said heat treatment tube for introducing heated fluid from said annular chamber into said heat treatment tube to thereby further heat said continuous lengths of fibrous materials passing through said heat treatment tube; and

heat recovery means connected at the outlet end of said cylindrical heat treatment tube for receiving at least a portion of the heat passing through the outlet end of said cylindrical heat treatment tube and for delivering said heat into said annular chamber.

2. In an apparatus for thermally processing continuous lengths of fibrous materials as defined in claim 1, said means for introducing the heated fluid into said heat treatment tube comprises:

aspirator means coaxially mounted at the inlet end of said heat treatment tube for receiving the heated fluid from said annular chamber and aspirating the fluid into said heat treatment tube.

3. In an apparatus for thermally processing continuous lengths of fibrous materials as defined in claim 2, and wherein the improvement further comprises:

means connected to said aspirator means for introducing auxiliary heating fluid into said aspirator, and thereby said heat treatment tube, from a source of heated fluid remote from said annular chamber.

4. In an apparatus for thermally processing continuous lengths of fibrous materials as defined in claim 2, and wherein the improvement further comprises:

preheat means coaxially connected at an inlet end of said aspirator means and being selectively operable to receive heated fluid from said annular chamber for introducing heated fluid into the inlet end of said aspirator means and for minimizing the reception of relatively cooler ambient air into said aspirator means.

5. In an apparatus for thermally processing continuous lengths of fibrous materials as defined in claim 4, wherein said preheat means further comprises:

means for selectively regulating the quantity of heated fluid introduced from said annular chamber into said preheat means.

6. In an apparatus for thermally processing continuous lengths of fibrous materials as defined in claim 1, wherein said heat recovery means comprises:

a porous tube coaxially mounted at the outlet end of said cylindrical heat treatment tube; and

housing means concentrically positioned about said porous tube and being in fluid communication with said annular chamber for receiving heated fluid passing through said porous tube and delivering the thus received heated fluid into said annular chamber.

7. In an apparatus for thermally processing continuous lengths of fibrous materials as defined in claim 6, wherein said heat recovery means further comprises:

an annular restriction connected at the outlet end of said porous tube to promote passage of heated fluid from said cylindrical heat treatment tube through said porous tube and into said housing means for delivery to said annular chamber.

* * * * *