SILICON CARBIDE FIBERS ESSENTIALLY DEVOID OF WHISKERS AND METHOD FOR PREPARATION THEREOF

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ABSTRACT

Silicon carbide fibers are produced by mixing discontinuous isotropic carbon fibers with a silica source and exposing the mixture to a temperature of from about 1450° C, to about 1800° C. The silicon carbide fibers are essentially devoid of whiskers have excellent resistance to heating and excellent response to microwave energy, and can readily be formed into a ceramic medium employing conventional ceramic technology. The fibers also may be used for plastic and metal reinforcement.
SILICON CARBIDE FIBERS ESSENTIALLY DEVOID OF WHISKERS AND METHOD FOR PREPARATION THEREOF

BACKGROUND OF THE INVENTION

[0001] 1. Field of Invention

[0002] The present invention is directed to discontinuous silicon carbide fibers and a process for producing them. In particular, the invention is directed to discontinuous silicon carbide fibers that retain the morphology of the carbon source, respond to microwave energy, and are essentially devoid of whiskers.

[0003] 2. Description of the Related Art

[0004] Silicon carbide is used as reinforcement for both ceramics and plastics subjected to high temperatures. Silicon carbide materials have many desirable qualities including high resistance to oxidation, excellent mechanical strength, and the ability to withstand multiple exposures to high temperatures without deformation. The importance of such qualities has led to the development of many methods by which various shapes of silicon carbide materials are made. The different shapes are useful in a plethora of industrially important products.

[0005] Silicon carbide is commonly available in particular, whisker, fiber, and cloth forms. Each form has distinct properties and characteristics exploitable in diverse industrial applications.

[0006] Various methods have been developed to produce silicon carbide having these forms. For example, Evans, GB 998,089, describes a method for making silicon carbide cloth. First, carbon cloth is heated in an inert atmosphere, then embedded in fine powdered silicon (99.9 percent purity). The silicon-embedded cloth is then heated in an inert atmosphere to 1410°C, i.e., just below the melting point of silicon, to produce a cloth of silicon carbide.

[0007] Methods for making silicon carbide whiskers, i.e., elongated single crystals of silicon carbide, are well-known. Liquid- and gas-phase reaction systems are often used to form these elongated single crystals. Typical methods of making silicon carbide whiskers include: (1) solidification from liquid silicon carbide at high temperature and high pressure, (2) dissolving carbon into molten silicon and crystallizing the silicon carbide, (3) sublimation of silicon carbide powder and subsequent re-deposition, and (4) deposition of silicon carbide crystals from the vapor of silicon compounds.

[0008] For example, Wainer, U.S. Pat. No. 3,269,802 is directed to preparation of metal carbide products by exposing a carbonized product to an atmosphere comprising volatile metal-containing material, such as a metal halide or a metal carbonyl. The product takes the general form of the carbonized material, but also appears in other forms, including whiskers, fibers, and coatings. Thus, the method does not form a single product and produces environmentally undesirable waste gas.

[0009] Another method for producing metal carbide shapes is set forth in Hamling, U.S. Pat. No. 3,403,008. Organic material in the desired shape is impregnated with a metal compound solution. The impregnated form then is heated in two steps: first, to carbonize the organic material, then to form the metal carbide.

[0010] Cutler, U.S. Pat. No. 3,754,076, is directed to a method for producing silicon carbide whiskers from rice hulls, which comprise about 15-20 percent silica and carbon. A metal-containing composition, typically metal oxide, is used to catalyze the reaction. Iron and iron oxide are suitable catalysts.

[0011] Yamada, U.S. Pat. No. 4,849,196, is directed to a process for producing silicon carbide whiskers. In Yamada's method, Fe, Co, or Ni are added in any combination to minimize the production of silicon carbide powder while maximizing the yield of silicon carbide whiskers.

[0012] Weaver, U.S. Pat. No. 4,873,069, discloses a process for producing silicon carbide whiskers. In accordance with Weaver's process, discontinuous fluffy carbonized fibers (having a void volume of at least 40 percent) and ultra fine silica are heated to 1600-1900°C for about 2 hours to produce silicon carbide whiskers. Boron oxide, alone or mixed with aluminum, serves as a catalyst. A preferred carbon source is carbonized cotton fiber having a diameter of 4-15 μm and an average length of about 2 mm. The whiskers have a smooth surface, a diameter of 0.5 to 10 μm and a length of up to 1 mm. Nixdorf, U.S. Pat. No. 5,087,272, uses the process described in Weaver to generate silicon carbide whiskers having a diameter of 1-3 microns which are then incorporated into ceramic filters for removing volatile organic compounds from gas streams.

[0013] Other methods for producing silicon carbide whiskers include use of iron to catalyze the formation of whiskers from rice hulls (Home, U.S. Pat. No. 4,283,375). Similarly, Home, U.S. Pat. No. 4,284,612, is directed to use of iron to catalyze production of silicon carbide whiskers from the combination of ground carbonized organic fibers, silica, and rice hulls.

[0014] Silicon carbide whiskers are not satisfactory for all purposes. For example, the production of respirable pollutants from silicon carbide whisker handling, from devices containing whiskers, and in particular from filtering devices that are repeatedly exposed to high temperatures, are sources of concern. As can be seen from the methods described herein, whiskers are relatively expensive and technically difficult to make. Proper handling of whiskers is especially important so as to minimize the number of inhalable fine particles. In addition, facilities which can sustain the high temperatures required for the production of silicon carbide whiskers are expensive to build and difficult to maintain. Thus, commercial production of silicon carbide whiskers is not entirely satisfactory.

[0015] Silicon carbide fiber and filament forms avoid some of the failings of silicon carbide whiskers. Woven and combed forms of silicon carbide materials may also avoid some of the problems presented by whiskers. Fiber, filament, and woven forms comprise particles larger than whiskers, and are therefore, less likely to yield airborne respirable particles.

[0016] Wei, U.S. Pat. No. 4,481,179, is directed to a method of producing silicon carbide bonded fiber composites, starting from a carbon-bonded carbon fiber composite. Galasso, U.S. Pat. No. 3,640,693, is directed to forming a silicon-containing fiber by casting silicon metal in a glass
tube, drawing composite filaments, removing the glass sheath, then exposing the silicon metal to carbon or nitrogen to produce silicon carbide or silicon nitride, respectively. Debolt, U.S. Pat. No. 4,127,659, is directed to coating a refractory substance, such as carbon, with silicon carbide by chemical vapor deposition to produce a silicon carbide filament containing a core and a coating of carbon-rich silicon carbide. Srinivasan, U.S. Pat. No. 5,729,033, is directed to a method of producing silicon carbide material (fiber, fabric, or yarn) by carbothermal reduction of silicon material. Particular proportions of silica and carbon are preferred. Delcruw, U.S. Pat. No. 5,071,600 and U.S. Pat. No. 5,268,336, are directed to methods for producing silicon carbide fibers by the reaction of polycarbosilane and methylpolysilazane resins in the presence of boron. Tokumoto, U.S. Pat. No. 5,344,709, describes a silicon carbide fiber produced from polycarbosilane fiber and having an amorphous layer of carbon thereon.

[0017] Each of these methods has disadvantages. The continuous silicon carbide filament produced by the chemical vapor deposition method are not homogeneous and, when chopped to obtain fibers, a core carbon is exposed. The resultant fiber product has reduced resistance to oxidation. All of the polymer conversion methods are disadvantageous in that they require synthesis of the starting material which must then be spun, cured, and pyrolyzed to burn off the organic material. The submicron silicon carbide powder process incorporated by reference in Srinivasan is expensive and difficult to implement because the polymer carrier requires further processing to effectuate its removal.

[0018] Other methods of producing silicon carbide fibers, essentially without whiskers have been recently developed. Okada et al. (Okada, et al. U.S. Pat. No. 5,618,510 and U.S. Pat. No. 5,676,918 and Nakajima et al. U.S. Pat. No. 5,922,300) developed methods which involve activating carbon fibers by, for example, contact with water vapor, to yield porous activated carbon fibers. The activated carbon fibers then are exposed to silicon monoxide gas generated by heating a mixture of silica and silicon to a temperature between about 1200-2000° C. under reduced pressure to minimize formation of whiskers. In accordance with the methods of the first two patents, range of surface area of the activated fibers is said to be from 100 to 3,000 m²/g, with the sole exemplification at 1,500 m²/g. The Nakajima patent teaches that the surface area of the activated carbon fibers must be at least 300 m²/g, lest unreacted carbon remain in the fibers because the reaction becomes difficult to carry out uniformly at a sufficiently high reaction rate. Yajima, U.S. Pat. No. 4,100,233, describes a method of producing silicon carbide fibers which involves dissolving or melting an organosilicon compound in a solvent and spinning the solution into filaments. The spun filaments are then heated to volatilize low molecular weight compounds, and, finally, baked to form silicon carbide fibers.

[0019] Thus, there remains a need for an easily implemented, economical, and environmentally benign method of producing homogeneous, discontinuous, silicon carbide fibers.

SUMMARY OF THE INVENTION

[0020] The invention is directed to a method for producing discontinuous silicon carbide fibers from the reaction of discontinuous carbon fiber and fine silica in the presence of promoters in a graphite resistance furnace under an inert atmosphere, and to the fibers thus made. Skilled practitioners recognize that such fibers are not single crystals. The silicon carbide fibers of the present invention are essentially devoid of whiskers, retain the morphology of the carbonized fiber if promoters are used, may have a silica coating, and are produced at a high yield. The silicon carbide fibers of the invention, and especially coated fibers of the invention, can be readily incorporated into other media, such as ceramics, plastics, and metals, via conventional processing technology. The silicon carbide fibers of the present invention are economically produced, are exceptionally responsive to microwave energy, and have excellent resistance to, e.g., oxidation during repeated exposures to microwave radiation. Thus, a ceramic medium having the fibers of the invention incorporated therein is especially suited for use as a regenerable filter medium in a device for removing volatile organic compounds from fluids.

[0021] The discontinuous silicon carbide fibers of the present invention are less expensive to produce, easier and less costly to process into substrate materials, such as ceramic filter media, and are produced by a method that is environmentally benign. Further, the fibers of the invention will not produce airborne, respirable particles, and so do not require expensive handling techniques and do not present health hazards associated with respirable particles.

[0022] These discontinuous silicon carbide fibers are particularly useful for, but not limited to, incorporation into a filter-heater apparatus for the removal of volatile organic compounds from a gas stream. For such a use, the fibers are formed via ceramic processing techniques into ceramic sheets or shapes which are then formed into filters. Microwave energy is then applied to the filter periodically, interacting with the silicon carbide fiber providing heat which then burns off any volatile organic compounds such as diesel soot. Originally, the use of silicon carbide whiskers was investigated for this purpose, but silicon carbide fibers as made by this invention were preferred over whiskers.

[0023] In accordance with the present invention, discontinuous silicon carbide fibers essentially devoid of whiskers are prepared by admixing discontinuous isotropic carbon fiber, silica, and preferably at least two promoters to form a fiber/silica mixture; drying the fiber/silica mixture; and reacting dried fiber/silica mixture in a resistance furnace for a time and at a temperature sufficient to form the discontinuous silicon carbide fibers of the invention essentially devoid of whiskers.

BRIEF DESCRIPTION OF THE DRAWINGS

[0024] FIGS. 1a-1d are SEM photographs of purchased Carbollex® isotropic carbon fiber grade P-200 at 200x (FIG. 1a) and 500x (FIG. 1c) magnification, and of silicon carbide fiber product of the invention made therefrom at 200x (FIG. 1b) and 500x (FIG. 1d) magnification.

[0025] FIG. 2 is an SEM photograph of silicon carbide product prepared from Cytec ThermalGraph® DKD X mesophase pitch at 200x magnification.

[0026] FIG. 3 is an SEM photograph of silicon carbide product prepared from Fortafil® PAN M275 carbon fiber at 200x magnification.
FIG. 4 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from P-200 isotropic carbon fiber pretreated 1 hr at 1500°C.

FIG. 5 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from P-200 isotropic carbon fiber pretreated 1 hr at 1800°C.

FIG. 6 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from P-200 isotropic carbon fiber pretreated 7 hr at 1800°C.

FIG. 7 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from a stoichiometric blend of P-200 isotropic carbon fiber and fumed silica.

FIG. 8 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from Carbolux® P-600 isotropic carbon fiber.

FIG. 9 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from P-200 isotropic carbon fiber without calcium oxalate.

FIG. 10 is an SEM photograph at 200x magnification of silicon carbide fiber of the invention made from P-200 isotropic carbon fiber without ferrous sulfate.

FIGS. 11a and 11b are SEM photographs of silicon carbide fiber of the invention made from P-200 isotropic carbon fiber without any promoters at 500x (FIG. 11a) and 5000x (FIG. 11b) magnification.

FIG. 12 is a plot of x-ray diffraction data for silicon carbide fiber of the invention.

FIG. 13 is an SEM photograph at 200x magnification of silicon carbide fiber made under a nitrogen atmosphere.

Two long lines of text follow, discussing the invention, its properties, and the manufacturing process.
duced during spinning. In contradistinction, the structure of isotropic pitch carbon fibers is derived almost entirely from heating the fiber after stabilization to produce isotropic pitch carbon fibers. Graphitic structure thus is not found in such fiber as it is supplied, although such structure can be introduced by heating the fiber to a high temperature (at least about 1800° C.) for at least about 7 hours. However, with isotropic fibers, such graphitic structure in an isotropic fiber does not cause production of whiskers.

[0045] Mesophase pitch fibers may be readily distinguished from isotropic pitch fibers by examination under a light microscope under crossed Nichols, which skilled practitioners recognize will reveal the high degree of structure in the mesophase fiber. Mesophase pitch fibers yield superior physical properties. Similarly, PAN fibers also exhibit high levels of fiber structure and superior mechanical properties. Such fibers are not suitable for use in the invention.

[0046] Isotopic carbon fiber suitable for use in the invention is essentially isotropic, i.e., its mechanical properties are essentially the same in each direction. Such fiber is not “high-performance” fiber. For example, the tensile strength of an isotropic carbon fiber is approximately 10 percent of that of a “high performance” fiber. Typically, such fibers are straight and smooth, essentially without kinks, knots, or other surface defects, as can be seen in FIGS. 1a and 1c. When isotropic carbon fibers are heated with silica in accordance with the invention, the resulting silicon carbide fiber product is essentially devoid of whiskers. In contrast, when mesophase pitch or PAN is used as the carbon fiber, significant whisker contents are visible in an SEM photograph at 200x magnification.

[0047] The length of isotropic carbon fiber suitable used in the invention is limited only by economics and commercial practicability. Isotopic carbonized fiber of essentially any length can be used in the method of the invention to yield silicon carbide fiber in accordance with the invention. However, typically, the length of commercially available fiber does not exceed about 20 mm. Fibers that are relatively long, i.e., longer than about 1 mm, will yield silicon carbide product having low bulk density, increasing the cost of furnace treatment, packaging, transportation, and storage.

[0048] The diameter of isotropic carbon fiber typically is less than about 25 microns. Diameters greater than this are not preferred because it is difficult to ensure completeness of reaction at the core of such a relatively large diameter fiber. Typically, such carbon fiber has a surface area of less than about 100 m²/g, more typically between about 10 and about 50 m²/g, and most typically between about 20 and about 35 m²/g.

[0049] A preferred source of discontinuous carbon fibers is Anshan East Asia Carbon Fiber Co., Ltd., particularly grades P-200 and P-600. Carboflex® P-200 is especially preferred. The fibers typically have an average length of 200 microns; diameters range from about 5 to 25 microns with an average of 15 microns. Longer or shorter lengths can be used depending on end use. If the fibers are longer than 1000 microns (1 mm), the blend bulk densities are significantly lowered and processing costs thereby increased, as set forth above. These Anshan fibers are believed to be milled, then sieved to remove fines.

[0050] The Carboflex® P-200 fiber as obtained typically has a surface area of between about 20 and 30 m²/g. Heat treatment of P-200, for example at a temperature of at least about 1500° C. for at least about 1 hour, reduces the average length and diameter. The surface area of the heat treated fibers was markedly reduced, to about the same level as the mesophase and PAN fibers investigated, or about 0.5 m²/g.

[0051] Particulate silica from any source may be used in the present invention, including, but not limited to, granular silica and colloidal suspensions of silica. Regardless of the silica source, it is preferred that the particles be no larger than about 0.1 μm (1000 A). A preferred silica source is Cab-O-Sil® grade M5, available from Cabot Corp., Tuscola, Ill. This product is fused silica having a surface area of about 220 m²/g and an approximate bulk density of 0.07 g/cc.

[0052] Promoters preferably are used in the method of the invention to enhance the integrity of the silicon carbide fiber formed. Use of promoters yields integral silicon carbide fibers having essentially the same morphology as the isotropic carbon fiber starting material. Promoters most preferably are used in combination. One type of promoter is a metal containing promoter selected from the group consisting of salts, compounds, and complexes of iron, cobalt, or nickel, and blends thereof. These salts, compounds, and complexes may be converted to oxides of iron, cobalt, or nickel at a temperature less than about 1650° C. A second type of promoter is selected from the group consisting of the salts, compounds, and complexes of alkali metals or alkaline earth metals, and blends thereof. These salts, compounds, and complexes may be converted to oxides of these materials at the reaction temperature employed. When a single type of promoter is used, it may be any of the promoters. If two types of promoters are used, one promoter is selected from each type. A blend of promoters of one type will be referred to as “one promoter” herein for convenience.

[0053] Preferred metal-containing promoters include iron oxide, ferrous sulfate, potassium ferrocyanide, cobalt oxide, cobalt sulfate, nickel oxide, and nickel sulfate. Ferrous sulfate (FeSO₄) is an especially preferred promoter. The especially preferred metal-containing promoter is present in an amount between about 0.5 and about 5.0 wt percent of the fiber/silica blend; preferably between about 0.7 and about 3.0 wt percent; more preferably between about 1.0 and about 2.0 wt percent; and most preferably between about 1.3 and about 1.7 wt percent. Metal-containing promoters other than ferrous sulfate are present in an amount sufficient to provide the mole quantity of metal equivalent to the mole quantity of iron.

[0054] Preferred alkali metal- and alkaline earth metal-containing promoters include calcium oxide, barium oxide, strontium oxide, and potassium oxide. Calcium oxide is especially preferred. This promoter typically is present in an amount between about 0.2 and about 3.0 wt percent of the fiber/silica blend; preferably between about 0.25 and about 2.0 wt percent; more preferably between about 0.4 and about 1.0 wt percent; and most preferably between about 0.5 and about 0.7 wt percent. Alkali metal- and alkaline earth metal-containing promoters other than calcium oxide are present in an amount sufficient to provide the mole quantity of alkali metal or alkaline earth metal equivalent to the mole quantity of calcium.

[0055] The metal-containing promoter and the alkali metal- or alkaline earth metal-containing promoter may be
provided in a single composition. Thus, a single composition that contains both metal promoter and alkali metal or alkaline earth metal promoter may be used to provide at least two promoters in accordance with the method of the invention.

[0056] With the guidance provided herein, a skilled practitioner can select suitable salts, compounds, and complexes to serve as promoters. For example, a skilled practitioner recognizes that, at the reaction temperatures used in the method of the invention, most promoters will be converted to an oxide form. However, one must exercise care in selecting promoter compositions. For example, ferrous nitrate (Fe(NO₃)₂·H₂O) is not a suitable promoter composition because it degrades if the feed mixture is heated while still wet, whereas ferrous sulfate (FeSO₄) is a preferred promoter composition.

[0057] The promoters are used in a quantity sufficient to assist the conversion of carbon fibers to silicon carbide and to promote fiber quality. Suggested quantities of promoter compositions are specified herein; with this guidance, skilled practitioners will be able to determine appropriate quantities of other suitable promoters.

[0058] The inventors have observed that the combination of a metal-containing promoter and an alkali metal- or an alkaline earth metal-containing promoter is particularly effective in providing high quality fibers essentially devoid of whiskers and having essentially the same morphology as the carbon fiber from which it is made. Skilled practitioners recognize that the essentially complete absence of whiskers is a completely unexpected result, as either calcium or iron is used individually in whisker manufacture to promote whisker production.

[0059] Because silicon carbide fiber of the invention may maintain the morphology of the isotropic carbon fiber from which it is made, or may be degraded, the largest expected size range is that of the starting carbon fiber, i.e., diameter between about 5 and about 25 microns and typical lengths up to about 1 mm. However, it is expected that smaller diameters, between about 3 and about 15 microns, and typical lengths up to about 500 microns, will be realized. Thus, the expected diameter of silicon carbide fiber product is between about 3 and 25 microns, with lengths up to about 1 mm.

[0060] SEM photographs show that silicon carbide fiber products of the invention are essentially devoid of whiskers even when promoters are used individually or are omitted completely. FIGS. 1b, 1d, and 4 through 11 show that no whiskers can be seen in product of the invention at 200x magnification.

[0061] In particular, SEM photographs of the product of the invention made with promoters (FIGS. 1b, 1d, and 4-8) illustrate that the morphology of the silicon carbide fibers is essentially the same as that of the isotropic carbon fiber starting material. This result is completely unexpected, as prior methods used iron and calcium salts as promoters of whisker growth.

[0062] FIG. 9 shows that no whiskers can be seen at 200x magnification in an SEM of silicon carbide fiber produced without calcium promoter, and FIG. 10 illustrates the same phenomenon for silicon carbide fiber produced without iron promoter. FIGS. 11a and 11b show that no whiskers can be seen at 500x and 5000x magnifications, respectively, in silicon carbide fiber of the invention made without promoters.

[0063] FIGS. 9-11 illustrate that, whereas promoters are not needed to inhibit whisker production, use thereof produces silicon carbide fibers that are not degraded. FIGS. 9-11 illustrate that without both iron and calcium salts present, the silicon fiber product fibers of the invention are degraded and form smaller particles of fiber, but do not form whiskers. Promoters do not affect conversion of the carbon and silica starting materials to silicon carbide.

[0064] FIGS. 1a and 1c are SEM photographs of Carboflex® P-200 isotropic carbon fiber at 200x and 500x magnification, respectively. The isotropic carbon fibers appear to have very smooth surfaces. Table 1 below summarizes physical properties and characteristics of various discontinuous carbon fibers, including the samples depicted in FIGS. 1a and 1c. The specific surface area of this fiber as obtained was determined to be 28.7 m²/g, which would indicate the fiber has significant void volume. However, it was found to be very difficult to obtain a good reproducible specific surface area. Preheating the fiber made it easier to obtain reproducible specific surface area measurements and reduced the sulfur content of the fiber. The specific surface areas of heated fibers obtained were much closer to that expected of a void-free, smooth-surfaced fiber, as set forth in Table 1.

<table>
<thead>
<tr>
<th>Discontinuous Carbon Fiber</th>
<th>Density, g/cc</th>
<th>Surface Area, m²/g</th>
<th>Sulfur Content, wt %</th>
<th>Length, microns</th>
<th>Diameter, microns</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isotopic Pitch, Carboflex® P-200</td>
<td>1.98</td>
<td>28.7</td>
<td>1.43</td>
<td>201</td>
<td>15.1</td>
</tr>
<tr>
<td>Isotopic Pitch, Carboflex® P-900</td>
<td>1.98</td>
<td>0.59</td>
<td>331</td>
<td>14.2</td>
<td></td>
</tr>
<tr>
<td>Carboflex® P-200 pretreated for 1 hour at 1500° C.</td>
<td>1.53</td>
<td>0.5</td>
<td>0.85</td>
<td>314</td>
<td>11.9</td>
</tr>
<tr>
<td>Carboflex® P-200 pretreated for 1 hour at 1800° C.</td>
<td>1.53</td>
<td>0.4</td>
<td>0.28</td>
<td>314</td>
<td>12.1</td>
</tr>
<tr>
<td>Carboflex® P-200 pretreated for 7 hours at 1800° C.</td>
<td>1.54</td>
<td>0.3</td>
<td>0.36</td>
<td>333</td>
<td>11.6</td>
</tr>
<tr>
<td>Mesophase Pitch Fiber, Cytec ThermalGraph® DKD X PAN, Fortafil® M375</td>
<td>2.19</td>
<td>0.5</td>
<td>20.1</td>
<td>118</td>
<td>6.9</td>
</tr>
</tbody>
</table>

[0065] The Carboflex® carbon fibers described in Table 1 are commercially available from Anshan East Asia Carbon Fiber Co., Ltd., Anshan, Liaoning, China. Both P-200 and P-600 are isotropic pitch fibers. The Anshan pretreated fibers also were heated to the indicated temperature and held for the stated time. The pretreated fibers were somewhat degraded in length and diameter but otherwise appeared unchanged. XRD (x-ray diffraction) showed the incipient formation of graphic structure in the fiber heated for 7 hours.

[0066] The mesophase pitch fiber was obtained from Cytec Carbon Fibers, 7139 Augusta Road, Piedmont, S.C. 29673. The PAN fiber was obtained from Fortafil Fibers, Inc., P. O. Box 357, Roane County Industrial Park, Rochester, Mich. 48306.
[0067] FIGS. 1b and 1d show that silicon carbide fibers produced using promoters in accordance with this invention have higher specific surface areas than the heat treated precursor carbon fibers. The surfaces of the silicon carbide product look smooth even at 500x magnification.

[0068] Silicon carbide fiber of the invention is a gray-green fiber material that is essentially all β-silicon carbide and has essentially the same morphology as the carbonized fiber starting material if promoters are used. X-ray diffraction analyses confirm that silicon carbide fiber of the invention is essentially β-silicon carbide. FIG. 12 is a plot of x-ray diffraction data for a silicon carbide fiber of the invention exemplified in Example 1.

[0069] FIG. 12 illustrates that the silicon carbide fiber of the invention is essentially β-silicon carbide. As can be seen on that figure, there is a large peak labeled “PDF 73-1708.” Skilled practitioners recognize that “PDF” stands for Powder Diffraction File, as compiled by the National Institute of Standards and Technology Crystal Data Center and the International Centre for Diffraction Data. In that identification scheme, PDF 73-1708 is the identification of β-silicon carbide. There also is a small peak at the 2θ H value of 22 degrees, which represents cristobalite, a high temperature form of silica. Another very small peak occurs at 2θ of 45 degrees, representing hexagonal silicon carbide. Thus, it can be seen that product of the invention is essentially all β-silicon carbide.

[0070] Silicon carbide fibers of the invention also may comprise a coating of silica. Silica-coated fibers of the invention are more easily processed into ceramic filter media than uncoated fibers and are better able to resist oxidation during repeated microwave energy exposures.

[0071] The silicon carbide fiber product quality is improved by employing a non-stoichiometric ratio of carbon fiber and silica. Whereas the stoichiometric ratio is 3 moles of carbon per mole of silica, silicon carbide fiber of the invention is made using a mole ratio of carbon to silica of between about 2.4:1 to about 3.5:1, preferably between about 2.5:1 and about 3.0:1, and most preferably between about 2.6:1 and about 2.8:1. If an excess of silica is employed in the initial blend, the silicon carbide fiber may be coated with silica. If an excess of carbon is employed in the initial blend, unreacted carbon may be found in the core region of the silicon carbide fiber. Any silica coating due to excess silica in the feed may be removed by washing the fiber with hydrofluoric acid (HF) if so desired.

[0072] Skilled practitioners recognize that the water-gas reaction,

\[2C + H_2O \rightarrow CH_4 + CO\]

[0073] will cause loss of some carbon during the drying period if the temperature exceeds about 250°C. Thus, this reaction must be considered when determining the relative quantities of carbon and silica in the reactant mix. For example, skilled practitioners recognize that the amount of water in the reactant mixture, and in the atmosphere in the reaction boat, will affect how much carbon may be lost to this reaction, and thus can take steps to minimize the quantity of free water present.

[0074] In accordance with a preferred method of the invention, the carbon fiber first is “opened,” or decom- pressed. Such decompacting helps ensure that the various components can be thoroughly mixed before heating. Typically, the “opening” can be effectuated by a laboratory single blade mixer, especially a mixer in which the feed component admixture is to be formed. A short period (less than 5 minutes) is sufficient for thorough mixing.

[0075] The metal-containing promoter, preferably FeSO₄, then is added. While the FeSO₄ fiber mixture is being blended, the preferred calcium-containing promoter, calcium oxide, is added immediately after the other promoter is added. Because the quantity of calcium oxide to be added is small relative to the volume of the fibers, it is preferred to add this promoter in a volatile carrier (e.g., ethanol or water). Preferably, a suspension of calcium oxide is prepared, then added to the reactants during agitation. Skilled practitioners are familiar with techniques for adding such quantities of promoters. Typically, 3 minutes of blending is sufficient at this step. It is especially preferred that both promoters be added simultaneously by forming a suspension of calcium oxide in an aqueous solution of ferrous sulfate. This embodiment not only shortens the mixture preparation time, but also minimizes the quantity of water present in the reactant mixture. The silica then is added to the admixture from the feed. Blending for about another two minutes typically is sufficient to form a homogenous, free-flowing blend. The quantity of silica and carbon fiber preferably is selected to provide a molar reactant ratio of carbon to silica of between about 2.6:1 and about 2.8:1.

[0076] For drying and subsequent reaction, the reactant blend is loaded into a graphite “boat” which then is capped. The “boat” is passed into a resistance furnace through a muffle furnace. While the boat is in the muffle furnace, the temperature is increased in steps, e.g., to 250°C, then to 500°C, and then to 750°C. During this heating, the water in the reactants and in the atmosphere reacts with carbon in accordance with the water-gas reaction described above, and some carbon may be lost.

[0077] In accordance with the method of the invention, the boat containing the dried reactant mixture then is moved into a graphite resistance furnace and heated in an essentially inert atmosphere at a temperature between about 1450°C-1800°C for a time sufficient to form the silicon carbide fibers of the invention. If the temperature of the furnace is low, the reaction rate is slow, especially below about 1450°C. At temperatures above 1800°C, the quality of the fibers deteriorates; fiber length is degraded and detrinit is formed.

[0078] The preferred temperature for the reaction is between about 1500°C-1775°C, more preferably between about 1650°C-1750°C. At 1675°C, more than 95 percent of the carbon is converted to silicon carbide fibers.

[0079] As used herein, an “essentially inert” atmosphere is an atmosphere which is essentially inert to all reactants and the environment (e.g., the furnace itself and other objects in it), and which does not produce whiskers. Argon is a preferred gas for use as an essentially inert atmosphere in the invention. It is likely that the other inert gases, also known as the “Noble gases,” i.e., Group 18 (formerly Group VIIIa) of the periodic table of the elements, and helium also are suitably used in the invention.

[0080] Nitrogen is not suitable for use as an “essentially inert” atmosphere gas. The inventors have found that use of
nour nitrogen contributes to whisker formation. With the guidance provided herein, a skilled practitioner will be able to identify suitable “essentially inert” atmospheres for use in the invention.

[0081] The reaction is carried out in any suitable furnace. Graphite resistance furnaces are particularly suitable. Such furnaces are well known to skilled practitioners. One such furnace is described in Beatty, U.S. Pat. No. 4,837,924, the entirety of which is incorporated herein by reference.

[0082] The yield of silicon carbide fiber by the method of the invention is high. Conversion of 100 percent of the reactants to silicon carbide would yield 41.7 wt percent bound silicon carbide. For example, after 1 hour in argon at 1675°C, a blend of silica and carbon in a molar ratio of 2.7 carbon per 1 silica, together with 1.5 wt percent FeSO₄ and 0.6 wt percent calcium oxalate, yielded 96.1 percent conversion to silicon carbide. In accordance with the method of the invention, conversion generally is at least 80 percent, preferably is about 85 percent, more preferably is at least about 90 percent, and most preferably is at least above 95 percent.

[0083] It has been discovered that the sulfur content of isotropic pitch carbon fiber, which exceeded about 0.25 wt percent even after pre-treatment, surprisingly did not have an adverse effect on conversion of carbon to silicon carbide. At a sulfur concentration greater than about 0.25 wt percent, skilled practitioners would have been expected an adverse effect on quality and conversion.

[0084] Product composition may be reliably determined by means of a hot HF extraction technique, which removes any silica present. The equivalent silica that has chemically reacted may then be calculated by difference and translated into the quantity of silicon carbide present. The unreacted carbon also may be computed by difference. The total carbon present in the product, both in the silicon carbide and in the unreacted carbon, was determined using equipment manufactured by the Laboratory Equipment Company of Benton Harbor, Mich. (LECO). This provides an independent cross-check on the unreacted carbon computed from HF extraction.

[0085] The absorption of microwave energy is easily and quickly confirmed. A cavity 1.0 inches in diameter by 0.25 inches deep in a 3×3×2 inch rigid Kaowool® insulation block (microwave transparent) is filled with silicon carbide fibers of the invention. The fiber-filled block is placed at a specific spot in a kilowatt, 2.45 GHz microwave oven and heated until the fibers achieved red heat, i.e., about 750-800°C. Each of the preferred fiber products described in the Examples achieved red heat in between about 3 and about 6 seconds. Silicon carbide whiskers require about 5 seconds to achieve red heat under the same conditions.

[0086] As set forth above, the silicon carbide fibers of the present invention are β-silicon carbide. This determination is made by x-ray diffraction techniques in a manner known to skilled artisans.

[0087] The absence of whiskers in silicon carbide fiber product of the invention is illustrated by SEM photographs of well-dispersed samples. Whiskers are not found in degraded silicon carbide of the invention in SEM photographs at 200x magnification.

EXAMPLES

[0088] The following examples are meant to illustrate the invention, not to limit it in any way. For example, isotropic pitch carbon fibers from any source maybe used. Similarly, other forms of silica can be used. The scope of the invention is limited only by the claims.

[0089] Throughout the Examples, “wt percent” means “weight percent based on the combined weight of the carbon fiber and silica” when referring to starting materials.

Example 1

[0090] Quantities of discontinuous divers carbon fibers were used to form silicon carbide fiber. Carbon fiber was placed in a plow mixer equipped with a high-speed chopper and “opened” for 1 minute. With the mixer still running, an aqueous dispersion of ferrous sulfate and calcium oxalate was added. The quantity of FeSO₄ was sufficient to provide 1.5 wt percent FeSO₄ based on the combined weight of the carbon fiber and silica to be added immediately thereafter. The quantity of calcium oxalate was sufficient to provide 0.6 wt percent calcium oxalate based on the combined weight of carbonized fiber and silica to be added.

[0091] After 3 minutes of blending, a quantity of Cab-O-Sil® grade M5, a fumed silica, sufficient to provide 2.7 moles of carbon per mole of silica was added. Two additional minutes of blending followed.

[0092] The blend product was loaded into capped graphite crucibles, placed in a laboratory graphite furnace and slowly dried at less than 250°C under flowing argon. Then, the temperature was raised to 1675°C and held for one hour under flowing argon. The sample was allowed to cool and was examined.

[0093] The carbon fibers used in this Example are the same as those described in Table 1, and were used in equal mass quantities. Only the silicon carbide product made from Carbosel® P-200 is an example of the invention; silicon carbide fibers from the other two fibers are comparative examples.

[0094] Product properties are summarized and related SEM photographs are identified in Table 2 below:

<table>
<thead>
<tr>
<th>Discontinuous Carbon Fiber, Source</th>
<th>Product</th>
<th>Isotropic Fiber, Carbosel® P-200</th>
<th>MoStophase Pitch Fiber, Cytec</th>
<th>ThermaKring® DND X</th>
<th>PAN Fiber, Fortafil® M275</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whiskers, wt %</td>
<td>Unreacted Carbon, wt %</td>
<td>Extractable Silica, wt %</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>96.1</td>
<td>None</td>
<td>Detected</td>
<td>None</td>
<td>Detected</td>
<td></td>
</tr>
<tr>
<td>97.0</td>
<td>Numerous</td>
<td>2</td>
<td>0.3</td>
<td>0.2</td>
<td></td>
</tr>
<tr>
<td>103.6</td>
<td>Numerous</td>
<td>3</td>
<td>4.6</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

[0095] X-ray diffraction analysis shows the fibers prepared from P-200 to be predominantly P-silicon carbide, as illustrated in FIG. 12.
The unreacted carbon composition was determined by LECO analysis, and the extractable silica by HF extraction. The wt percent silicon carbide conversion was determined by calculating what fraction of the carbon fiber (less unreacted carbon) formed silicon carbide fiber. The failure of the fractional compositions of the various samples to sum to 100 percent is not surprising, but rather falls within the range of experimental error. The analyses indicate the absence of unreacted carbon and of extractable silica in silicon carbide fiber of the invention. The data also show that product of the invention is essentially devoid of whiskers, as none were found in the product. However, use of other discontinuous carbon fiber types resulted in products having numerous whiskers.

As can be seen from Table 2, the isotropic pitch fiber of the invention yielded silicon carbide fiber product having 96.1 wt % silicon carbide (i.e., essentially all silicon carbide within the range of experimental error), with neither unreacted carbon nor extractable silica. Also, no whiskers are detectable, even at 500x magnification, as can be seen in FIGS. 1b and 1d. In contradistinction, the comparative examples resulted in significant quantities of whiskers, as can be seen in FIGS. 2 and 3. Also, FIGS. 1a-1d highlight the fact that the smoothness of the isotropic fibers is maintained in the resultant silicon carbide fiber product.

Quantities of Carboflex® P-200 isotropic carbon fibers were heated under argon for times and at temperatures as set forth in Table 3 below. The treated carbon fibers then were reacted with silica in the laboratory furnace in accordance with the method of Example 1. FIGS. 4, 5 and 6 of samples of the resultant silicon carbide fiber thus produced, show that no whiskers were produced. Indeed, no whiskers were found after thorough examination of each of the product samples.

The following Table 3 summarizes characteristics of the resulting silicon carbide products of the invention:

<table>
<thead>
<tr>
<th>Blend</th>
<th>Product</th>
<th>molar ratio</th>
<th>Wt % Carbon/</th>
<th>Wt % SiC Conversion</th>
<th>Specific Surface</th>
<th>Length, microns</th>
<th>Diameter, microns</th>
<th>FIG.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>wt %</td>
<td>m/g</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carboflex® Isotropic Carbon Fiber, P-200</td>
<td>As received</td>
<td>96.1</td>
<td>None</td>
<td>11.2</td>
<td>188</td>
<td>14.5</td>
<td>1b, 1d</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pretreated 1 hr at 1500° C</td>
<td>97.5</td>
<td>None</td>
<td>8.6</td>
<td>114</td>
<td>11.9</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pretreated 1 hr at 1800° C</td>
<td>95.8</td>
<td>None</td>
<td>6.4</td>
<td>105</td>
<td>12.3</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pretreated 7 hr at 1800° C</td>
<td>97.3</td>
<td>None</td>
<td>2.8</td>
<td>133</td>
<td>11.6</td>
<td>6</td>
<td></td>
</tr>
</tbody>
</table>

Neither unreacted carbon nor extractable silica was found in any product. The quality of the product fibers was very good, as can be seen from the Figures. The fiber length and diameter of the products of pretreated fibers were somewhat lower than those of the product of the untreated fibers, reflecting the same reductions in the carbon fibers, as set forth in Table 1.

The preheat treatment of the isotropic fibers (Table 1) shows that the carbon fibers are reduced in length and diameter. These analyses, together with the data in Table 3 for the resultant silicon carbide fibers of the invention produced, show that the effect of preheat treating of Carboflex® P-200 carbon fiber was to reduce both the average lengths and diameters, reduce surface areas markedly, and lower the sulfur content of the carbon fiber. During the heat treatment of the fiber, sulfur and sulfurous products were detected in the exhaust gases.

FIGS. 4, 5 and 6 illustrate that the fibers of the invention of Example 2 are essentially devoid of whiskers. The reductions of length and fiber diameter occur mainly during the preheat treatment, rather than during conversion to silicon carbide fiber.

The data also show that surprisingly, the sulfur present in the fiber in concentrations between 0.26 and 1.43 wt % did not inhibit conversion to silicon carbide fiber.

The graphic structure developed in the precursor due to preheating the isotropic carbon fiber for 7 hours does not result in the presence of whiskers in the silicon carbide fiber product of the invention. However, both the mesophase pitch fiber and PAN fiber have very low sulfur content less than (0.1 wt %) and give rise to whiskers when reacted with silica.

Data for the silicon carbide product is set forth in Table 4, which also contains data relating to Carboflex® P-200 from Example 1.

<table>
<thead>
<tr>
<th>Blend</th>
<th>Product</th>
<th>molar ratio</th>
<th>Wt % Carbon/</th>
<th>Wt % SiC Conversion</th>
<th>Specific Surface</th>
<th>Length, microns</th>
<th>Diameter, microns</th>
<th>FIG.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.7:1.0</td>
<td>96.1</td>
<td>None</td>
<td>11.2</td>
<td>188</td>
<td>14.5</td>
<td>1b, 1d</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.0:1.0</td>
<td>98.4</td>
<td>0.2</td>
<td>None</td>
<td>12</td>
<td>122</td>
<td>12.0</td>
<td>7</td>
</tr>
</tbody>
</table>

The fiber quality of the fibers produced from the 2.7:1 blend of Example 1 was superior to that of those produced from the stoichiometric blend (3:1) of this Example, but both were acceptable. No whiskers were produced in either case. The quality differences are difficult to see by comparing Figs. 1b and 1d with FIG. 7, but the greater reduction in length and diameter of the product fiber of this Example lends support to this observation.

Silicon carbide fiber of the invention was prepared in accordance with Example 1, except that the Carboflex® P-200 of Example 1 was replaced by an equal weight of Carboflex® P-600, a lower sulfur grade of isotropic carbon fiber. The molar ratio of carbon to silica remained 2.7:1.
No whiskers were produced. Table 5 sets forth product characteristics for silicon carbide fiber of this Example, together with comparable data from Examples 1 and 2.

<table>
<thead>
<tr>
<th>Source, Treatment</th>
<th>Sulfur, wt %</th>
<th>SiC Conversion</th>
<th>Fiber Density, g/cc</th>
<th>Figure</th>
</tr>
</thead>
<tbody>
<tr>
<td>P-200, as received</td>
<td>1.43</td>
<td>96.1</td>
<td>3.19</td>
<td>1b, 1d</td>
</tr>
<tr>
<td>Pretreated 1 hr at 1500°C</td>
<td>0.85</td>
<td>96.5</td>
<td>3.18</td>
<td>4</td>
</tr>
<tr>
<td>Pretreated 1 hr at 1800°C</td>
<td>0.26</td>
<td>95.8</td>
<td>3.20</td>
<td>5</td>
</tr>
<tr>
<td>Pretreated 7 hr at 1800°C</td>
<td>0.36</td>
<td>97.3</td>
<td>3.20</td>
<td>6</td>
</tr>
<tr>
<td>P-600, as received</td>
<td>0.59</td>
<td>99.3</td>
<td>3.20</td>
<td>8</td>
</tr>
</tbody>
</table>

Table 5 suggests that the effect of sulfur content on the production of silicon carbide fiber produced was minimal. It is surprising that the sulfur is not detrimental to the conversion to silicon carbide. Further, the presence of sulfur in the carbon fiber did not reduce the quality of the fibers.

Example 5

Silicon carbide fiber of the invention was prepared with each type of promoter individually, and without any promoters. The fibers were prepared in accordance with the method of Example 1.

Data on the composition and unreacted carbon for each of the fibers made using only one or no promoter is set forth in Table 6 below. For comparison, the same information of product of Example 2 made using both promoters also is set forth in Table 6.

<table>
<thead>
<tr>
<th>Starting Materials</th>
<th>Product per 1 mole of Si</th>
<th>1.5 wt % FeSO₄</th>
<th>0.6 wt % Ca(OOC)₂</th>
<th>SiC Conversion</th>
<th>Carbon, Fig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>None</td>
<td>4</td>
</tr>
<tr>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>Detected</td>
<td>9</td>
</tr>
<tr>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>92.0</td>
<td>10</td>
</tr>
<tr>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>97.3</td>
<td>11a, Detected</td>
</tr>
</tbody>
</table>

No whiskers were observed in any of the product samples, as can be seen in the Figures. This result is surprising, as the skilled practitioner would have expected the presence of promoters to have produced whiskers. Both iron and calcium salts are reported to be whisker promoters.

The Figures illustrate the degraded quality of the fibers. Crystalline structure and granularity of the silicon carbide fiber of the invention are seen most clearly in FIGS. 11a and 11b. Such degraded fiber material may have different commercial uses from the uses of non-degraded fiber.

Thus it can be seen that the presence of both types of promoters greatly increases fiber quality. Silicon carbide fiber of the invention produced in accordance with a preferred embodiment, in which two promoters are present, essentially retains the morphology of the isotropic pitch carbon fiber used. Thus, the resultant silicon carbide fiber is formed in smooth-surfaced cylinder sharing dimensions similar to the dimensions of the carbon fiber starting material. Although both the diameter and length of product fibers typically have reduced diameter and length, as illustrated in these Examples.

Example 6

Silicon carbide fiber was prepared in accordance with the method of Example 1, except that the atmosphere was nitrogen rather than argon. FIG. 13 is an SEM photograph at 200x magnification of the silicon carbide fiber thus produced. A significant quantity of whiskers can be seen in the photograph. Comparison of FIG. 13 with FIG. 16 illustrates the whisker-laden nature of fiber produced under nitrogen with the whisker-free nature of silicon carbide fiber of the invention prepared under argon.

Specific surface areas of both the initial carbon fibers and the silicon carbide fiber product were determined employing the BET method. Densities were determined using helium pycnometry. The specific surface area of the carbon fibers was about 30 m²/g. The silicon carbide fibers made in accordance with the method of the Examples had specific surface areas of between about 2.5 and 12.0 m²/g and densities of about 3.2 g/cc.

Any source of fine silica can be used in the claimed invention. Furthermore, the method of the claimed invention results in essentially no whiskers and P-silicon carbide fiber of high quality and gray-green color when using promoters. Fiber quality is degraded if only one or no promoters are used, but the resultant silicon carbide fiber of the invention is essentially devoid of whiskers. The conversion of carbon fiber to silicon carbide fiber does not significantly change the morphology when using two promoters. The degradation in fiber quality experienced in the absence of both promoters changes the morphology, as shown in FIGS. 9, 10, 11a, and 11b.

We claim:

1. A method for making discontinuous silicon carbide fibers essentially devoid of whiskers, comprising the steps of:
   (a) admixing discontinuous isotropic carbon fiber and silica to form a fiber/silica mixture;
   (b) drying the fiber/silica mixture; and
   (c) reacting the dried fiber/silica mixture in an essentially inert atmosphere in a resistance furnace for a time and at a temperature sufficient to form the discontinuous silicon carbide fibers essentially devoid of whiskers.
2. The method of claim 1 wherein the discontinuous isotropic carbon fiber is isotropic pitch carbon fiber.
3. The method of claim 1 wherein the silica is present in a molar excess relative to the discontinuous isotropic carbon fibers.
4. The method of claim 1 wherein the essentially inert atmosphere comprises argon.
5. The method of claim 1 wherein the discontinuous isotropic carbon fibers has a sulfur concentration greater than about 0.25 wt percent.
6. Discontinuous silicon carbide fibers essentially devoid of whiskers, said fibers prepared by
   (a) admixing discontinuous isotropic carbon fiber and silica to form a fiber/silica mixture;
   (b) drying the fiber/silica mixture; and
   (c) heating the dried fiber/silica mixture in a resistance furnace in an essentially inert atmosphere for a time and at a temperature sufficient to form the discontinuous silicon carbide fibers.
7. The fibers of claim 6 wherein the silicon carbide fibers are essentially pure β-silicon carbide.
8. Discontinuous silicon carbide fibers essentially devoid of whiskers made by reacting discontinuous isotropic carbon fibers and silica, said silicon carbide fibers comprising at least about 90 wt percent β-silicon carbide, a diameter between about 3 and about 25 microns, a length of less than about 1 mm, and essentially the same morphology of the discontinuous isotropic carbon fibers.
9. A method for making discontinuous silicon carbide fibers essentially devoid of whiskers comprising the steps of:
   (a) admixing discontinuous isotropic carbon fiber and silica, and a promoter to form a fiber/silica mixture;
   (b) drying the fiber/silica mixture; and
   (c) reacting the dried fiber/silica mixture in an essentially inert atmosphere in a resistance furnace for a time and at a temperature sufficient to form the discontinuous silicon carbide fibers essentially devoid of whiskers.
10. The method of claim 9 wherein the essentially inert atmosphere comprises argon.
11. The method of claim 9 wherein the discontinuous isotropic carbon fiber is isotropic pitch carbon fiber.
12. The method of claim 9 wherein the silica is present in a molar excess relative to the discontinuous isotropic carbon fibers.
13. The method of claim 9 wherein the discontinuous isotropic carbon fiber has a sulfur concentration greater than about 0.25 wt percent.
14. The method of claim 9 wherein the promoter is selected from the group consisting of the salts, compounds, and complexes of iron, cobalt, or nickel, and blends thereof, and the salts, compounds, and complexes of alkali metals or alkaline earth metals and blends thereof.
15. The method of claim 14 wherein the promoter is selected from the group consisting of ferrous sulfate and calcium oxalate.
16. A method for making discontinuous silicon carbide fibers, essentially devoid of whiskers comprising the steps of:
   (a) admixing discontinuous isotropic carbon fiber and silica, and at least two promoters to form a fiber/silica mixture;
   (b) drying the fiber/silica mixture; and
   (c) reacting the dried fiber/silica mixture in an essentially inert atmosphere in a resistance furnace for a time and at a temperature sufficient to form the discontinuous silicon carbide fibers essentially devoid of whiskers.
17. The method of claim 16 wherein the essentially inert atmosphere comprises argon.
18. The method of claim 16 wherein the discontinuous isotropic carbon is isotropic pitch carbon fiber.
19. The method of claim 16 wherein the silica is present in a molar excess relative to the carbonized fibers.
20. The method of claim 16 wherein the discontinuous isotropic carbon fibers has a sulfur concentration greater than about 0.25 wt percent.
21. The method of claim 20 wherein the promoters comprise
   (a) a first promoter selected from the group consisting of the salts, compounds, and complexes of iron, cobalt, or nickel, and blends thereof; and
   (b) a second promoter selected from the group consisting of the salts, compounds, and complexes of alkali metals or alkaline earth metals, and blends thereof.
22. The method of claim 21 wherein promoter (a) comprises an amount equivalent to about 0.5 to about 5.0 wt percent of ferrous sulfate based on the combined weight of the carbonized fiber and silica and promoter (b) comprises an amount equivalent to about 0.2 to about 3.0 wt percent of calcium oxalate, based on the combined weight of the carbonized fiber and silica.
23. The method of claim 22 wherein promoter (a) is ferrous sulfate and promoter (b) is calcium oxalate.
24. A regenerative medium for filtering volatile organic compounds from fluids comprising silicon carbide fiber made in accordance with the method of claim 16.