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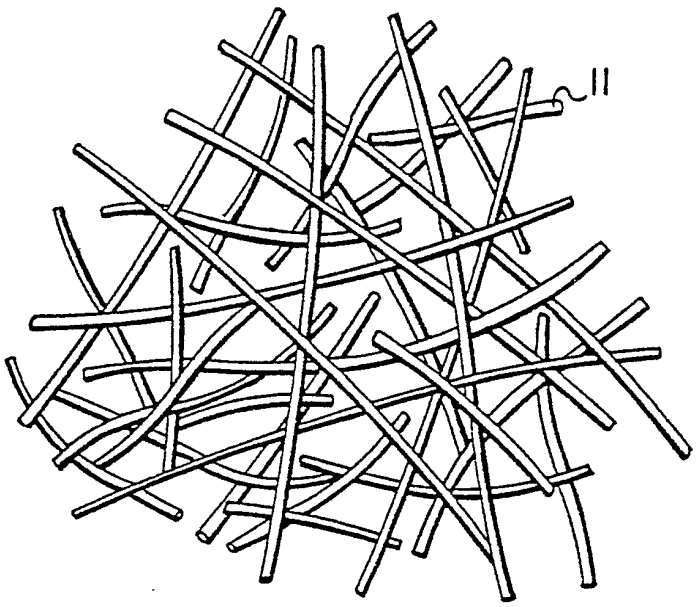

(57) Abstract

A composite material comprising an ultra-high molecular weight polymer and fibers having lengths of about 0.05 inch (1270 micrometers). This composite is formed by first dispersing the fibers with particles of the polymer in an inert liquid carrier. The dispersion is drawn through a filter which collects the particles and fibers as an intermediate structure in which the fibers are oriented in a quasi-isotropic array and the particles are dispersed throughout this fiber network. The intermediate structure is removed from the filter, dried, compacted, and then compressed at an elevated temperature such that the polymer melts, while the fibers remain unmelted. Upon cooling, there is formed a composite comprising a continuous polymer matrix reinforced with the randomly oriented and intertwined fibers of relatively long length. This material can be formed into articles, such as piston rings or seals, to provide a self-lubricating structure having good wear resistance and creep resistance.

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FIBER-REINFORCED COMPOSITE AND METHOD OF MAKING SAME

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates, generally, to a solid-solid composite material comprising a polymer and fibers, and, more particularly, a composite material formed of an ultra-high molecular weight polymer reinforced with relatively long fibers having lengths in excess of 0.05 inch (1270 micrometers), and which is self-lubricating and has good wear resistance and creep resistance; and to a method of making such a composite.

2. Description of the Prior Art

Ultra-high molecular weight polymers generally have a molecular weight in excess of 1.0×10^6. Polyethylene and polytetrafluoroethylene are typical ultra-high molecular weight materials. These materials are self-lubricating in nature. However, because of their ultra-high molecular weight, these materials also have low-flow properties, which makes them difficult to mix with reinforcing fibers. In addition, mixtures of these ultra-high molecular weight polymers with fibers cannot be molded by conventional techniques, other than sintering and/or compression molding, without suffering degradation caused by reduction in the molecular weight of the material. Currently, it is

the practice to reinforce such materials during extrusion with powders and very short fibers, such as glass, having diameters of about 10 micrometers (μm) and aspect ratios (i.e., length to diameter ratio) of about 1 to 10. Although such materials have many useful applications, they are not suitable for use in some aerospace applications where the extreme conditions of use require more highly specialized materials. An example of the need for such specialized materials is for piston ring seals for cryogenic refrigeration equipment to be used in space. These piston rings must be generally of a self-lubricating nature and exhibit low wear rates over long periods of time so that there will not be any loss of sealing performance. However, existing materials containing glass fibers generate abrasive debris, which accelerates wear processes and is undesirable.

It is a particular purpose of this invention to provide a fiber-reinforced polymer which may be used to form a piston ring seal that will be able to withstand the extreme conditions of space, exhibit low wear, and be of a non-abrasive nature.

It is a further purpose to provide a method for forming such a fiber-reinforced polymer.

SUMMARY OF THE INVENTION

We have invented a new composite material which comprises a polymer reinforced with fibers, and exhibits low wear and low creep and has low abrasive characteristics. This composite is made from ultra-high molecular weight polymers reinforced with fibers having lengths of at least about 0.05 inch (1270 micrometers). The aspect ratio of these fibers ranges between about 100:1 and about 300:1.

We have further invented a novel method of making this composite which comprises the steps of:

a) providing the ultra-high molecular weight polymer in the form of particles of predetermined size;

b) providing an inert liquid carrier capable of uniformly dispersing the fibers and the particles of the polymer;

c) blending the fibers and particles of the polymer in the inert liquid carrier to form a uniform dispersion;

d) passing the dispersion through a filter so that there is collected on the filter an intermediate structure comprising a network of quasi-isotropic fibers oriented randomly and intermingled, with the particles of polymer dispersed throughout the free volume space between the fibers in the network;

e) drying the intermediate structure to remove excess liquid carrier therefrom;

f) applying positive pressure to the intermediate structure at ambient temperature to remove entrapped air and to compact the intermediate structure;

g) compressing the compacted intermediate structure in a mold at an elevated temperature at which the polymer matrix melts, but the fibers do not melt, to form upon cooling a fiber-reinforced solid polymer composite comprising a continuous polymer matrix reinforced by a network of nearly planar fibers oriented randomly and intermingled in two dimensions.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a perspective view of the intermediate structure formed prior to the formation of the compressed solid composite product of the present invention.

FIG. 2 is a magnified schematic representation of a portion of the structure shown in FIG. 1.

FIG. 3 is a schematic view of one suitable apparatus for carrying out one step of the method of the present invention.

FIG. 4 is a magnified schematic view of a portion of the structure of FIG. 1 after compaction, with the spheres shown in FIG. 2 now forming a continuous matrix around the fibers.

FIG. 5 is a perspective view of the final compressed composite product of the present invention made in the form of a disk.

FIG. 6 is a perspective view of an annular member formed from the composite material of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The polymer used to form the composite of the present invention has a molecular weight in excess of 1.0×10^6 and is preferably either polyethylene, polytetrafluoroethylene, or some other ultra-high molecular weight or low-flow material. An example of a low-flow polymer is polytetrafluoroethylene, which has a melt viscosity of about 10^{11} poise at its melting temperature of 700°F (371°C). Such materials are of a self-lubricating nature and are generally non-abrasive. However, additional lubricating materials and pigments may be added to these polymers. For example, molybdenum disulfide or graphite powders are suitable lubricants for many applications. It is desirable that the polymer be provided in a particulate state with the size of the particles selected so that they will neither clog the filter employed for filtration nor pass through the filter during the process steps discussed herein.

The fibers used to form the composite of the present invention must be able to withstand the melt temperature for the ultra-high molecular weight polymer matrix without losing their fibrous nature. For use

in self-lubricating applications, such fibers are preferably made of an organic polymer which exhibits non-abrasive characteristics. Suitable polymers are, for example, Kevlar or Nomex, aromatic polyamide materials, both manufactured by E. I. Dupont and Company. When it is not important that the composite be of a non- or low-abrasive nature, other fibers such as boron, glass, graphite, quartz, or metal may be used instead of, or along with, the polymer fibers.

The length of the fibers used in the present invention falls within the following limits: (a) the fibers are not so long that they become tangled into clumps during the wet blending process described herein; and (b) the fibers are not so short that they approximate the behavior of particles which can be more readily mixed with the ultra-high molecular weight polymer by conventional techniques, such as ball milling. With regard to the former limit, it should be noted that the length at which such tangling occurs depends also on the diameter of the fiber and the concentration of the fiber in the suspension. The preferred length is in excess of about 0.05 inch (1270 micrometers), with lengths of about one-eighth inch (3175 micrometers) being particularly effective.

The preferred method of blending the fibers with the polymer is to disperse them in an inert liquid carrier using, for example, a shearing action provided by a Waring blender employing a flat disk type blade. Typically, a mixture of such fibers and polymer ranges between 5 and 60% by weight fibers and between 40 and 95% by weight polymer; and preferably between 5 and 15% fibers and between 85 and 95% polymer. This mixture of fibers and polymer comprises no more than about 3 percent of the blend of carrier liquid and fiber/polymer.

The inert liquid carrier should wet the polymer and fiber materials to provide a good suspension of these materials and should be easily removed by drying. One suitable liquid carrier for polyethylene is a mixture of isopropyl alcohol and water, comprising two parts by volume of alcohol to one part of water. Other known carriers may be used depending on the particular polymer and fiber components used. The combination of the polymer particle size, fiber size, and liquid carrier is chosen to provide a good dispersion of the solids in the liquid carrier. Properties of these materials which affect this dispersion are viscosity, surface tension, density, length, aspect ratio, and particle size. If good dispersion of the solids in the carrier is not achieved, then the polymer and fiber materials will, upon filtration, form separate layers.

As the liquid suspension of polymer particles and fibers is drawn through the filter in accordance with the method of the present invention, the fibers orient themselves in a random fashion on the filter pad, generally intermingled in a quasi-isotropic or nearly three-dimensional network. The particles of polymer are dispersed throughout the free volume space between this fiber network and are held in place mechanically. After filtration, the network of fibers and polymer is dried, typically at approximately 150°F (66 °C) for two hours or more in a vacuum, preferably at about 20 millimeters of mercury or better, and then compacted under pressure at ambient temperature to form a compacted preform. The compaction step serves to remove entrapped air, as well as to produce interpenetration of the polymer particles with each other on a macroscopic level, to form a continuous polymer matrix on a macroscopic level.

When the compacted preform is then further compressed in a mold and simultaneously subjected to heat, the polymer melts and, upon cooling, fuses to bind the fibers together in the polymer matrix. The final product has the shape of the mold. During this final step when the polymer particles melt, an interpenetration of the molecular chains of the polymer particles occurs on a microscopic level to produce a continuous polymer matrix on a microscopic level.

In accordance with conventional practice, when powders and short fibers are dispersed throughout the polymer, they provide a material which does not have the desired wear resistance. This is evidenced by the filler being pulled from the rubbing surface of the material, leaving small pit marks in this surface. However, when randomly oriented long fibers are employed in accordance with the present invention, this filler-pulling does not occur. The fibers, particularly the polymer fibers, will wear away slowly, but will not be pulled from the composite material. The high aspect ratio of the fibers used in the present invention provides both increased surface interaction with the polymer matrix and increased inter-fiber mechanical locking. The energy necessary to pull out such long fibers is much larger than that required to pull out conventionally used chopped fibers. Thus, both the high aspect ratio and the mechanical interlocking of the fibers used in the present invention prevent these fibers from being removed from the polymer matrix during wear, despite low bond strengths typically encountered between fibers and the ultra-high molecular weight polymers previously discussed. Consequently, the composite material using long fibers in accordance with the present invention exhibits low wear and does

not generate abrasive debris. In addition, the non-abrasive nature of certain polymer fibers which may be used in the present invention further adds to the low abrasiveness of the composite material of the present invention.

The advantages of the composite material of the present invention are apparent from the previous description. This composite material is self-lubricating and has low wear, low creep and low abrasiveness. Most importantly, it can be formed into various types of structures, for example, an annular member suitable for use as a piston ring seal and for other aerospace applications.

The features of the present invention can further be understood, together with the advantages discussed above and other advantages, by reference to the following description taken in connection with the drawings.

A mass of discontinuous fibers, such as Kevlar, is used as one of the starting materials to make the composite of this invention. These fibers are chopped and have an aspect ratio of about 250:1 and are generally of approximately 1/8 inch (3175 micrometers) in length and about 12 micrometers in diameter. These fibers cannot ordinarily be adequately blended with ultra-high molecular weight materials in an extruder. We have discovered, however, that these fibers can be blended with particles of, for example, an ultra-high molecular weight polyethylene polymer manufactured by Hercules Corporation and sold under the trade name 1900 UHMW polymer. This particular polymer generally has a molecular weight ranging between about 2×10^6 and about 6×10^6 and a particle size of about 300 micrometers in diameter.

By mixing the fibers with the 1900 UHMW polymer in an isopropyl alcohol/water mixture and dispersing the fibers and the polymers by a shearing means, such as a Waring blender using a disk type blade, there is formed a dispersion of fibers and polymers in liquid which will, upon filtration, form the intermediate structure 10 shown in FIG. 1 and comprising a fiber network/polymer mixture. FIG. 2 presents a magnified schematic view of a portion of the intermediate structure 10 of FIG. 1, comprising the fibers 11 which are oriented in a quasi-isotropic or nearly three-dimensional network, with the particles 12 of polymer dispersed throughout the spaces between the fibers and mechanically held in place within the fiber network. It should be noted that FIG. 2 is of a schematic nature and illustrates only the general orientation of the fibers and polymer particles. However, this figure is not to scale and does not show the actual aspect ratio of the fibers of the present invention and the relative size of the polymer particles, which dimensions may be readily determined from the disclosure herein.

In order to accomplish the method of the present invention, a laboratory apparatus such as that shown in FIG. 3 may be used. As illustrated in FIG. 3, the dispersion or blend 13 of polymer particles and fibers is contained within a beaker 14 and is drawn through a Beuchner funnel 16 having a 40/60 weave screen 18 carried in its base which serves as a filter. A cylindrical adapter 20 is used to draw the dispersion of materials from the beaker 14 into the funnel 16 when vacuum is applied. A tube 21 connects the exit end of the funnel 16 to a conventional Erlenmeyer flask 22, passing through a plug 23. A port 24 in the flask 22 is connected to a vacuum-producing means (not shown), such as a small capacity laboratory pump. The vacuum

produced forces the dispersion 13 of materials in the beaker 14 up into the filter 18, with the liquid being drawn through the tube 21 and directed into the base of the flask 22. As the fibers are drawn into the funnel 16, they assume the quasi-isotropic orientation illustrated in FIG. 2, with the polymer particles 12 caught between the fibers 11, to form the intermediate structure 10 shown in FIG. 1. In addition, the filtered mixture assumes the shape of the filtering surface and surrounding structure. This intermediate structure 10 is removed from the filter and dried under vacuum at an elevated temperature and for a period of time sufficient to remove moisture and excess carrier liquid (e.g. 150°F or 66°C for two hours at 20 millimeters of mercury for an isopropyl alcohol/water carrier liquid and 1900 UHMW polymer). The dried mixture is compacted at ambient temperature under pressure, such as 5,000 pounds per square inch (34 x 10^6 pascals), to remove entrapped air. FIG. 4 presents a magnified schematic view of a portion of the compacted mixture, comprising the fibers 11 which are intertwined and oriented in an essentially two-dimensional network, with a continuous matrix of polymer (not illustrated) dispersed throughout the spaces between the fibers. As previously discussed, the continuous polymer matrix is formed during compaction by the interpenetration of the polymer particles with each other on a macroscopic level. As discussed with respect to FIG. 2, FIG. 4 is also of a schematic nature only and not to scale.

Finally, the compacted mixture is compressed at 5000 psi, for example, in a mold of the desired shape, and heated at or above the fusion/melting temperature of the specific polymer used (e.g. 400°F or 204°C for 1900 UHMW polymer), and then cooled so that the polymer particles are fused together and bind the unmelted

fibers together in the polymer. The specific time, temperature, and pressure required for the process steps described herein depend upon the particular polymer used in the matrix. The resulting product may be in the form of a disk 28 which is illustrated in FIG. 5, and comprises a continuous polymer matrix reinforced by a network of high-aspect-ratio fibers randomly oriented primarily in two dimensions and mechanically entwined and interlocked. Such a structure has inherent multi-directional structural properties due to the high degree of fiber entanglement. Thus, the fiber network restricts movement of the resin matrix with little or no stress transfer at the fiber/matrix interfaces. By minimizing the need for a strong fiber/matrix bond, the present invention enables the use of new combinations of fibers and polymers to produce new composite materials and structures. In addition, the process of the present invention enables low- or non-flow polymers to be used as the matrix, which could not be achieved by known processes for composite fabrication. Further, the polymer/fiber composite of the present invention possesses the self-lubricating, low-friction properties of the polymer material, as well as increased wear resistance and creep resistance due to the fiber network structure discussed above.

From this disk 28 of FIG. 5 may be made an annular member 30, as shown in FIG. 6, by machining out the central portion of the disk 28. The edges of the member 30 may be rendered smooth by placing the ring in a suitable die and compressing it at an elevated temperature of, for example, 350°F (177 °C). Other structures having different geometric configurations may also be formed from the polymer/fiber composite of the present invention using known molding and forming

techniques. In addition, the suspension may be filtered into a structure having a different geometry than that illustrated herein to form a structure other than a disk.

EXAMPLE 1

A composite material was formed in accordance with the present invention as described herein in detail using ultra high molecular weight polyethylene (1900 UHMW polymer) and Kevlar fibers in an alcohol/water carrier.

A mixture was prepared from 6.8 grams of 1900 UHMW polymer having a molecular weight of 2×10^6 to 6×10^6 and a particle size of about 300 micrometers in diameter, and 1.2 grams of chopped Kevlar fibers having lengths of 1/8 inch (3175 micrometers), an aspect ratio of about 250:1, and a diameter of about 12 micrometers, in 300ml. of a 2:1 mixture of isopropyl alcohol and water. The mixture was blended in a Waring blender using a flat disk type blade for 2 minutes. Using the apparatus discribed in FIG. 3, the dispersion of fibers and polymer in the liquid carrier was placed in the beaker 13. A vacuum of about 20 inches of mercury (3.4×10^3 pascals) was drawn on the dispersion for about 20 seconds until all of the dispersion had been drawn up from the beaker 13. After filtration, the mixture which had collected on the wire mesh was dried at 150°F (66°C) for 2 hours under a vacuum of 20 inches of mercury (3.4×10^3 Pa). The dried material was then compressed at ambient temperature at 5000 pounds per square inch (34×10^6 Pa) for 5 minutes to form a compacted preform. Then the preform was placed in a mold which was heated to and maintained at a temperature of 400°F (204°C). Contact pressure was

applied to the mold, and the mold was then cooled at a rate of 10°F/minute (6°C/minute) down to ambient temperature. Pressure was applied slowly as the mold and preform cooled. A maximum pressure of 5000 psi (34×10^6 Pa) was reached by the time the material had cooled to 200°F (93°C), which was 20 minutes after the cooling was started. The molded product was then removed from the mold. The final product was in the form of a disk, as shown in FIG. 5.

The disk of final product was tested for wear resistance by rubbing the disk against a stainless steel countersurface at about 190 pounds per square inch (1.3×10^6 Pa) pressure and a rubbing speed of 17 feet per minute (5.2 m/minute), and was found to wear at a comparable rate to neat ultra-high molecular weight polyethylene. In addition, this product was tested for creep properties by compressing it at 3000 psi (21×10^6 Pa) at 150°F (66°C) for 24 hours with the load applied perpendicular to the structure tested. Under these test conditions, a 0.5 inch (1.27 cm) cube of neat polyethylene deformed 0.16 inch (0.41 cm), as contrasted to the composite material of the present invention which deformed only 0.011 inch (0.027 cm). Further, since the product of the present invention comprises polymeric fibers which are softer than glass reinforcing materials conventionally used, the product of this invention has improved nonabrasive properties as compared to many conventional composites.

EXAMPLE 2

A composite material may be formed in accordance with the present invention as set forth in Example 1, except using polytetrafluoroethylene (PTFE) as the ultra-high molecular weight material. Since PTFE is more dense than ultra-high molecular weight polyethylene, the combination of polymer particle size, fiber size, and liquid carrier is adjusted so that a good dispersion of these materials is formed, as previously described.

The previous description presents the best mode contemplated of carrying out the present invention. This invention is, however, susceptible to modifications and alternate constructions from the embodiments shown in the drawings and described above. Consequently, it is not intended to limit this invention to the particular embodiment disclosed, but rather to cover all modifications and alternate constructions falling within the intention and scope of the invention as expressed in the appended claims. In particular, it is not intended to limit the present invention to the particular polymers disclosed herein by way of example, but to include any such polymers or polymer blends having low flow properties which render them incapable of being combined with randomly oriented, intertwined, high aspect ratio fibers by conventional techniques. Further, it is not intended to limit the process of the present invention to the particular process details disclosed herein, but to include variations therein which would be required for the use of materials other than those specified herein.

CLAIMS

What is Claimed is:

1. A composite material comprising an ultra-high molecular weight solid polymer matrix having randomly dispersed therein reinforcing fibers of lengths greater than about 0.05 inch (1270 micrometers).

2. The composite of Claim 1 wherein the fibers are randomly oriented primarily in two dimensions.

3. The composite of Claim 1 wherein the polymer has a molecular weight in excess of 1.0×10^6.

4. The composite of Claim 3 wherein the polymer is selected from the group consisting of polyethylene and polytetrafluoroethylene.

5. The composite of Claim 1 wherein the fibers are made of an organic polymer.

6. The composite of Claim 5 wherein the fibers are made of an aromatic polyamide material.

7. The composite of Claim 1 wherein the fibers are selected from the group consisting of boron, glass, graphite, quartz, and metal.

8. The composite of Claim 1 wherein the fibers have an aspect ratio within the range of about 100:1 to about 300:1.

9. The composite of Claim 1 wherein the mixture of fibers and polymer ranges between 5 and 60 percent by weight fiber and between 40 and 95 percent by weight polymer.

10. The composite of Claim 9 wherein the mixture of fibers and polymer ranges between 5 and 15 percent by weight fiber and between 85 and 95 percent by weight polymer.

11. The composite of Claim 1 wherein:

a) said polymer is polyethylene having a molecular weight in the range of 2×10^6 to 6×10^6 and a particle size of about 300 micrometers in diameter; and

b) said fibers are formed of an aromatic polyamide material and have lengths of about 3175 micrometers and as aspect ratio of about 250:1.

12. An article of manufacture comprising a structure formed from the composite material of Claim 1.

13. The article of manufacture of Claim 12 wherein said article comprises an annular member.

14. A method for forming the composite of Claims 1-11, comprising the steps of:

a) providing the ultra-high molecular weight polymer in the form of particles of predetermined size;

b) providing an inert carrier liquid capable of uniformly dispersing the fibers and the particles of the polymer;

c) blending the fibers and the particles of the polymer in the inert carrier liquid to form a uniform dispersion;

d) passing the dispersion through a filter so that there is collected on the filter an intermediate structure comprising a network of fibers oriented randomly in a quasi-isotropic arrangement with the particles of polymer dispersed throughout the network;

e) drying the intermediate structure to remove excess liquid carrier therefrom;

f) applying positive pressure to the intermediate structure at ambient temperature to remove entrapped air and to compact the intermediate structure; and

g) compressing the compacted intermediate structure in a mold at an elevated temperature at which the polymer melts but the fibers do not melt, to form upon cooling a fiber-reinforced polymer composite.

15. The method of Claim 14 wherein the mixture of fibers and polymer comprises no more than about 3 percent by weight of the blend of liquid carrier and fiber/polymer.

16. The method of Claim 14 wherein:

a) said polymer is polyethylene having a molecular weight in the range of 2×10^6 to 6×10^6 and a particle size of about 300 micrometers in diameter;

b) said fibers are formed of an aromatic polyamide material and have lengths of about 3175 micrometers and an aspect ratio of about 250:1;

c) said positive pressure for compacting is about 5000 pounds per square inch (34×10^6 pascals);

d) said compressing comprising applying a positive pressure of 5000 pounds per square inch (34×10^6 pascals) maximum; and

e) said elevated temperature is about 204°C (400°F).

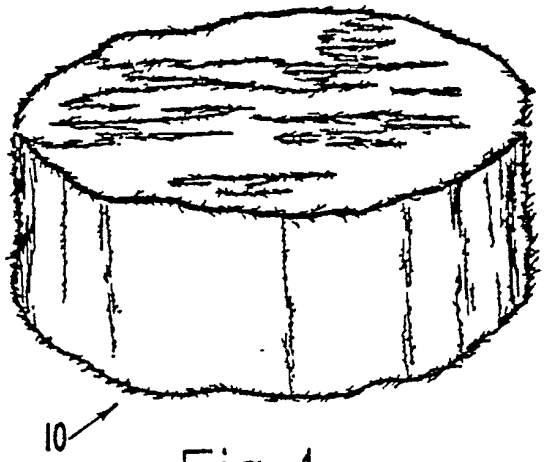


Fig. 1.

Fig. 2.

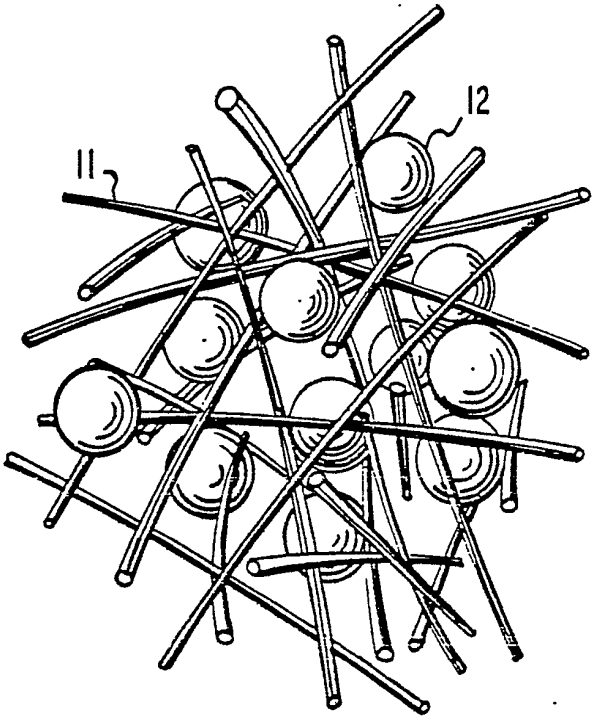
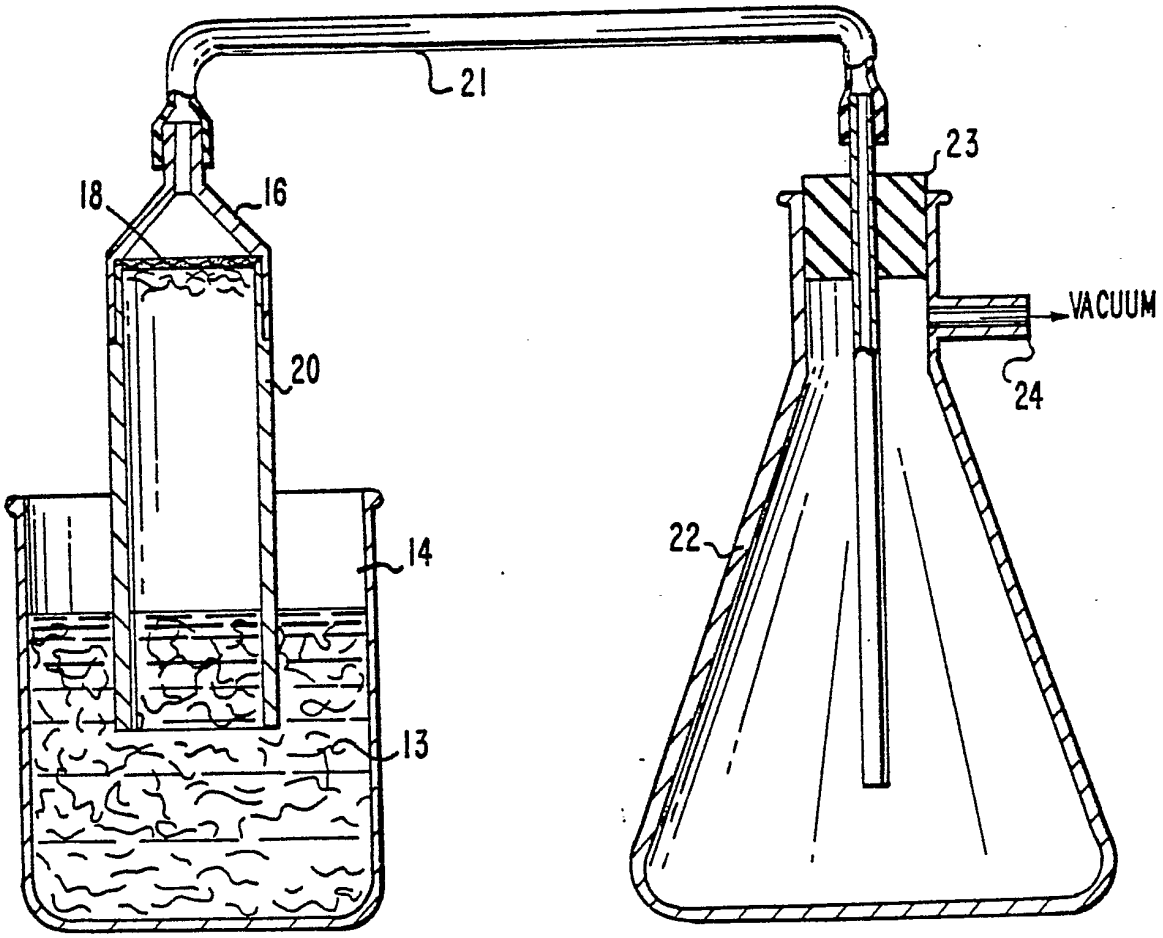


Fig. 3.



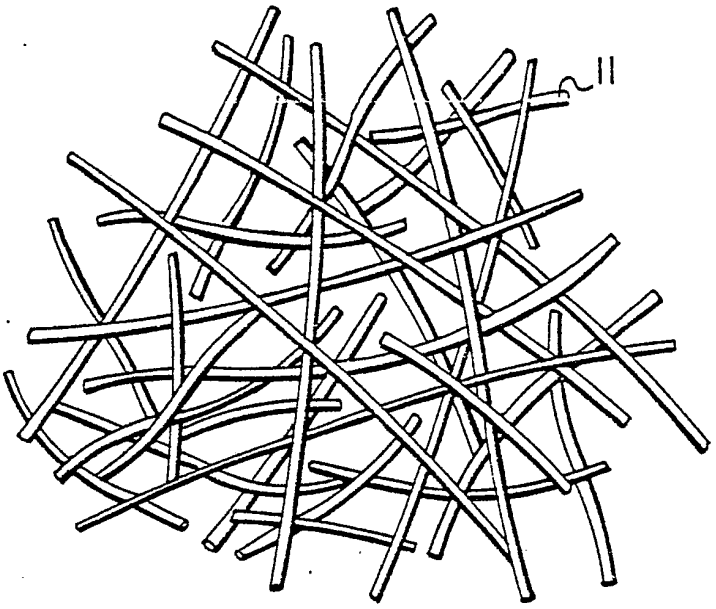


Fig. 4.

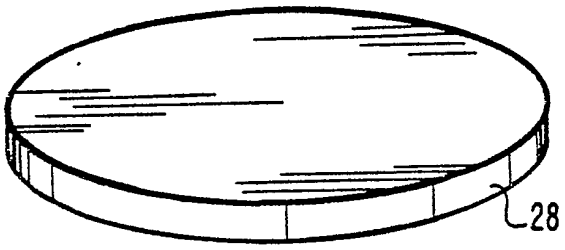


Fig. 5.

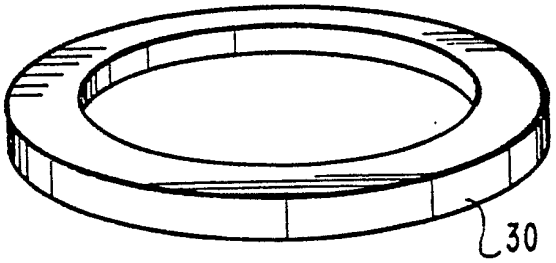


Fig. 6.

INTERNATIONAL SEARCH REPORT

International Application No PCT/US 85/00612

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC[4]: C 08 K 7/02; C 08 J 5/04; C 08 L 23/06; C 08 L 27/18

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC[4]	C 08 K C 08 L C 08 J

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category [10]	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
A	GB, A, 2014158 (DU PONT) 22 August 1979, see claims 1-2,8-12; page 1, lines 22-26	1-16
A	Chemical Abstracts, volume 86, nr. 24, 13 June 1977, Columbus, Ohio, (US) P. Walker et al.:"Variables affecting polymer wear in artificial human joints", page 376, abstract 177281u & Polym. Sci. Technol. 1974, vol. 5B, pages 553-67	1

* Special categories of cited documents: [14]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
14th October 1985	04 NOV. 1985
International Searching Authority	Signature of Authorized Officer
EUROPEAN PATENT OFFICE	G.L.M. Kruyssen