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(54) **COMPOSITE MATERIAL**

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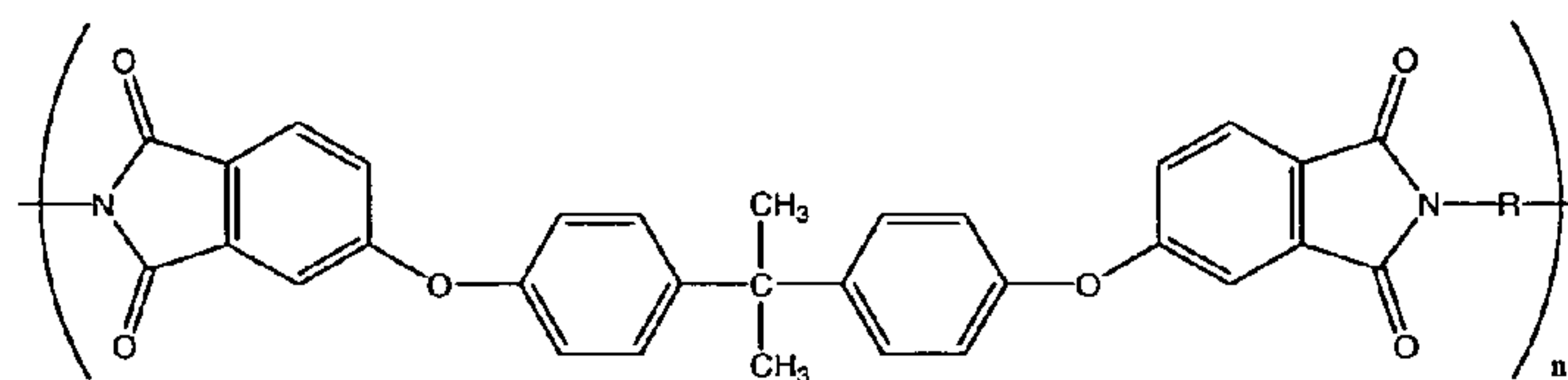
(57) **ABSTRACT**

A composite material comprises a matrix material, for
example, polyetheretherketone, and a first fibrous material
which includes alumina, such as alumina fibres. The mate-
rial may be used in applications where strength and dura-
bility are required, such as in CMP.

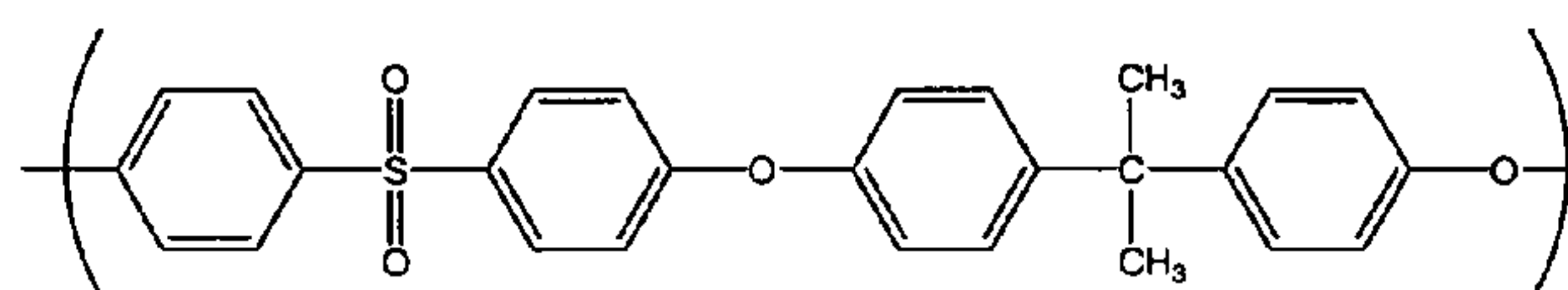
Fig. 1

PEI (polyetherimide)

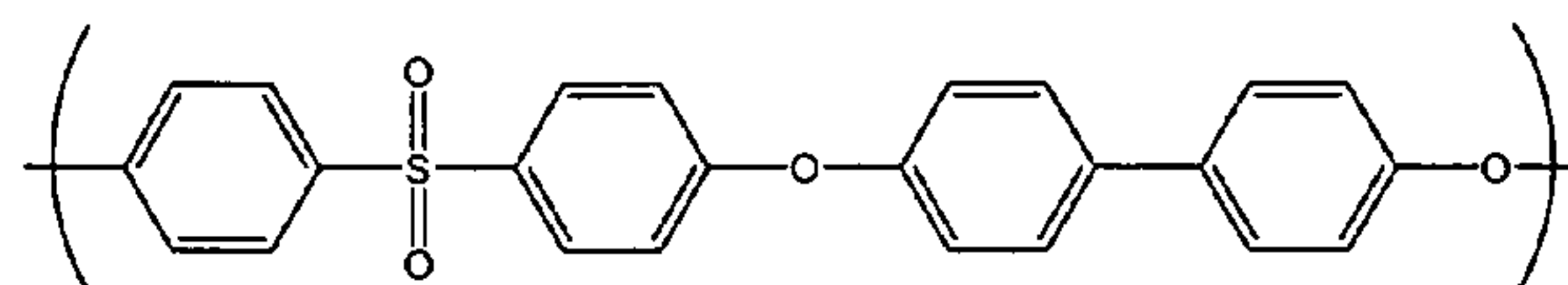
R = aryl, alkyl, aryl
ether or alkylether



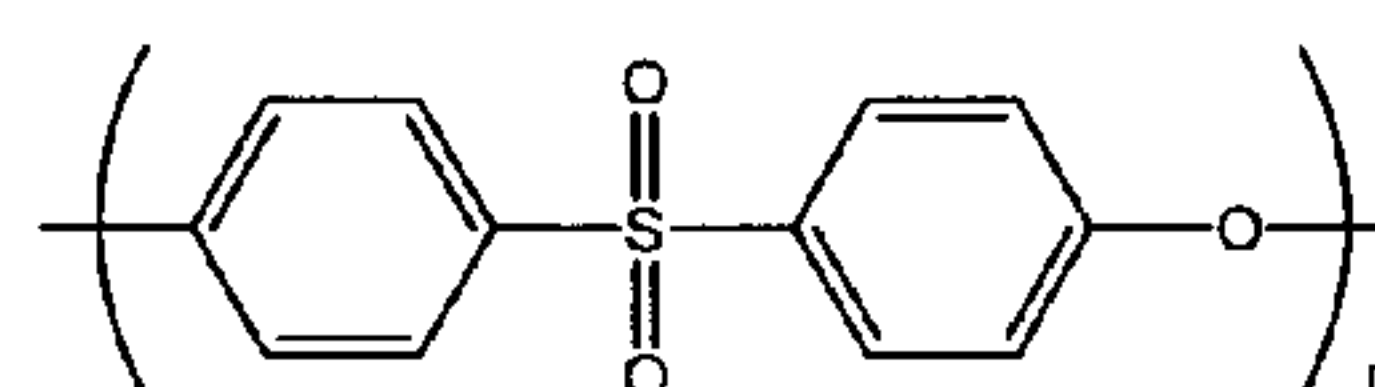
Udel polysulfone



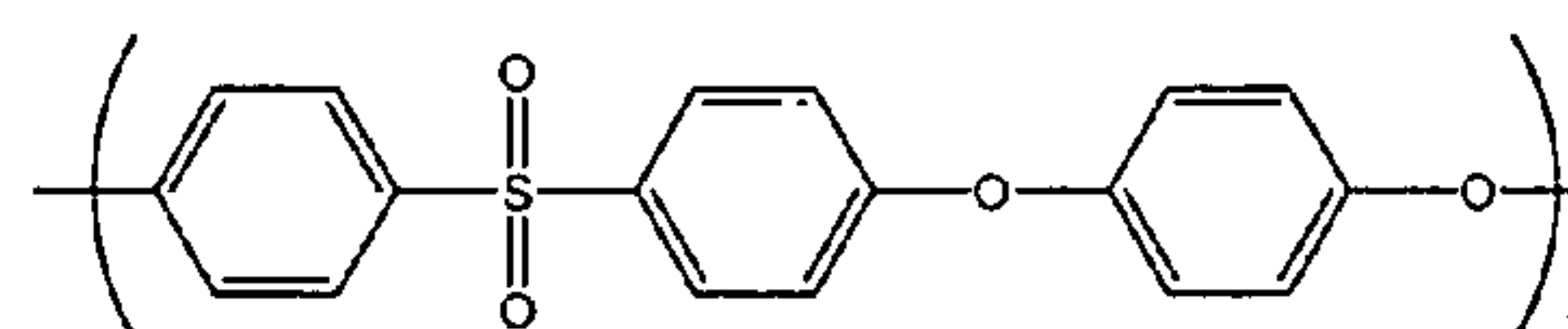
Radel R
polyphenylsulfone



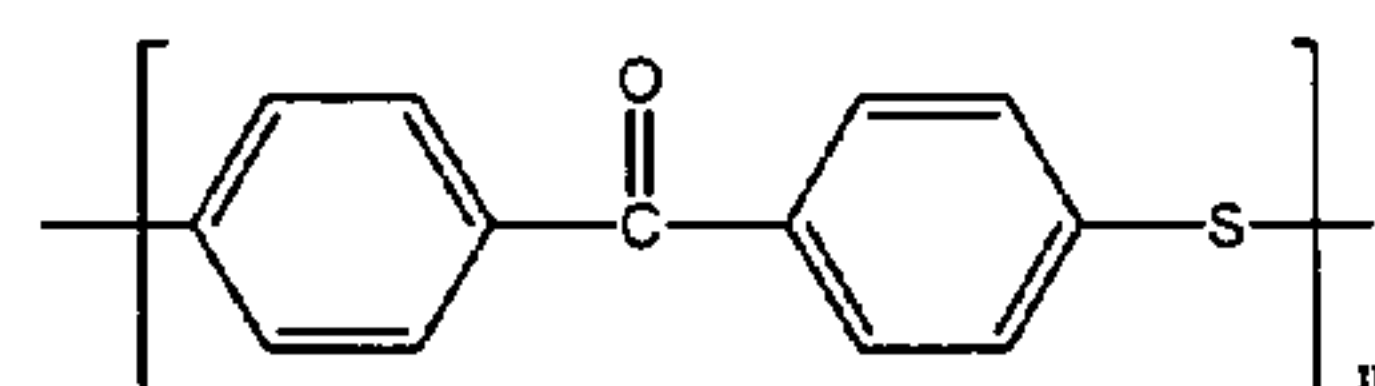
Radel A
polyethersulfone



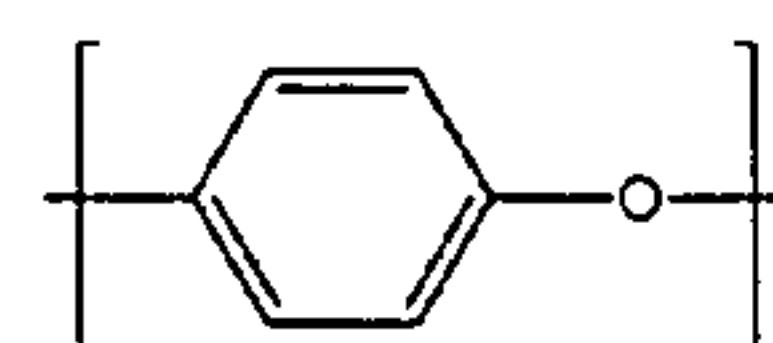
and



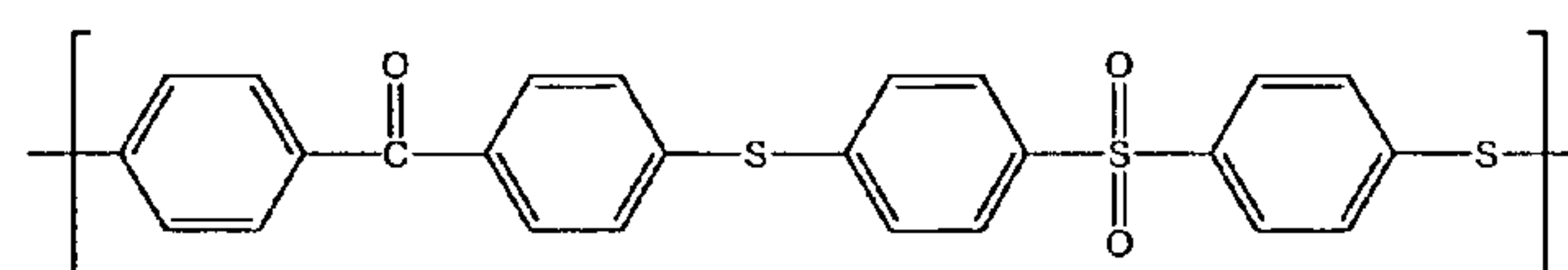
PBPS
(poly(benzophenone
sulfide))



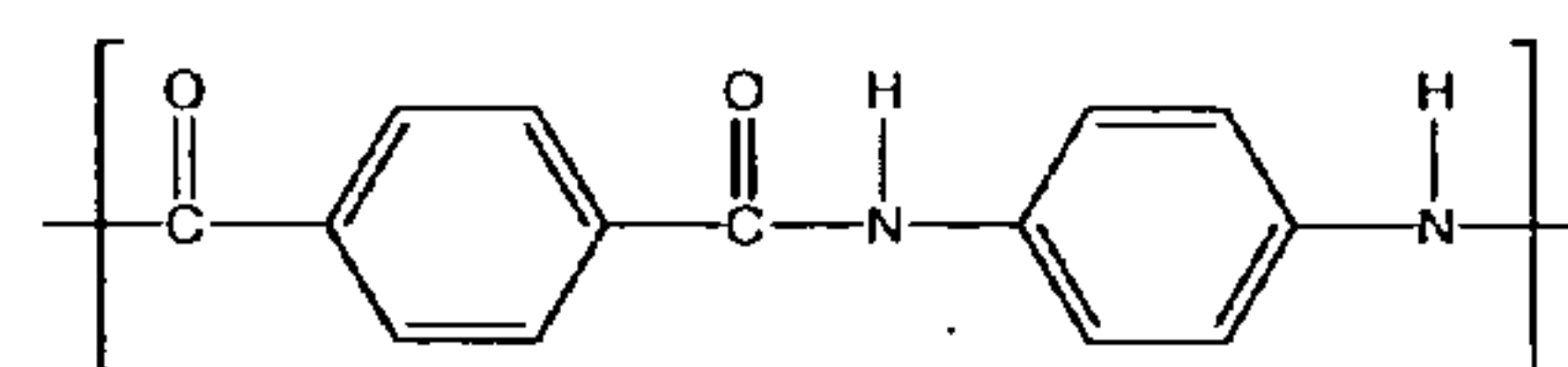
(PPO) poly(1,4-
phenylene oxide)



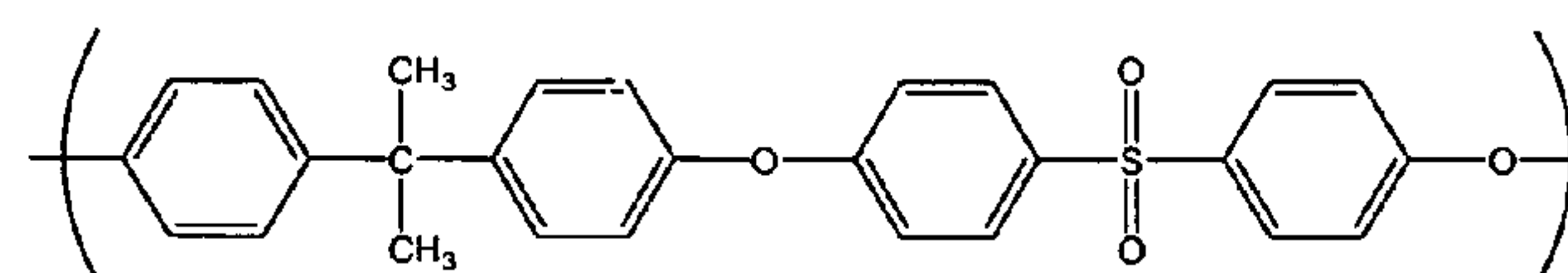
Poly(benzophenone
sulfide-phenylsulfone-
sulfide)



PAR (polyaramid or
Kevlar®)



PSU (polysulfone)



PES (polyether sulfone)

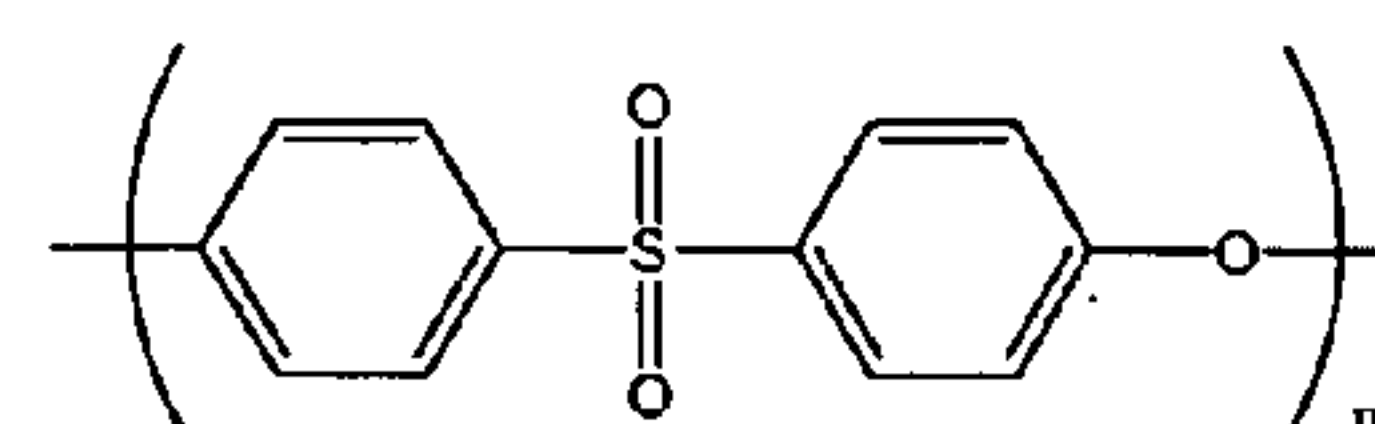
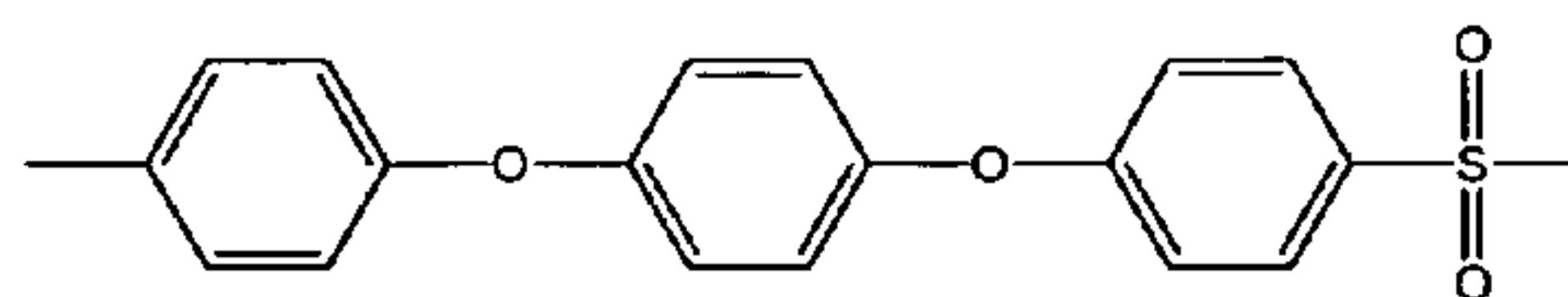
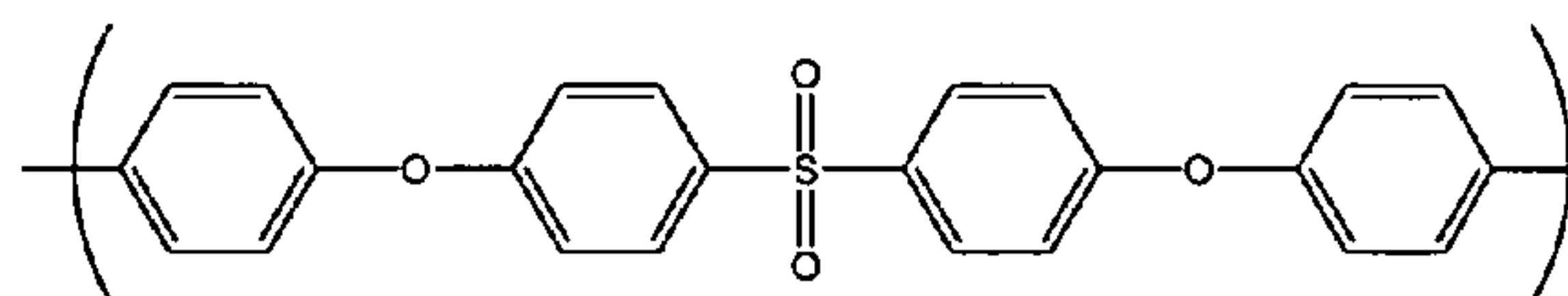


Fig. 1 (continued)

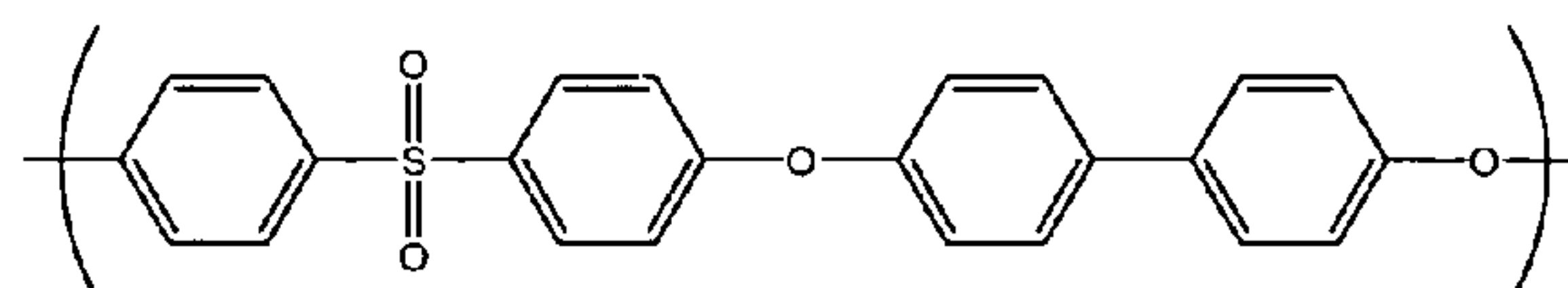
PEES (polyether-ether sulfone)



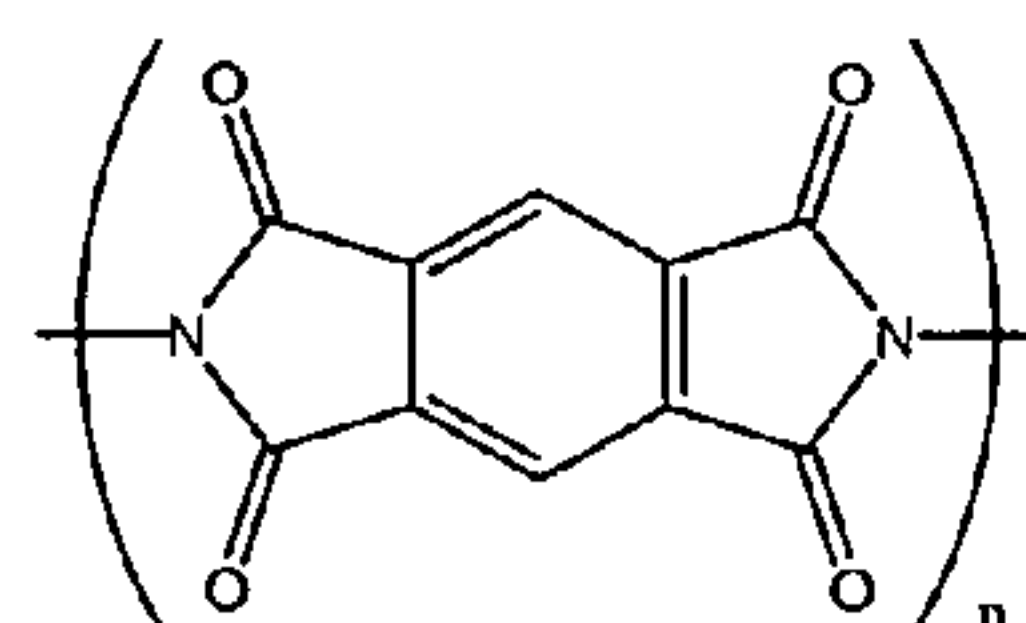
PAS (polyarylether sulfone)



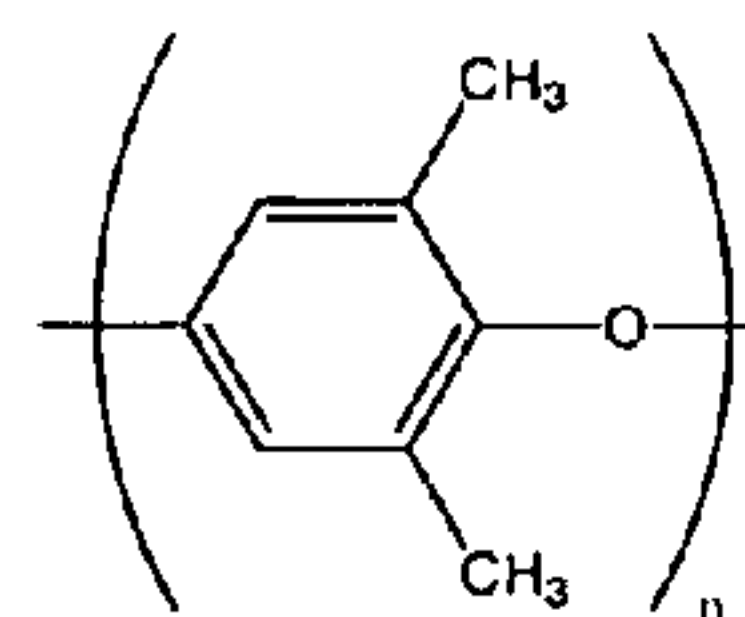
PPSU (polyphenylene sulfone)



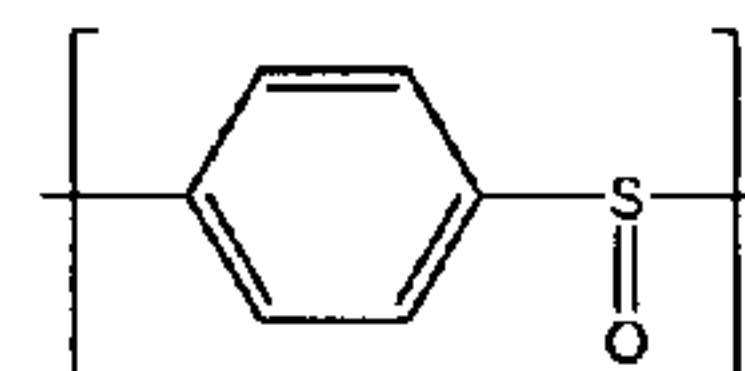
PI (polyimide)



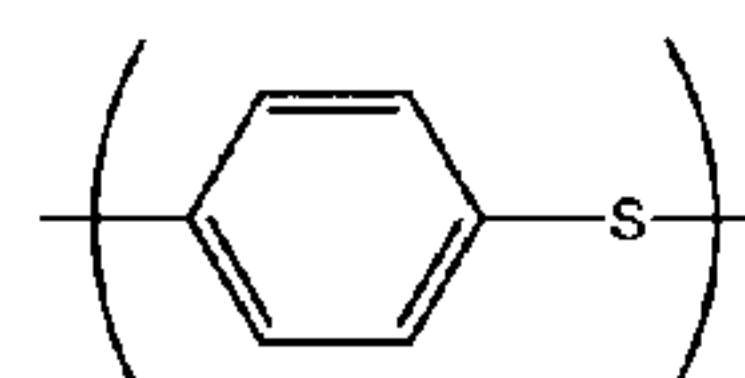
PPO (polyphenylene oxide)



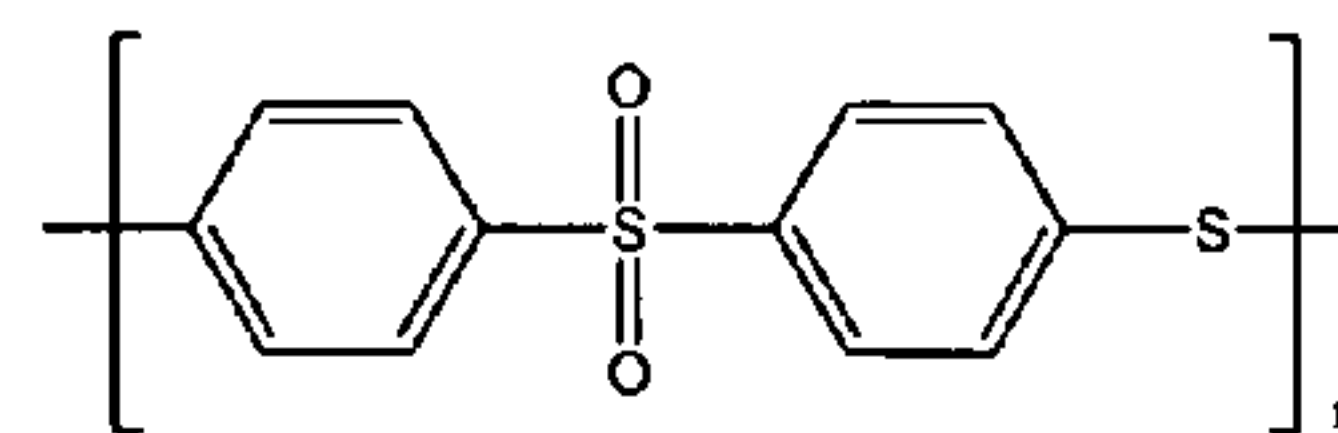
PPSO (polyphenylene sulfoxide)



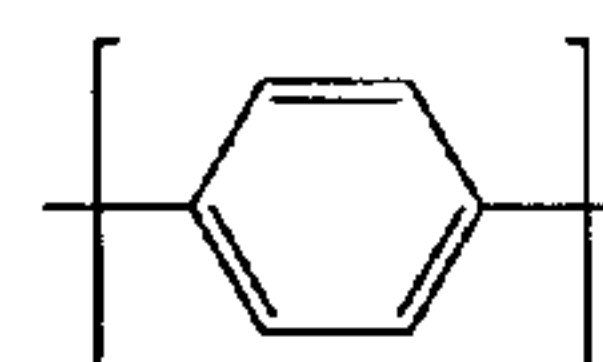
PPS (polyphenylene sulfide)



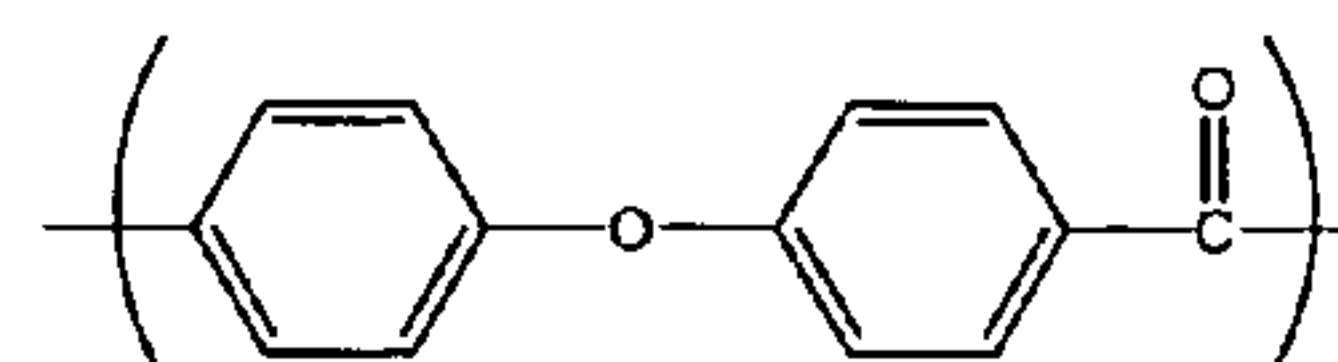
PPS / SO₂
(polyphenylenesulfide sulfone)



PPP
(polyparaphenylene)



(polyetherketone)



(polyetheretherketone)

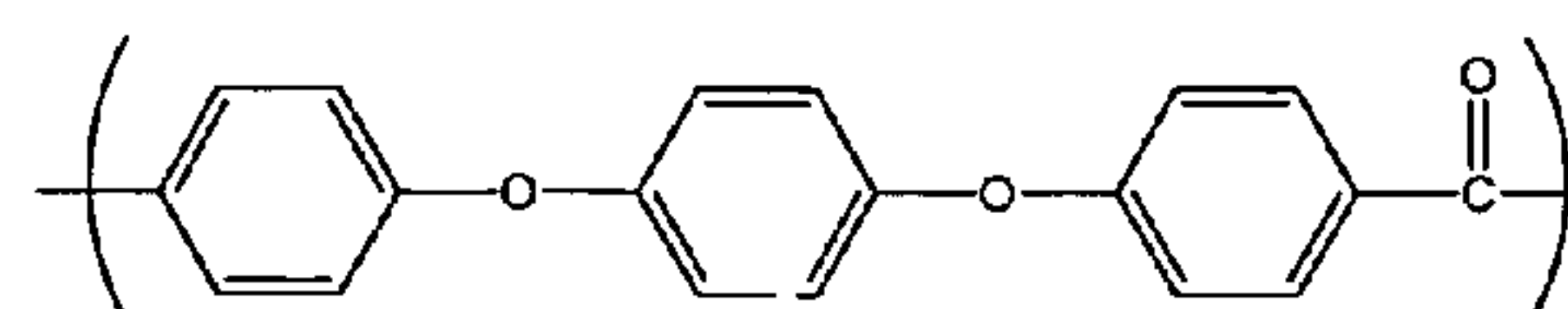
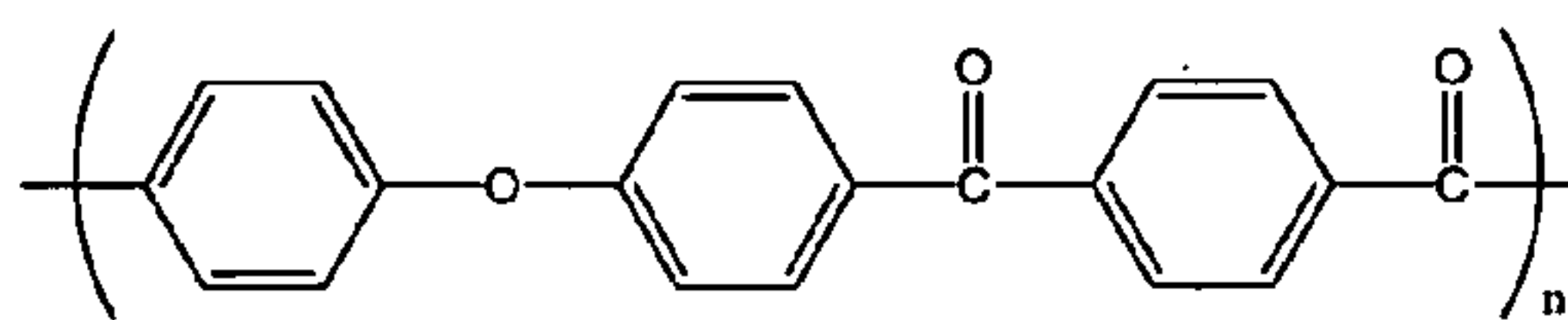
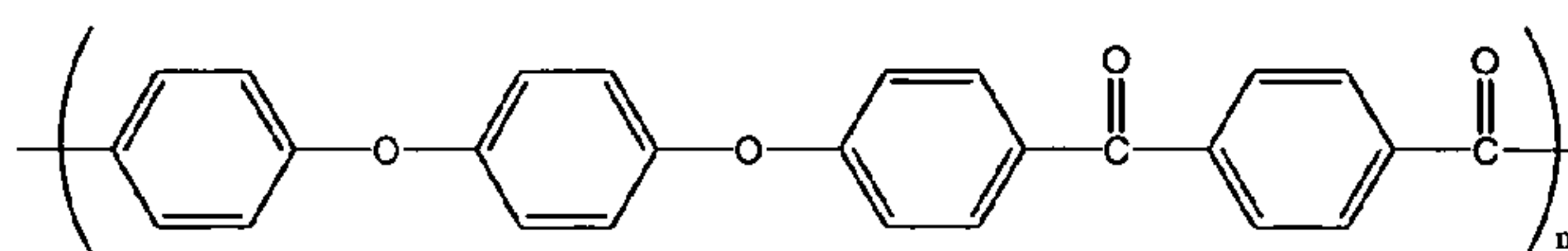


Fig. 1 (continued)

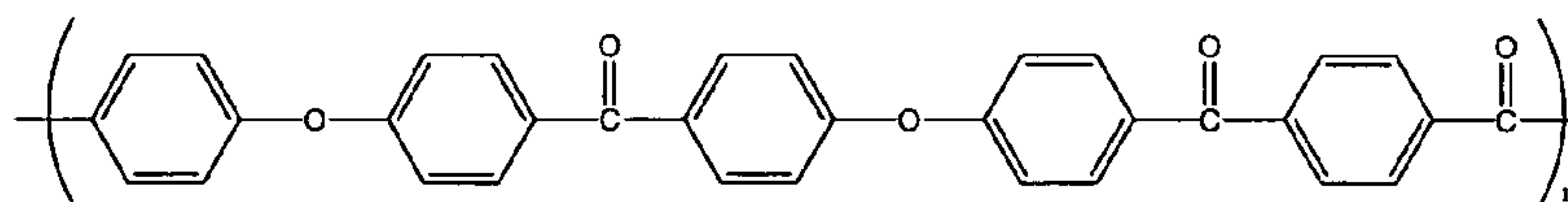
(polyetherketoneketone)



(polyetheretherketone-
ketone)



(polyetherketone-
etherketone-ketone)



COMPOSITE MATERIAL

[0001] This invention relates to a composite material, its preparation and uses thereof.

[0002] As technology progresses, new materials are required to have increased strength and durability to meet more demanding applications. For example, materials may be required for use in an abrasive environment such as during the chemical mechanical polishing (CMP) wherein electronic parts are made.

[0003] CMP is used in the semi-conductor industry for planarizing wafers. In conventional CMP techniques a wafer carrier assembly is in contact with a polishing pad mounted on a CMP apparatus. The wafers are typically mounted on a carrier or polishing head which provides a controllable pressure urging the wafers against the rotating polishing pad. The pad has a relative movement with respect to the wafer driven by an external driving force. Thus, the CMP apparatus effects polishing or rubbing movement between the surface of each thin semiconductor wafer and the polishing pad whilst dispersing a polishing slurry containing abrasive particles in a reactive solution to effect both chemical activity and mechanical activity while applying a pressure between the wafer and the polishing pad. U.S. Pat. No. 6,468,136 B1 (Applied Materials, Inc) describes a method of CMP on a substrate surface which involves mounting the substrate in a carrier comprising a retaining ring having a mechanical hardness greater than 85 durometer and then subjecting the surface to CMP using a polishing pad having a hardness of less than about 60 durometer. The document advocates use of retaining rings comprising a polymer reinforced with carbon fibres. Whilst fibre reinforced polymers having relatively high hardness may be made and are well known, disadvantageously, in some situations, any wear of retaining rings made from such fibre reinforced polymers may be problematic. In particular, carbon and carbon fibres are electrically conductive and even small levels may detrimentally contaminate semi-conductor wafers that may be polished in CMP. Furthermore, carbon/carbon fibres may be abrasive and therefore scratch the wafer surface. Thus, whilst wear rates of carbon or carbon fibre-based materials may be relatively low, even a relatively small level of wear may necessitate replacement of a retaining ring made out of the composite material and loss of some production.

[0004] In the manufacture of vehicles or parts thereof, for example engines, there is an ongoing desire and/or need to replace metal parts with parts made out of lighter materials such as plastics materials. Thus, it is an ongoing problem to produce plastics based composite materials for use in a wide range of applications including vehicular applications.

[0005] It is an object of the present invention to provide an improved composite material for use in a wide range of applications.

[0006] According to a first aspect of the invention, there is provided a composite material comprising a matrix material and a first fibrous material which includes alumina.

[0007] Said first fibrous material preferably comprises, more preferably consists essentially of, alumina fibres.

[0008] Said first fibrous material is preferably discontinuous.

[0009] Said first fibrous material preferably has a resistivity of greater than 10^6 ohms per sq.

[0010] Said first fibrous material is preferably a substantially non-fibrillated fibre.

[0011] Said first fibrous material suitably comprises some fibres having diameters in the range 3 to 10 μm , preferably in the range 3 to 6 μm . Suitably, said first fibrous material includes less than 10%, preferably less than 5%, especially less than 1% of fibres having a diameter of less than 1 μm . Suitably, greater than 50%, preferably greater than 75%, more preferably greater than 90% of the fibres in said first fibrous material have a diameter in the range 3 to 10 μm , especially in the range 3 to 6 μm . Suitably, less than 10%, preferably less than 5%, more preferably less than 1% of fibres in said first fibrous material having a diameter greater than 10 μm .

[0012] The use of a first fibrous material which comprises fine diameter material facilitates the preparation of a first component having ceramic properties.

[0013] Said matrix material preferably comprises an engineering resin, for example a tough semi-crystalline resin. Examples are provided in **FIG. 1** hereinafter.

[0014] Said matrix material preferably comprises a polyether, especially an aromatic polyether, for example an aromatic polyetherketone or polyethersulphone. Preferred polyethers are those shown in **FIG. 1**.

[0015] Said matrix material may include a polyarylene sulphide.

[0016] Said matrix material preferably comprises a polyaryletherketone or sulphone. Said matrix material preferably comprises a polyaryletherketone, with polyetherketone, polyetheretherketone and polyetherketoneketone being preferred and polyetherketone and polyetheretherketone being especially preferred. Polyetheretherketone is the most preferred matrix material. Said matrix material may comprise a mixture of polyetheretherketone and polyarylene sulphide. Preferably, said matrix material consists essentially of polyetheretherketone.

[0017] Suitably at least 50 wt %, preferably, at least 80 wt %, more preferably at least 90 wt %, especially 100 wt % of said matrix material is made up of a single type of engineering resin.

[0018] Said composite material may include at least 20 wt %, suitably at least 25 wt %, preferably at least 30 wt %, more preferably at least 35 wt %, especially at least 40 wt % of said matrix material. The amount of said matrix material may be 80 wt % or less, preferably 70 wt % or less, more preferably 65 wt % or less, especially 60 wt % or less.

[0019] Said composite material may include at least 15 wt %, suitably at least 25 wt %, preferably at least 30 wt %, more preferably at least 35 wt %, especially at least 40 wt % of said first fibrous material. The amount of said first fibrous material may be 80 wt % or less, preferably 70 wt % or less, more preferably 60 wt % or less, especially 50 wt % or less.

[0020] The ratio of wt % of said matrix material to the wt % of said first fibrous material is suitably in the range 0.3 to 3, preferably in the range 1 to 2, more preferably 0.6 to 1.5.

[0021] In one embodiment, said composite material may comprise a said matrix material, a said first fibrous material and a second fibrous material. Preferably, said second fibrous material is other than carbon fibre. Although, said second fibrous material may be chemically the same as said first fibrous material (but may be in a different physical form), it is preferably a chemically different material. Preferably, said second fibrous material is physically different from said first fibrous material. Said second fibrous material is preferably fibrillated. Said second fibrous material is preferably a pulp. Said second fibrous material is preferably a polyaramid. Said composite material may include less than 55 wt %, suitably less than 25 wt %, preferably less than 15 wt %, more preferably less than 10 wt %, especially less than 6 wt % of said second fibrous material. In preferred embodiments, the composite material includes at least 0.5 wt %, preferably at least 1 wt %, more preferably at least 2 wt %, especially at least 3 wt % of said second fibrous material. The ratio of the wt % of said second fibrous material to said first fibrous material in said composite is suitably in the range 0 to 1, is preferably in the range 0.005 to 0.7, is more preferably in the range 0.01 to 0.5 and, especially, is in the range 0.03 to 0.15. Said second fibrous material preferably has a specific surface area of greater than 5 m²/g, more preferably greater than 7 m²/g, especially greater than 8 m²/g. The specific surface area of the second fibrous material may be in the range 5-14 m²/g, preferably 7-12 m²/g.

[0022] The sum of the wt % of all fibrous materials (including said first and second fibrous materials and any further fibrous materials) in said composite may be at least 15 wt %, suitably at least 25 wt %, preferably at least 30 wt %, more preferably at least 35 wt %, especially at least 40 wt %. The sum of the wt % may be 80 wt % or less, preferably 70 wt % or less, more preferably 60 wt % or less. The ratio of the wt % of said matrix material to the sum of the wt % of all fibrous materials in said composite is suitably in the range 0.3 to 6, preferably 0.3 to 3, more preferably, 0.6 to 1.5.

[0023] According to a second aspect of the invention, there is provided a method of manufacturing a first component, the method comprising forming the first component from a composite material in accordance with said first aspect or otherwise as described herein.

[0024] Preferably, in the method, said first component is formed from said composite material by injection moulding or extrusion. This may involve use of injection moulder or extruder barrel temperatures of at least 100° C., preferably at least 200° C., more preferably at least 300° C. and especially at least 350° C.

[0025] Advantageously, composite materials described (especially ones comprising 55 to 65 wt % of matrix material, e.g. polyetheretherketone; and 35 to 45 wt % of a said first fibrous material which comprises alumina fibres) can be machined into desired shapes for example to define nozzles or other structures having openings and or which have complex shapes which cannot easily be made by injection moulding or extrusion alone. Thus, the method may include machining a precursor of said first component to manufacture said first component.

[0026] Said first component may include an opening which may have a diameter of less than 0.5 m, preferably

less than 0.1 m, especially less than 0.05 m. The diameter may be at least 0.1 mm, preferably at least 0.5 mm, especially at least 1 mm.

[0027] Said first component may be selected from a bearing, a nozzle, a ring, a washer (e.g. a thrust washer), and any part of any of the aforesaid.

[0028] In one embodiment, said first component may include a composite material comprising 50 to 70 wt % (more preferably 55 to 65 wt %, especially 58 to 62 wt %), of matrix material (e.g. a polyether such as polyetheretherketone or other preferred matrix materials as described herein) and 30 to 50 wt % (more preferably 35 to 45 wt %, especially 38 to 42 wt %) of alumina fibres.

[0029] The method of the second aspect may be for manufacturing a first component which is arranged to be subjected to an abrasive force in use when incorporated into a device.

[0030] Said first component may be arranged to be contacted in use by a second component wherein said first and second components are movable relative to one another so that a frictional force acts between the first and second components. Said first and second components are preferably made of different materials.

[0031] Said first component may be arranged to be contacted in use by a second component which comprises an abrasive fluid or a solid which would tend to abrade the first component when contacting it. An abrasive fluid will be more abrasive than air. It may be arranged to move relative to the first component at a velocity of at least 0.1 m/s, suitably at least 1 m/s, preferably at least 5 m/s, especially at least 10 m/s. In some cases, the velocity may be greater than 100 m/s or even 200 m/s. A said abrasive fluid may have a temperature of greater than 30° C., preferably greater than 100° C., more preferably greater than 200° C. A said abrasive fluid may comprise a gas or a liquid. A said abrasive fluid may include solid particles which may be abrasive in use. A said abrasive fluid may comprise a fluid jet. A said abrasive fluid may be at a pressure of greater than ambient pressure, for example of at least 10 atmospheres. Said abrasive fluid may comprise a plasma jet.

[0032] When said second component is a solid, the solid may be made of any other type of material. For example, it could be a metal or a plastics material.

[0033] For example, said first component may comprise said composite material and said second component may comprise a different composite material or may comprise a said matrix material as described herein in the absence of a said fibrous material. In use in the device, a fluid may contact the first and second components. The fluid may be a lubricant or a chemical polishing fluid. Alternatively, in some cases, no fluid may be used.

[0034] Preferably, said first component is such that, in use, when subjected to a said abrasive force, it is also subjected to a temperature above ambient temperature. The temperature it is subjected to may be greater than 30° C., preferably greater than 50° C., preferably greater than 100° C. or even greater than 150° C.

[0035] A first component as described may be used in close association with electronic parts. Advantageously, the composite material, when abraded, may be substantially

non-electrically conductive (unlike composite materials which include a substantial amount of carbon fibre) so the abraded material may have little detrimental effect on the electronic part. Thus, in one preferred embodiment, said method is a method of manufacturing a first component for use in the manufacture of an electronic part, for example in the manufacture of integrated circuits and/or semi-conductor devices such as semi-conductor wafers. Suitably, said first component may be used in and/or during the treatment of a substrate, for example in an abrasive treatment thereof, such as chemical mechanical polishing (CMP). In this case, said first component may comprise a retaining means, for example a retaining ring, for retaining a substrate to be subjected to the treatment (e.g. CMP) in position. In cases wherein the first component is contacted in use by a second component, the second component may be a treatment means, for example a polishing means, arranged to contact the substrate to treat it.

[0036] As described above, composite materials which include carbon fibre may be disadvantageous in, for example, CMP applications. Suitably, said composite material includes less than 20 wt %, preferably less than 10 wt %, more preferably less than 5 wt %, especially less than 1 wt % of carbon fibres. Advantageously, said composite material includes no carbon fibres.

[0037] Said composite material preferably includes less than 20 wt %, preferably less than 10 wt %, more preferably less than 5 wt %, especially less than 1 wt % of fibrous material (preferably of any material) having a resistivity of greater than 10^6 ohms per sq., preferably greater than 10^{12} ohms per sq. Advantageously, said composite material includes no fibrous material (and preferably no component at all) having a resistivity of less than 10^6 ohms per sq.

[0038] A method of manufacturing said composite material of said first aspect may comprise:

[0039] contacting said matrix material and said first fibrous material in a liquid medium so as to provide a mixture of the matrix material and first fibrous material in the liquid medium; and

[0040] removing at least some of the liquid medium thereby to produce a mass comprising matrix material and first fibrous material.

[0041] Preferably, each fibrous material (e.g. said first fibrous material and said second (and any other) fibrous material(s) when provided) are contacted with said matrix material in said liquid medium.

[0042] In the method, said liquid medium may be at a temperature of less than 0°C . when said matrix material and fibrous material(s) are initially contacted with the liquid medium. Preferably, however, said liquid medium is at a temperature of greater than 0°C . The temperature may be greater than 5°C ., preferably greater than 10°C ., more preferably greater than 15°C ., especially 20°C . or greater. The temperature could be greater than 100°C ., preferably less than 80°C ., more preferably less than 50°C ., especially less than 30°C . Advantageously, said liquid medium may be at ambient temperature when said matrix material and fibrous material(s) are initially contacted with liquid medium.

[0043] Preferably, the liquid medium is at ambient pressure when said matrix material and fibrous material(s) are

initially contacted therewith and, preferably, at all times prior to removal of said liquid medium.

[0044] The method suitably involves mixing the matrix material and fibrous material(s) in said liquid medium. Any suitable mixing means may be used. It is preferred to use a mechanical mixing means, for example a paddle mixer. Preferably, the mixing means is selected to minimise forces on the fibrous material(s) which could size reduce the material detrimentally. During said mixing, the mixture may be maintained at a temperature in the range 10°C . to 50°C ., suitably at ambient temperature. Suitably, it is not necessary to apply heat to the mixture during its mixing.

[0045] Preferably, in the method, a slurry of said matrix material and said fibrous material(s) in said liquid medium is produced. Thus, preferably, both the matrix material and fibrous material(s) are substantially insoluble in said liquid medium. Preferably, said matrix material and fibrous material(s) are substantially homogeneously dispersed in said slurry.

[0046] The amount of said liquid medium in said mixture may be selected so that the matrix material and fibrous material(s) can be intimately mixed in the method, suitably so that the matrix and fibrous material(s) are substantially uniformly dispersed throughout the liquid medium. The ratio of the weight of liquid medium to the weight of fibrous material(s) may be at least 5, suitably 10 or more. Such relatively low ratios may be sufficient for fibrous material(s) having relatively short lengths to be dispersed. Where the fibrous material(s) comprise(s) relatively long fibres, the ratio may be higher—it may be 50, 100 or 150 or more. Preferably, however, the ratio is less than 500, more preferably less than 250, especially less than 200.

[0047] Preferably, in the method, the filler material(s) is/are separated, as far as possible, so that there are individual elements of said fibrous material(s) in said mixture—that is, the mixture formed is preferably substantially homogenous.

[0048] Preferably, in the method, the mixture of matrix material and fibrous material(s) is contacted with a support and liquid medium removed from the mixture. Preferably, the support supports the forming composite material as liquid medium is removed therefrom. Preferably, the support includes openings therein via which said liquid medium may be removed, for example by said liquid medium passing from one side to an opposite side of said support. Said support is preferably perforate. It is preferably part of a sieve means.

[0049] Preferably, in the method, liquid medium is allowed to drain away from the forming composite material suitably under the action of gravity. Removal of liquid medium may be vacuum assisted. Suitably, a layer of composite material is formed on said support. In some situations, a further layer may be formed over said layer, for example by contacting said layer with more of said matrix material and fibrous material(s) in said liquid medium.

[0050] Preferably, the method includes a drying stage. Preferably, after contact with said support and suitably after an initial amount of liquid medium has been removed, for example drained away, the formed composite material is dried. This may be effected by application of heat, suitably at a temperature which is greater than the boiling point of

said liquid medium. Said temperature is preferably less than 300° C. and, more preferably, is less than 200° C. Preferably, said formed composite material is dried in an oven.

[0051] The method may include a step of increasing the density of the formed composite material after at least some of said liquid medium has been removed from said mass. This may be achieved by use of a press or other compaction means.

[0052] Said step of increasing the density may be carried out before or after said drying stage.

[0053] In some embodiments, continuous sheets of the composite material may be formed, suitably using pressure to increase the density of the composite material. Parts having desired shapes may be stamped out of or otherwise formed from the sheets.

[0054] Said composite material may be divided up into smaller parts for example having a maximum dimension of less than 5 cm, preferably less than 3.5 cm, more preferably less than 2 cm. Said smaller parts are preferably of a size such that they can feed processing apparatus, for example an injection moulder or extruder which may be used to produce said first component from the composite material. The parts may have a maximum dimension of greater than 0.5 mm, preferably greater than 1 mm.

[0055] Said liquid medium used in the method is preferably unreactive to the matrix material and fibrous material(s) under the conditions of the method. Said liquid medium preferably has a boiling point of less than 200° C., preferably less than 150° C. The boiling point may be at least 50° C., preferably at least 80° C. Said liquid medium could include one or more co-solvents it preferably does not. Thus, preferably said liquid medium consists essentially of water.

[0056] As an alternative to the above method, said composite material may be prepared using a single or twin-screw extruder or any other suitable method of incorporating the fibre into the matrix material.

[0057] According to a third aspect of the invention, there is provided an assembly comprising a first component as described according to said first or said second aspects incorporated into a device.

[0058] In said assembly, said first component is preferably arranged to be subjected to an abrasive force in use. For example, it may be arranged to be contacted in use by a second component which comprises an abrasive fluid or a solid as described according to said first aspect.

[0059] Said device is preferably for use during the manufacture of electronic parts for example in CMP.

[0060] According to a fourth aspect of the present invention, there is provided an electronic part manufacturing process, the process using a first component and/or a device according to the first, second and/or third aspects.

[0061] Preferably, the process involves retaining a substrate, for example a wafer, in position using a first component as described herein.

[0062] According to a fifth aspect of the present invention, there is provided a method of polishing a substrate, for example in CMP, which involves using a retaining means comprising a composite material as described herein to

retain the substrate in position and polishing the substrate. In the method, a polishing means used to polish the substrate may also contact said composite material.

[0063] Composite materials comprising a first relatively non-fibrillated fibrous material and a second relatively fibrillated material have been found to be unexpectedly advantageous. Accordingly, in a sixth aspect, the invention provides a composite material which comprises a first fibrous material, a second relatively fibrillated fibrous material and a matrix material.

[0064] Said first fibrous material may be any fibrous material, including but not limited to those described above. Said first fibrous material could be carbon fibre. Said first fibrous material is preferably non-fibrillated.

[0065] Said second fibrillated fibrous material could be any material available in fibrillated form. Said second fibrous material is preferably a pulp.

[0066] Said matrix material may be any thermofusible material. Suitably, the matrix is available in particulate form so that it can be incorporated into a composite material using the slurry methodology described in the first aspect. Said matrix material may be a metal, for example aluminium or an alloy thereof. Preferably, said matrix material is a high temperature engineering resin as described herein. In a preferred embodiment, said composite material of the seventh aspect comprises a matrix of polyetheretherketone, a second fibrillated polyaramid and a first fibrous material which is preferably a ceramic and/or metal oxide fibrous material.

[0067] The wt % of the matrix material and first and second fibrous materials of the sixth aspect may be as described in any aspect herein. Suitably, said composite material includes less than 15 wt %, more preferably less than 10 wt %, especially 6 wt % or less of said second fibrillated fibrous material. Said composite material suitably includes at least 0.5 wt %, preferably at least 1 wt %, more preferably at least 2 wt %, especially at least 3 wt % of said second fibrillated fibrous material. Said composite material suitably includes 20-80 wt % of said matrix material and 20-80 wt % of said first fibrous material.

[0068] Materials described herein may be used as ceramic replacements which have excellent wear properties. It is believed the wear properties are attributable, in part, to the sizes, especially the diameters, of first fibrous materials used in the composite material. Accordingly, in a sixth aspect, there is provided a composite material comprising a matrix material as described herein and a first fibrous material as described herein, wherein said first fibrous material comprises fibres having diameters in the range 3 to 10 μm .

[0069] Said first fibrous material suitably comprises some fibres having diameters in the range 3 to 10 μm , preferably in the range 3 to 6 μm . Suitably, said first fibrous material includes less than 10%, preferably less than 5%, especially less than 1% of fibres having diameters of less than 1 μm . Suitably, greater than 50%, preferably greater than 75%, more preferably greater than 90% of the fibres in said first fibrous material have a diameter in the range 3 to 10 μm , especially in the range 3 to 6 μm . Suitably, less than 10%, preferably less than 5%, more preferably less than 1% of fibres in said first fibrous material having a diameter greater than 10 μm .

[0070] According to a seventh aspect of the invention, there is provided a material in granular or a powderous form comprising, preferably consisting of, a composite material as described herein.

[0071] Said powder or granules suitably comprises particles having a maximum dimension of less than 10 mm, preferably less than 7.5 mm, more preferably less than 5 mm, especially 3 mm or less.

[0072] Said material preferably comprises granules. Each granule in said material preferably has a maximum dimension as aforesaid.

[0073] Said material may be a composite material comprising 50 to 70 wt % (more preferably 55 to 65 wt %, especially 58 to 62 wt %), of matrix material (e.g. a polyether such as polyetheretherketone or other preferred matrix materials as described herein) and 30 to 50 wt % (more preferably 35 to 45 wt %, especially 38 to 42 wt %) of a said first fibrous material (e.g. an inorganic discontinuous fine diameter material such as one comprising a metal oxide, e.g. alumina fibres).

[0074] According to an eighth aspect, there is provided a package comprising a material according to the seventh aspect. The package may comprise a plastics receptacle which contains the material.

[0075] Any feature of any aspect of any invention or embodiment described herein may be combined with any feature of any aspect of any other invention or embodiment described herein mutatis mutandis.

[0076] Specific embodiments of the invention will now be described, by way of example, with reference to **FIG. 1** of the accompanying figures which details a range of engineering resins.

[0077] The following materials are referred to hereinafter.

[0078] Saffil LA (Trade Mark)—a low alpha form of alumina fibre obtained from Saffil Ltd of Cheshire, England. Rf 85 material was used unless otherwise specified.

[0079] Saffil HA (Trade Mark)—a high alpha form of alumina fibre obtained from Saffil Ltd of Cheshire, England.

[0080] PEEK™ 450 G—a standard melt viscosity granular grade of polyetheretherketone obtained from Victrex Plc of Thornton Cleveleys, U.K.

[0081] PEEK™ 150 G—a low melt viscosity granular grade of polyetheretherketone obtained from Victrex Plc.

[0082] PPS—polyphenylenesulphide.

[0083] TWARON PULP—a para-Aramid pulp (i.e. poly-parahenylene terephthalamide) having a specific surface area (mean) of 12-15 m²/g; an arithmetic average fibre length of 0.5 mm, obtained from Teijin Twaron bv.

EXAMPLE 1

General Procedure for Preparation of Composites

[0084] Varying amounts of selected fibrous materials were mixed with water in a plastic bucket to form a slurry. The amount of water used may be selected according to the nature of the fibrous material, for example in terms of its type and volume fraction. By way of example, making the

material of Example 6 used about 10 litres of water for 800 g of composite. For short fibres (e.g. of 200 microns or less) the wt % of water to fibres may be 10:1; for longer fibres (e.g. of 6 mm or more), it may be 200:1 or more.

[0085] Polyetheretherketone powder was added to the fibrous material/water slurry under the action of a stirrer. The mixture was sieved using a 200 mm round sieve (200 μ m mesh size) to allow the water to separate from the solid material and thereby form a pad (retained within the sieve) comprising fibrous material and polyetheretherketone. The pad was dried in an oven at a temperature of about 150° C. The dry material is in the form of a cake that crumbles easily into a flock-like material that can be fed directly into an injection moulder or extruder.

EXAMPLE 2

General Procedure for Preparation of Test Samples

[0086] Flock-like materials prepared as described in Example 1 were injection moulded into plaques. 1 inch (2.5 cm) disks were cut from the plaques.

EXAMPLES 3 TO 20, AND COMPARATIVE EXAMPLES C1, C2 AND C3

Preparation of Specific Test Samples

[0087] Following the procedures described in Examples 1 and 2, plaques were made using the components described in Table 1 below.

TABLE 1

Example No	Thermoplastic Polymer used	Amount of thermo-plastic polymer used (wt %)	Fibrous material(s) used	Amount(s) of fibrous material used (wt %)
C1	PEEK™ 450G	100	None	0
3	PEEK™ 150G	25	Saffil LA	75
4	PEEK™ 150G	30	Saffil LA	70
5	PEEK™ 150G	40	Saffil LA	60
6	PEEK™ 150G	50	Saffil LA	50
7	PEEK™ 450G	30	Saffil LA	70
8	PEEK™ 450G	50	Saffil LA	50
C2	PPS	100	None	0
9	PEEK™ 150G	30	Saffil HA	70
10	PEEK™ 450G	45	Saffil HA	55
12	PEEK™ 450G	60	Saffil HA	40
13	PEEK™ 150G	40	Saffil HA	60
C3	PEEK™ 450G	95	Twaron Pulp	5
14	PEEK™ 150G	38	Saffil LA and Twaron Pulp	57 wt % Saffil LA 5 wt % pulp
15	PEEK™ 150G	38	Saffil LA and Twaron Pulp	57 wt % Saffil LA 5 wt % pulp
16	PEEK™ 150G	45	Saffil LA	50% Saffil 5% pulp
17	PEEK™ 450G	50	Saffil LA	45% Saffil 5% pulp
18	PEEK™ 450G	55	Saffil LA	40% Saffil 5% pulp
19	PEEK™ 150G	45	Saffil HA	50% Saffil 5% pulp
20	PEEK™ 450G	47	Saffil HA	50% Saffil 3% pulp

[0088] It should be noted that comparative example C2 relates to a PPS test sample which is the standard material used in CMP applications.

GENERAL METHOD OF TESTING MATERIALS

[0089] The 1 inch (2.5 cm) disks prepared as described in Example 2 were tested using a CETR wear tester using a standard set of conditions. Two different slurries were used, the first being SSW-2000 Tungsten Fluid with 3% hydrogen peroxide and the second being S-12 Oxide slurry. For each disk, the wear rate was assessed in both milligrams abraded per hour (mg/hr) and in microns abraded per hour (micron/hr). Results are provided in Tables 2 to 8 below.

RESULTS

[0090] (A) Slurry used: SSW-2000 Tungsten Fluid with 3% hydrogen peroxide.

TABLE 2

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
C1	17.9	27.3
3	9.8	8.6
4	7.2	6.3
5	5.1	5.4
6	4.1	4.5
7	7	6.5
8	6.5	7.1
18	—	1.9

[0091] It will be appreciated from Table 2 that the addition of hard saffil LA (Trade Mark) fibres significantly improved the wear characteristic over the unfilled material (Example C1). Also, increasing the content of Saffil LA above 50 wt % caused a deterioration in wear rates. Additionally, composites comprising PEEK™ 150G were seen to yield slightly better wear rates compared to composites comprising PEEK™ 450G, possibly due to the improved chemical resistance to the hydrogen peroxide used of the more crystallisable PEEK™ 150G material.

[0092] Slurry used: S-12 Oxide Slurry.

TABLE 3

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
C2	24.8	36.6
C1	18	27.5
3	21.3	18.7
4	18.3	16
5	17.3	18.3
6	16.1	17.5
7	16.3	15
8	11.6	12.8
12	8.1	9.4
13	5.5	5
16	7.6	8.2
17	7.2	8.1
18	5.8	6.8
19	7.5	7.9
20	6.4	6.8

[0093] It will be appreciated that relatively low amounts of fibrous material tend to yield the best results. Also, using the

S-12 oxide slurry the tougher PEEK™ 450G based materials tended to be better compared to the more crystalline PEEK™ 150G based materials.

[0094] (C) Slurry used: SSW2000 Tungsten fluid with 3% hydrogen peroxide.

TABLE 4

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
9	5.2	5.2
10	2.6	2.6

[0095] It will be noted that the use of Saffil HA reduced the wear rates significantly.

[0096] (D) Slurry used: SS-12 Oxide Slurry

TABLE 5

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
9	12.5	11.3
10	10.6	10.7

[0097] Again, the Saffil HA yielded improved wear rates compared to Saffil LA.

[0098] (E) Slurry used: SS-12 Oxide Slurry

TABLE 6

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
12	8.1	9.4
13	5.5	5

[0099] (F) Slurry used: SS-12 Oxide Slurry

TABLE 7

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
C3	22	33.2
5	17.3	18.3
14	9.2	9.2
15	8.6	8.7

[0100] It should be noted that addition of a small amount (5 wt %) of Twaron pulp in a polyetheretherketone and Saffil composite resulted in a surprising improvement in the wear rate (compare Example 20 and Example 14 in Table 7). Also, the use of a longer Saffil fibre (Example 15) resulted in an improved wear resistance (compare Example 14).

[0101] (G) Slurry used: SSW-2000 Tungsten Fluid with 3% hydrogen peroxide.

TABLE 8

Material of Example No.	Wear rate mg/hr	Wear rate micron/hr
C3	17	25.6
5	5.1	5.4
13	2.8	2.8

[0102] Again, as for (F) above, the exceptional wear of the Twaron/Saffil composite material of Example 13 should be noted. There appears to be a synergistic effect exhibited in composites comprising fibrillated polyaramid fibres (ie polyaramid pulp) and the stiffer Saffil fibre. It may be that there is a three-dimensional effect of the polyaramid fibre holding and protecting the brittle, rod-like hard Saffil fibres from impact.

EXAMPLES 21 TO 29

[0103] Saffil RF alumina fibres and polyetheretherketone powder were slurried together in water using a water to fibre weight ratio of about 20:1. The mixture was sieved as described in Example 1 to give a dense pad which was dried. The density of the pad is dependent inter alia on fibre lengths—for the finest fibre grade the density is about 0.3 g/cc. The pad could be broken up by hand to give small flock-like granules which could be fed directly into an injection moulder or pre-granulated in an extruder.

[0104] The relative amounts of fibres and polyetheretherketone were varied in respective examples and tensile strength, elongation and tensile modulus assessed according to ASTM D638-99. Results are provided in Table 9 below.

TABLE 9

Material of Example No.	Summary of constituents	Detail on process	Tensile strength (MPa)	Elongation (%)	Tensile Modulus (GPa)
21	PEEK TM 150PF (90 wt %) & fibres (10 wt %)	Pad pre-granulated in extruder	98	2.5	6.4
22	PEEK TM 150PF (90 wt %) & fibres (10 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	88	2.1	5.7
23	PEEK TM 150PF (80 wt %) & fibres (20 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	105	2.8	8.3
24	PEEK TM 150PF (70 wt %) & fibres (30 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	120	1.5	13.6
25	PEEK TM 150PF (50 wt %) & fibres (50 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	137	1.0	24
26	PEEK TM 450PF (50 wt %) & fibres (50 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	129	1.2	27
27	PEEK TM 150PF (30 wt %) & fibres (70 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	170	0.7	44
28	PEEK TM 150PF (20 wt %) & fibres (80 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	—	—	—
29	PEEK TM 450PF (30 wt %) & fibres (70 wt %)	Flock-like granules of pad (produced by hand) fed directly into injection moulding machine	—	—	—

[0105] Referring to Table 9, the Example 15 and 16 materials were too stiff to process in the sample mould. However, the samples could be processed by other means. For example, the material of example 16 was made into a 150×150×10 mm plaque.

[0106] It will be appreciated from Table 9 that progressively higher strength and stiffness materials were obtained with increasing fibre fractions showing that the process described is an advantageous way of making materials from otherwise difficult to handle fibrous materials.

EXAMPLE 30

[0107] Saffil LA (42 wt %) was slurried with PEEKTM 150PF (58 wt %) and pads of material produced as described above. The pads were cut up into 15 mm cubes and these low density cubes were fed without further consolidation into an injection moulder and test pieces formed and tested as described in Example 21. Results are provided in Table 10. The dry strength of the pad improved with the longer fibres as did the tensile strength of the final material when compared to Example 25 which was similar but had a higher fibre fraction.

TABLE 10

Material of Example No.	Summary of Constituents	Details on process	Tensile strength (MPa)	Elongation (%)	Tensile Modulus (Gpa)
30	PEEK TM 150PF (58 wt %) & fibres	Flock-like granules of pad fed	137	0.7	22

TABLE 10-continued

Material of Example No.	Summary of Constituents	Details on process	Tensile strength (MPa)	Elonga- tion (%)	Tensile Modulus (Gpa)
	(42 wt %)	directly into extruder.			

1. A composite material comprising a matrix material and a first fibrous material which includes alumina.

2. A composite material as claimed in claim 1, wherein said first fibrous material comprises alumina fibres.

3. A composite material as claimed in claim 1, wherein said first fibrous material comprises alumina fibres having diameters in the range 3 to 10 μm ; and includes less than 10% of fibres having a diameter of less than 1 μm .

4. A composite material as claimed in claim 1, wherein greater than 90% of the fibres in said first fibrous material have a diameter in the range 3 to 10 μm .

5. A composite material as claimed in claim 1, wherein said matrix material is selected from polyetherketone, polyetheretherketone and polyetherketoneketone.

6. A composite material as claimed in claim 1, wherein said matrix material comprises polyetheretherketone.

7. A composite material as claimed in claim 1, which includes at least 40 wt % and 80 wt % or less of said matrix material.

8. A composite material as claimed in claim 1, which includes at least 35 wt % and 60 wt % or less of alumina fibres.

9. A composite material as claimed in claim 1, which comprises a matrix material comprising polyetheretherke-

tone and a first fibrous material which includes alumina fibres, wherein said composite material includes at least 40 wt % of polyetheretherketone and at least 30 wt % of alumina fibres.

10. A composite material as claimed in claim 1, which includes a first fibrous material which comprises alumina fibres and a second fibrous material which is fibrillated and comprises a para aramid pulp.

11. A composite material as claimed in claim 10, wherein said composite material includes less than 10 wt % of said second fibrous material.

12. A composite material as claimed in claim 1, wherein the sum of the wt % of all fibrous materials in said composite material is at least 35 wt %.

13. A method of manufacturing a first component, the method comprising forming the first component from a composite material as claimed in claim 1.

14. A method as claimed in claim 13, which includes the step of machining a precursor of said component which precursor comprises a said matrix material and a said first fibrous material, to manufacture said first component.

15. A method as claimed in claim 14, wherein said first component includes an opening which has a diameter of less than 0.1 m.

16. A method as claimed in claim 14, wherein said first component includes a composite material comprising 50 to 70 wt % of polyetheretherketone and 30 to 50 wt % of alumina fibres.

17. A first component which is a machined component having an opening which has a diameter of less than 0.1 m and which component is made of a composite material comprising 50 to 70 wt % of polyetheretherketone and 30 to 50 wt % of alumina fibres.

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