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(54) **A composite polytetrafluoroethylene article and a method for its manufacture**

(57) A porous polytetrafluoroethylene (PTFE) article is made up of a number of smaller articles 4 with a microstructure of nodes interconnected by fibrils, these articles having been joined to one another such that their microstructure is virtually unaltered across the join. Such a PTFE article is produced by abutting small-shaped PTFE segments 4 and applying a force perpendicular to the seam while heating to a temperature above the crystalline melt point of the segments. As shown, a tube is made by butt joining segments 4 laid on a mandrel 2 and a strip of expanded P.T.F.E. film is wound spirally therearound and secured against unwinding. The wrapped tube is heated in a salt bath, an air oven or a radiant oven so that the restraining film shrinks and applies pressure to the segments.

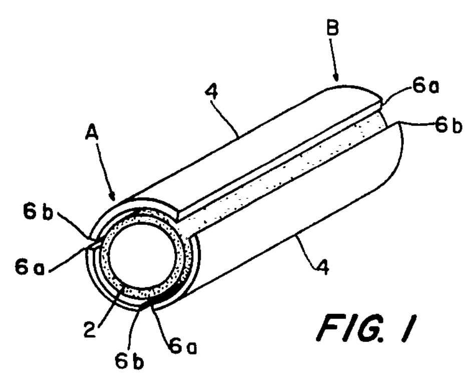

FIG. 1

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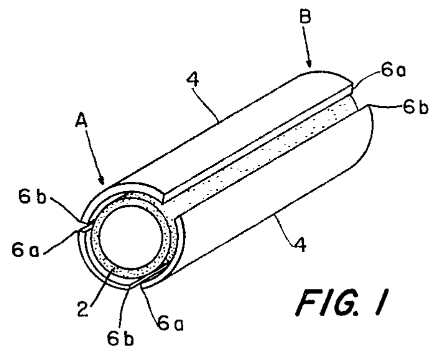

FIG. 1

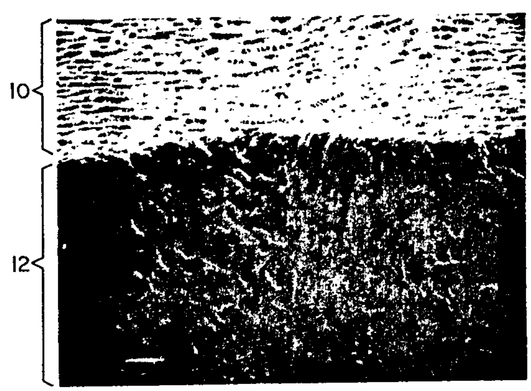

FIG. 2

FIG. 3(b)

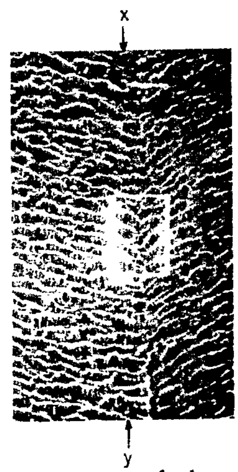

FIG. 3(a)

FIG. 4(b)

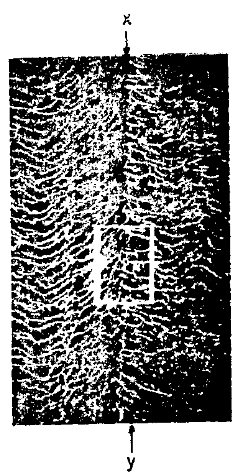

FIG. 4(a)

SPECIFICATION

A composite polytetrafluoroethylene article and a method for its manufacture

This invention relates to a process for making an article of expanded polytetrafluoroethylene.

Polytetrafluoroethylene (hereinafter "PTFE") has excellent heat resistance, chemical resistance, insulation resistance, non-adhesiveness and self-lubrication. This polymer has found wide use in medical, industrial and recreational fields.

A recent invention (which is described in U.S. Patent Specification No. 3,953,566) provides a process for manufacturing highly porous, yet high strength, shaped, PTFE articles. This process involves blending highly crystalline, fine powder PTFE with a liquid lubricant, extruding this mixture through a die which may have desired cross-sectional configuration, and subsequently expanding the shaped article in one or more directions at rates in excess of 10% per second.

Products produced by this process have found widespread acceptance in the industrial, medical, electrical, and clothing arts. The process is somewhat limited in that it is not readily adaptable to the product of large articles with complex cross-sections. A need for such articles is found, for example, in the industrial filtration arts and in large vessel vascular surgery. Although large composite articles can be manufactured by joining smaller articles together by such conventional methods as sewing, welding or gluing, such articles have a discontinuity at the seam. While in many applications this does not present any severe problems, in others such as filtration and body part replacement, it is extremely important that the structure be as uniform as possible over the entire article. If welding or gluing is used to produce large articles, a dense non-porous area is produced. On the other hand, sewing may produce areas which have a greater porosity than the rest of the article. It has been found that the microstructure of nodes and fibrils present in products produced by U.S. Patent 3,953,566 is particularly desirable as both a filter media and as surface for contacting blood and other body fluids. It would, therefore, be desirable to produce composite, complex shapes by joining articles of expanded PTFE with this microstructure in such a manner that the microstructure remains virtually uninterrupted across the join or seam.

According to the present invention there is provided a process for joining a plurality of polytetrafluoroethylene segments each having a microstructure of nodes interconnected by fibrils in such a manner that said microstructure is virtually uninterrupted at the join comprising:

(a) causing said segments to be held in close contact by mechanical means;

(b) causing said segments to be restrained from shrinking in any direction;

(c) heating said segments, while they are so held, to a temperature above the crystalline melt point of said segments for a predetermined time; and

(d) allowing said segments to cool below the crystalline melt point of said segments while still being held in close contact.

The present invention also provides a process for welding together two laminar pieces of PTFE each having a microstructure of nodes interconnected by fibrils, which comprises bringing the two pieces together to form a butt seam, holding the pieces in position and applying pressure to the pieces in the vincinity of the seam and in a direction parallel to the abutting surfaces of the pieces so as to cause some pressure to be exerted between the abutting surfaces, heating said segments, while they are so held, to a temperature above the crystalline melting point of said pieces until welding has occured, and allowing said segments to cool below the crystalline melting point of said pieces while they are still held under pressure.

The invention will now be particularly described, by way of example, with reference to the accompanying drawings, in which:–

Figure 1 is a schematic illustration of one of the embodiments of the present invention.

Figures 2–4(b) are electromicroscopic photographs of various surfaces of a tube produced in accordance with the invention.

The process of bonding together two parts, both of PTFE, by mechanically holding the two parts in contact and heating them above the crystalline melt point of PTFE, has generally resulted in a solid non-porous seam. The present invention utilizes a modification of the process to produce a product in which the node-fibril microstructure present in both parts is maintained virtually uninterrupted across the seam. The result is that a seam-free product is produced. In the present context, the term "edges" is used to refer to that portion of the expanded PTFE article which is to be bonded together and "seam" refers to the area so bonded. Articles refer to any shaped cross-section, e.g., tube, rod, sheet or segment.

PTFE material is available in a variety of shapes, including sheets, rods and tubes from W.L. Gore & Associates, Inc. The articles to be bonded are cut to the required size. Care must be taken to ensure that the edges to be joined are clean, that is neither ragged nor dirty. The two edges are then brought together, i.e. touching one another.

If expanded PTFE is heated above its crystalline melt point while unrestrained, the material will tend to shrink and coalesce into a solid mass. In order, therefore, to ensure that the two articles to be bonded together remain in contact while being heated, mechanical means must be employed to so hold them.

For example, as illustrated in Fig. 1, a large tube can be made from sheets or sections from a number of smaller tubes. The sheets 4 are trimmed at their edges 6(a) and 6(b) to ensure that these edges are clean, that is, not ragged or dirty. The sheets 4 are then laid around a mandrel 2. The edges 6(a) and 6(b) of each sheet 4 are closely butted to the edges 6(a) and 6(b) of the adjoining sheet. The ends of the sheets, A and B, are fixed at these points to the mandrel. This can be achieved in a variety of ways, such as hose clamps or tying the sheets to the mandrel by wire. The reason for so fixing the tube, is to prevent longitudinal retraction of the PTFE on heating.

A strip of expanded PTFE film about .75 inches wide and having a longitudinal Matrix Tensile Strength of about 70,000 p.s.i. is spirally wound around the sheets on the mandrel and fastened at the end of the mandrel so it cannot unwrap. Upon heating, this restraining film shrinks, applying pressure on the sheets and keeping the edges 6(a) and 6(b) in close contact. A satisfactory film is commercially available as GORE-TEX (Trade Mark) expanded filament from W.L. Gore & Associates, Inc., P.O. Box 1220, Elkton, Maryland, 29121. Although wrapping with an expanded PTFE film is a preferred means of mechanically restraining and holding the edges of the sheets in contact during heating, other means may be used.

The important factor is that there must be some force perpendicular to the seam during sintering. When the film wrapped around the tube contracts, it supplies the necessary force.

The heating of the wrapped tube can be achieved in a salt bath, an air oven, a radiant oven or other heating means. A suitable salt bath can be molten mixture of sodium nitrites and nitrates and is maintained at a temperature above the crystalline melt point of the segments. The tube is then removed and allowed to cool while still being held restrained. The time above the crystalline melt point will vary depending on the mass of material involved and resin properties. The exact time to produce an optimum bond will depend on a number of factors such as mass of material and the configuration of the shape being produced. Such a time, however, is easily determined with a minimum of experimentation. The following example is intended to illustrate and not limit the present invention. The technique can be used in any of a variety of shapes and sizes where it is important to maintain a virtually uninterrupted microstructure across any joining line or seam.

EXAMPLE 1

Three 6.5 cm long, 120° segments were cut from 20 mm inside diameter tubes which had been produced according to the teachings of U.S. Patent 3,953,566. The resin used was Fluon 123 which is a fine powder, PTFE resin commercially available from ICI America. These segments were carefully trimmed to ensure that the edges to be butted were clean. The segments were then carefully laid around a smooth, 20 mm O.D., stainless steel tube. The segments were arranged so that they butted closely together. They were then spirally wrapped with a .75 inch wide expanded PTFE film having a Matrix Tensile Strength of about 70,000 p.s.i., which was manufactured according to U.S. Patent 3,962,153.

The mandrel was then placed in an air oven at 380°C for 12 minutes. Upon removal from the air oven, the mandrel was allowed to cool to room temperature and the formed PTFE tube was carefully slid off the mandrel. For the purpose of this experiment, the wrapping film was carefully removed from the tube in order to photomicrograph the structure at the seam.

Fig. 2 is an angled electromicroscopic photograph of one of the seams made in Example 1. The top portion 10 is a topographical view of the inside surface of the tube. The bottom portion 12 is a cross-section view of the tube. In Fig. 2, the seam runs from X to Y. The magnification is 146 times.

Fig. 3(a) is an electromicroscopic photograph of the inside surface of the tube made in Example 1. The seam runs from X to Y. The magnification is 122 times. Fig. 3(b) is an electromicroscopic photograph of the inserted area in Fig. 3(a). The seam runs from X to Y and the magnification is 610 times.

Fig. 4(a) is an electromicroscopic photograph of the outside surface, after removal of the film, of the tube made in Example 1. The seam runs from X to Y and the magnification is 90 times. Fig. 4(b) is an electromicroscopic photograph of the inserted area shown in Fig. 4(a). The seam runs from X to Y and the magnification is 450 times.

From these electromicroscopic photographs, it is surprising to observe that the node-fibril microstructure is virtually uninterrupted across the seam. Although a small scale example was used to illustrate this invention, this technique can be readily extended to cover large tubes up to several inches in diameter. Equally, the technique can be used with any of a variety of shapes and is not limited to tubular cross-sections. For example, sheets of uniaxially expanded PTFE with thicknesses ranging from about .005" to more than .100" could be joined together in the following manner. To restrain the PTFE sheets in the direction of their expansion, butt them in place between 1/16" thick sheets of 60 durometer silicon rubber (two sheets of rubber on each side of the PTFE that are not quite butted together with a gap of about .010" between them which corresponds to the seam in the PTFE sheets), and place the PTFE and rubber sheets in a press with platens heated to about 380°C. The press could be closed to apply a very small pressure to the sheets sitting on the rubber. This would supply the necessary perpendicular force to the seam, this time in a planar configuration. After an appropriate time, approximately 15 minutes, the electrical heaters on the press could be turned off and the platens cooled by a stream of compressed air. When the platens had cooled to room temperature, the pressure could be released and the sheets removed.

CLAIMS

1. A process for joining a plurality of polytetrafluoroethylene segments each having a microstructure of nodes interconnected by fibrils in such a manner that said microstructure is virtually uninterrupted at the join comprising:

(a) causing said segments to be held in close contact by mechanical means;

(b) causing said segments to be restrained from shrinking in any direction;

(c) heating said segments, while they are so held, to a temperature above the crystalline melt point of said segments for a predetermined time, and

(d) allowing said segments to cool below the crystalline melt point of said segments while still being held in close contact.

2. A process of welding together two laminar pieces of PTFE each having a microstructure of nodes interconnected by fibrils, which comprises bringing the two pieces together to form a butt seam, holding the pieces in position and applying pressure to the pieces in the vicinity of the seam and in a direction parallel to the abutting surfaces of the pieces so as to cause some pressure to be exerted between the abutting surfaces, heating said segments, while they are so held, to a temperature above the crystalline melting point of said pieces until welding has occurred, and allowing said segments to cool below the crystalline melting point of said pieces while they are still held under pressure.

3. A process according to claim 1 or claim 2 which comprises:–

(a) abutting said segment or pieces around a mandrel;

(b) spirally wrapping said segments or pieces with an expanded PTFE film.

4. A composite PTFE article made by the process according to claim 1, claim 2 or claim 3.

5. A welded composite PTFE product comprising a plurality of expanded PTFE segments having a microstructure of nodes interconnected by fibrils, said microstructure being substantially uninterrupted at said weld.

6. A welded composite PTFE article which has been made substantially as herein described.

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