A schematic drawing of crack bridging by (a) a straight fibers and (b) a bone-shaped fiber. For a weak interface, the straight fiber will debond and lose the load carried in it. However, the load transfer is not affected for the bone-shaped fiber, which enables effective crack bridging.

Short fiber–reinforced composite including a matrix material; and, a minor percentage of short–fibers having an altered fiber morphology, such a fiber morphology being wherein selected portions of said short–fiber are greater in cross–sectional diameter than the cross–sectional diameter of said fiber at other portions of said fiber, such cross–sectional diameter being perpendicular across said fiber’s main axis. Such fibers can be shaped as bone–shaped or dumbbell–shaped in structure.
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REINFORCED COMPOSITES INCLUDING BONE-SHAPED SHORT FIBERS

The present application claims the benefit of U.S. provisional application 60/074,726, filed on February 13, 1998.

FIELD OF THE INVENTION

The present invention relates to reinforced composites and more particularly to reinforced composites containing short fibers having a bone-shaped morphology. This invention is the result of a contract with the Department of Energy (Contract No. W-7405-ENG-36).

BACKGROUND OF THE INVENTION

Compared to continuous filament composites, short-fiber composites are advantageous. They often have improved strength and stiffness over the reinforced matrix. In addition, they can be adapted to conventional manufacturing techniques, such as powder metallurgy, casting, molding, drawing, extruding, machining and welding. As a result, the part fabrication cost can be relatively low, which is an important design criteria. Short-fiber composites also can be made with relatively isotropic mechanical properties and can easily be molded into the complex shapes required in some applications. These advantages have led to wide applications of these composites in automobile, sporting goods and cutting tools industries.

Interfaces between the fiber and the matrix in short-fiber composites play a critical role and, in many cases, become a limiting factor in improving such mechanical properties as strength and toughness of composites. For a short fiber composite, a strong interface is desired to effectively transfer load from the matrix to the fiber. A stronger interface can reduce the ineffective length at both ends of the fiber and, therefore, can increase the effective length that carries load. However, with a strong interface, it is difficult to relieve fiber stress concentration in front of an approaching crack; and such stress concentration can result in fiber breakage. This effect is particularly severe for ceramic matrix composites, because of their low matrix toughness and lack of plasticity. Even for a composite with a highly ductile matrix, such as plastics, too strong of an
interface may still cause successive adjacent fiber breakage and subsequently reduce composite toughness. Although crack bridging in weakly bonded continuous filament composites has proved to be useful in enhancing the composite fracture toughness, its usefulness in short-fiber composites is usually limited as a weak interface significantly decreases the fiber load-carrying length. A compromise in the interfacial bond strength may lead to a significant loss of the composite strength and only a minimal improvement of the composite toughness due to complete fiber interfacial debonding and pullout during loading.

The aforementioned problems are intrinsic with the conventional short straight-fiber composites. These problems are caused by fiber morphology, and cannot be solved by modifying the fiber/matrix interfacial property, which is a reason that worldwide efforts in the design and optimization of fiber-matrix interfaces in recent decades has failed to solve the low strength and toughness problem of short-fiber composites. The key to solving the problem is to obtain both a weak interface and a strong load transfer mechanism from the matrix to the fiber.

It is an object of the present invention to provide short fiber composites wherein said short fibers have an altered morphology, particularly wherein said short fibers have enlarged ends so as to resemble a bone shape or dumbbell shape.

It is a further object of the invention to provide short fibers having an altered morphology, wherein said short fibers have enlarged ends so as to resemble a bone shape or dumbbell shape.

It is still another object of the present invention to provide a method of forming short fiber composites wherein said short fibers have an altered morphology, particularly wherein said short fibers have enlarged ends so as to resemble a bone shape or dumbbell shape and a process of substantially aligning the short fibers having the altered morphology within the composites.
SUMMARY OF THE INVENTION

To achieve the foregoing and other objects, and in accordance with the purposes of the present invention, as embodied and broadly described herein, the present invention provides a short fiber-reinforced composite including a matrix material; and, a minor percentage of short-fibers having a fiber morphology wherein ends of said short-fiber are greater in cross-sectional diameter than a cross-sectional diameter of said fiber at a central portion of said fiber perpendicular across said fiber's main axis.

The present invention further provides a short fiber of a material selected from the group consisting of glass, polyesters, polyamides, polyacrylics, polyolefins, cellulose, cellulose derivatives, alumina, aluminum nitride, aluminum silicate, boron carbide, carbon, graphite, magnesium oxide, mullite, silicon carbide, silicon dioxide, silicon nitride, titanium nitride, zirconium dioxide, aluminum, iron, nickel, niobium, niobium, titanium and tungsten, said short fiber characterized as having a fiber morphology wherein ends of said short fibers are greater in cross-sectional diameter than a cross-sectional diameter of said fibers at a central portion of said fibers perpendicular across a main axis of said fibers.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1 shows a sample composite used in demonstration of the present invention.

FIGURE 2 shows stress-strain curves of both bone-shaped short-fiber composites and conventional straight short-fiber composites with nickel fibers in demonstration of the present invention.

FIGURE 3(a) is a schematic drawing of crack bridging by straight short fibers and FIGURE 3(b) is a schematic drawing of crack bridging by bone-shaped short (BSS) fibers.
FIGURE 4 shows stress-strain curves of both bone-shaped short-fiber composites and conventional straight short (CSS)-fiber composites with polyethylene fibers in demonstration of the present invention.

FIGURE 5 shows a fiber with enlarged fiber ends in accordance with the present invention.

FIGURE 6 shows load-displacement curves (four point bending) of both bone-shaped short-fiber composites and conventional straight short-fiber composites each with steel fibers in a concrete matrix, together with the stress-strain curve for an un-reinforced concrete matrix.

FIGURE 7 shows the crack length-load curves obtained from double cantilever beam tests of both bone-shaped short-fiber composites and conventional straight short-fiber composites with fiber lengths of 3 mm where the solid markers represent data points for bone-shaped short-fiber composites and the hollow markers represent data points for straight short-fiber composites.

FIGURE 8 (a) shows the total normalized energies, E(a) as a function of crack length for double cantilever beam tests of both bone-shaped short-fiber composites and conventional straight short-fiber composites with fiber lengths of 3 mm. The solid and hollow markers represent data points for bone-shaped short-fiber composites and conventional straight short-fiber composites, respectively and FIGURE 8(b) shows the supplied energy for a crack to propagate by a unit length, ε(a), as a function of crack length.

DETAILED DESCRIPTION

The present invention is concerned with short fiber composites wherein the short fibers have a modified or altered morphology. The modified morphology of the short fibers is such that they include one or more fiber regions or fiber portions of a greater
cross-sectional diameter than the predominant cross-sectional diameter of the short fibers in the majority portions of the fiber. By predominant cross-sectional diameter is meant that the majority portions of the short fiber have a selected cross-sectional diameter, while one or more minority portions of the short fiber have the greater cross-sectional diameter to yield the modified morphology. In one embodiment, the short fiber resembles a bone shape or dumbbell shape with the fiber regions having the greater cross-sectional diameters being situated at the ends. In another embodiment, the short fiber can have more than two fiber regions with the greater cross-sectional diameter with one or two of the fiber regions occurring at the ends of the fibers and the other enlarged fiber regions occurring somewhere along the longitudinal length of the fiber. The present invention includes reinforced composites containing the short fibers with the altered morphology whether the short fibers are bone-shaped, i.e. the enlarged fiber portions are at the ends of the short fiber, or the short fibers have other configurations, so long as the short fibers have the greater cross-sectional diameter in one or more fiber regions, preferably at least two fiber regions.

A bone-shaped short fiber with two enlarged ends can effectively transfer load from a composite matrix onto a fiber at both ends by matrix/fiber interlocking, hence minimizing the need for a strong interface to transfer load. As a result, crack bridging across weakly bonded fibers, a concept from continuous filament composites, can be used in short-fiber composites. The enlarged ends help to reduce the fiber stress concentration at a matrix crack tip by allowing interface sliding/debonding without complete fiber pullout. Since load can be transferred through mechanical interlocking between the enlarged fiber ends and the matrix, the load-carrying potential of short fibers can be better utilized. If the shape and size of the enlarged fiber ends can be optimized in such a way that the short fiber would be pulled out with difficulty, much more energy would be consumed during the crack propagation, which will significantly improve the toughness.
These advantages should translate into significantly higher strength for this class of bone-shaped short-fiber composites having weak-to-moderate interfaces.

Matrix materials in the present invention can include metals such as aluminum, and other suitable metals or metal alloys, ceramics such as alumina, molybdenum silicide, silicon nitride, silicon carbide, boron carbide and the like, polymers including thermoplastic polymers or thermoset polymers, and concretes. Matrix material polymers can be selected from materials such as polyester, epoxy, polycarbonate, and the like. Concretes generally are formed from a mixture of aggregate, water, and a binder such as portland cement. In addition to concrete, analogous matrix materials such as mortar, stucco, grout and the like can be reinforced with the short fibers of the present invention.

Fiber materials can include glass, polyesters, polyamides, polyacrylcs, polyolefins, cellulose, cellulose derivatives, alumina, aluminum nitride, aluminum silicate, boron carbide, carbon or graphite, magnesium oxide, mullite, silicon carbide, silicon dioxide, silicon nitride, titanium nitride, zirconium dioxide, and generally any metal or metal alloy, especially aluminum, iron, nickel, niobium, titanium and tungsten. Among the various classes of polymeric materials are included numerous species of polymers out of which the short fibers with the altered morphology as described in the present invention can be prepared. For example, among polyolefins can be included polyethylene, polypropylene, polybutylene and the like. Other polymers such as polyimides and polybenzimidazoles may be used as well. Celluloses can include materials such as rayon.

In one embodiment of the present invention, a short fiber composite is comprised of a matrix material selected from the group consisting of metals, ceramics, polymers and concretes, and a minor percentage of non-metallic short fibers having a fiber morphology wherein said short fibers have one or more fiber regions of a greater cross-sectional diameter than a predominant cross-sectional diameter of said short fibers at majority portions of said short fibers, said cross-sectional diameter measured perpendicular across a main axis of said short fibers.
In another embodiment of the present invention, a short fiber composite is comprised of a matrix material selected from the group consisting of ceramics, polymers and concretes, and a minor percentage of short fibers having a fiber morphology wherein said short fibers have one or more fiber regions of a greater cross-sectional diameter than a predominant cross-sectional diameter of said short fibers at majority portions of said short fibers, said cross-sectional diameter measured perpendicular across a main axis of said short fibers.

The fibers are a minor portion or percentage by volume of the short-fiber reinforced composites of the present invention. By "minor" is meant an amount less than 50 percent, more usually an amount less than about 30 percent, more generally an amount less than about 20 percent.

The bone-shaped fiber dimensions or sizes in the composites of the present invention will vary depending upon the particular application. In applications such as filled polymer composites the bone-shaped fiber can generally have an average fiber length within the range of about 0.1 millimeters (mm) to about 10.0 centimeters (cm), preferably of an average fiber length within the range of about 0.1 mm to about 1.0 cm. Longer fiber lengths may be employed so long as they remain processable into the composite matrix. Further, the bone-shaped fibers in the present invention are generally of an average fiber diameter within the range of about 5 microns (μm) to about 1000 μm, preferably of an average fiber diameter within the range of about 10 μm to about 100 μm. The aspect ratio of the bone-shaped fibers is generally within the range of about 10-50.

In the compositions of the present invention, mixtures of bone-shaped fibers may be employed wherein the mixtures include bone-shaped fibers having different fiber lengths and fiber diameters. For example, a mixture of bone-shaped fibers could include various fibers of lengths varying from about 0.1 cm to about 10.0 cm. Similarly, composites may
include a mixture of bone-shaped fibers with fibers of varying diameters or composites may include a mixture of bone-shaped fibers with fibers of varying diameters and lengths.

One or more fiber region in the short fibers utilized in the present invention has a greater cross-sectional diameter than the predominant cross-sectional diameter of the short fibers at majority portions of the short fibers. For example, the ends of the short fibers utilized in the present invention can be enlarged beyond the diameter of the central portion of the fiber such that the morphology of the fibers is bone-shaped or dumbbell shaped in appearance. The enlarged fiber regions or ends are generally at least about five percent greater in cross-sectional diameter than the central portion of the fiber, although the percentage may be changed depending upon the application for the final composite.

The altered fiber morphology can be formed in any suitable manner. For example, polymer short fibers with enlarged ends (dumbbell-shaped fibers) can be prepared by cutting a fiber at pre-selected intervals with, e.g., a torch flame such that the general length of the short fiber is selected. The ends of the short fibers can be enlarged by this process. In another manner, where a fiber is formed using an appropriate spinneret, the rate of formation (pulling) of the fiber can be regularly or intermittently altered to result in controlled alteration of the cross-sectional diameter. After a long length of fiber with regions of greater cross-sectional diameter are formed, the long fiber length can be chopped into the desired lengths of short fiber with the short fiber retaining the desired fiber regions or fiber portions with the increased cross-sectional diameter. In the case of metal fibers, the preferred geometry is of the bone-shape with the two ends of the greater cross-sectional diameter. Such metal fibers with the enlarged ends can be formed by, e.g., pounding the ends of a straight metal wire fiber to enlarge the ends. Other manners of forming the short fibers of the present invention with the altered morphology will be readily apparent to those of skill in the art.

The main axis of the short fibers in the present invention is along the length of the fiber.
The bone-shaped short-fiber reinforced composites of the present invention have enhanced properties over straight short-fiber reinforced composites. Enhanced properties include higher yielding and ultimate strength, higher Young's modulus, and greater toughness such as fracture toughness.

The present invention is more particularly described in the following examples which are intended as illustrative only, since numerous modifications and variations will be apparent to those skilled in the art.

EXAMPLE 1

Commercially pure nickel filament with a diameter of 76.2 μm was used as precursor of bone-shaped and straight short fibers. Bone-shaped short fibers were fabricated using an automatic machine described below. The machine automatically fed and swung the nickel filament through a flame from a mini-hydrogen torch. The flame cut the nickel filament by melting it, and the melted nickel formed two balls on the two cut ends for each cutting due to surface tension of the melted nickel. Average resultant ball size was determined by the size of the torch. One prepared batch of the bone-shaped nickel fibers had an average ball diameter of 183 μm and a length of 2.5 mm. Short straight nickel fibers were obtained by cutting the nickel filament with scissors.

After the nickel bone-shaped and straight fibers were fabricated, they were coated with a bond-weakening agent of n-octadecyltrichloro-silane (n-OTS). The coating procedure was as follows: the nickel fibers were washed in an ultra-sonic cleaner in acetone to degrease the fiber surface and then in ethanol to remove any acetone residue. The cleaned fibers were then dried and placed in a solution of ethanol and 1 percent by volume n-OTS and put into an oven at 50 °C for 1 hour. The final step of the fiber coating process was to pour out the ethanol/n-OTS solution and place the fibers back in the oven at 100 °C for 2 hours to bake on the n-OTS coating.
Polyester was chosen as the matrix material for the subsequent composites. Suspension of the nickel fibers in the uncured polyester during fabrication to obtain a spatially-random distribution of the nickel fibers was accomplished by addition of 0.39 g of amorphous fumed silica (Cab-O-Sil, Cabot Corporation, Tuscola, Illinois) to 10 ml of polyester as a thickening agent before mixing with the nickel fibers. The mixture of polyester and Cab-O-Sil was then placed through repeated cycles of vacuum/ambient pressure to remove any air bubbles introduced during mixing. A hardening agent of methyl ethyl ketone peroxide (0.6 percent by volume) and 1.5 g of nickel fiber was then added. The new mixture was again placed through vacuum/ambient pressure cycles to remove any air bubbles. Finally, the mixture was extruded into a sample mold through a syringe. The sample mold produced a net-shaped sample for mechanical testing. The extrusion process aligned the nickel fibers to some extent. Further alignment was obtained using the principle of elongation flow, which was achieved by sliding two mold parts against one another thereby forcing the mixture to flow in the longitudinal direction of the sample. The sample mold was then mounted onto a slowly rotating machine for four hours to prevent the fibers from settling down to the mold bottom. Afterward, the samples were allowed to cure at room temperature for seven days before mechanical testing. Excellent fiber alignment and random spatial-distribution were achieved using the above procedures.

Both bone-shaped short-fiber composite samples and straight short-fiber composite samples were fabricated using the above procedure. They all had a fiber length of 2.5 mm and fiber volume fraction of 1.7 percent. The only difference between the bone-shaped short-fiber composite samples and straight short-fiber composite samples was the fiber morphology, thus allowing examination of the effect of fiber morphology on the strength of composites. Fiber-free blank matrix samples were also fabricated for comparison.
The sample dimension is shown in Figure 1. Tensile testing was performed using a Model 1125 Instron testing machine. An extensometer with a one-inch gage length was used to measure the strain. A constant strain rate of 0.0001 s⁻¹ was employed for all samples. Fracture surface was investigated using a JEOL 6300FXV Scanning Electron Microscope (SEM). An optical microscope was used to investigate fiber alignment.

The stress-strain curves of both bone-shaped short-fiber composites and conventional straight short-fiber composites are shown in Figure 2. It can be seen that the strength of composite samples reinforced with bone-shaped short fibers was significantly higher than that of composite samples reinforced with straight short fibers. It can also be seen that the Young’s modulus of the bone-shaped short fiber composite samples was higher than that of the straight short fiber composite samples. The determination of yielding strength as shown in Fig. 2 is according to the ASTM standard D 638M-91a, which is for tensile properties of plastics. Listed in Table 1 is the yielding strength for each sample tested. The average strength of the bone-shaped short fiber composite samples improve by 10.2 percent over that of conventional straight short fiber composite samples.

Table 1. Yielding Strengths of bone-shaped and straight short-fiber composites and the polyester matrix.

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<tr>
<th>Sample Number</th>
<th>Bone-shaped (MPa)</th>
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<tr>
<td>1</td>
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<td>Average</td>
<td>24.43</td>
<td>22.17</td>
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Note that the fiber contents in both the bone-shaped short-fiber composites and straight short-fiber composites are only 1.7 percent. The 10.2 percent improvement in the yielding strength of bone-shaped short-fiber composites with a 1.7 percent fiber volume fraction indicates that the bone-shaped short fibers are much more effective in strengthening the polyester matrix than the straight short-fibers. Observation with the optical microscope of the composite samples showed that the short fibers in both the bone-shaped and straight short fiber composites were well aligned with the long axis of the short fibers aligned parallel to the long axis of the composite sample. Therefore, the composite samples can be considered as reinforced by unidirectional fibers. Using a simple rule of mixture, the composite strength can be described by the equation:

$$\sigma_c = V_f \sigma_{ef} + (1 - V_f) \sigma_m$$  \hspace{1cm} (1)

where $\sigma_c$ is the yielding strength of composites, $V_f$ is the fiber volume fraction, $\sigma_m$ is the average yielding stress in the matrix, $\sigma_{ef}$ is the effective fiber stress at the composite yielding. Note that the stress distribution along the fiber length is not uniform. For a straight short fiber, $\sigma_{ef}$ can be considered as the average stress along the fiber at composite yielding. For a bone-shaped short fiber, $\sigma_{ef}$ is the average stress along the fiber assuming the fiber length equals to the volume of a fiber divided by its cross-section, which is longer than the real bone-shaped fiber because of the enlarged ends. The effective stress for the bone-shaped and straight short fibers at composite yielding can be calculated from equation (1) as

$$\sigma_{ef} = \frac{\sigma_c - (1 - V_f) \sigma_m}{V_f}$$  \hspace{1cm} (2)

and

$$\sigma_{ef} = \frac{\sigma_c - (1 - V_f) \sigma_m}{V_f}$$  \hspace{1cm} (3)
where $\sigma^b_{ef}$ and $\sigma^s_{ef}$ are the effective stress at yielding for bone-shaped and straight fibers, respectively; $\sigma^b_{y}$ and $\sigma^s_{y}$ are the yielding strength of the bone-shaped and straight short fiber composites, respectively. Taking $\sigma_m = 20.65$ MPa, it can be calculated using the average strength data from Table 1 that $\sigma^b_{ef} = 243.65$ MPa, and $\sigma^s_{ef} = 110.06$ MPa. It can be seen that the effective stress of bone-shaped fibers at yielding is more than twice that of the straight fibers, i.e., the bone-shaped short fibers are more than twice as effective as the straight short fibers in reinforcing the composites.

As mentioned previously, the bone-shaped short fibers show greater advantage over the straight short fibers when a weaker fiber-matrix interface is engineered. The fibers in the above composite samples were treated with n-OTS to obtain a weak fiber-matrix interface. The weak interface allows the fiber to debond in front of an approaching crack, which will effectively alleviate the stress concentration that may otherwise break the fiber. In the straight short-fiber composites, the load transfer is completely dependent on the interface. When a crack passes through a straight fiber weakly bonded to the matrix (shown in Figure 3(a)), the fiber may be totally debonded, and lose all the load it carried due to the failure of load transfer. Even without the crack passing through a straight fiber, void tends to form at the end of a straight fiber, and debonding will start at the ends and propagate toward the center at a stage much earlier than the composite failure. This will result in a very low effective fiber stress, $\sigma^s_{ef}$. In contrast, the bone-shaped short fiber does not depend upon the interface for load transfer, which is attained through the interlocking mechanism at the fiber ends. Consequently, the load carried in a bone-shaped short fiber is not affected by debonding (shown in Figure 3(b)), which results in a higher effective fiber stress, $\sigma^b_{ef}$.

SEM micrographs of fracture surfaces have shown that the fracture of the bone-shaped short fiber composite sample was originated from an area where there is a high concentration of ball ends. Small cracks initiated from several ball ends, and coalesced
later to form a larger unstable crack, which led to the failure of the composite. Several holes with the size of fiber diameter and broken fibers ends without balls indicate that the bone-shaped fibers were broken before being pulled out. Therefore, the bone-shaped short fibers effectively bridged the matrix cracks before they broke, which explains the high yielding strength and Young’s modulus of bone-shaped short fiber composites. However, the bone-shaped short fiber composites had a smaller final strain because the fibers were broken instead of being pulled out with the fiber. The balls at the fiber ends make it consume more energy to pull out a fiber, when the ball size is optimized to allow fiber pull out. This will increase both the strength and toughness of short fiber composites.

The fracture surface of a straight short-fiber composite sample clearly showed pulled-out fibers and holes. Due to the weak interface, the fibers were easily pulled out, which resulted in lower yielding strength and Young’s modulus. The straight short fiber composites had a larger final strain than the bone-shaped short fiber composites because the weak interface allows the straight fibers to be easily pulled out without damaging the matrix.

The reinforcement morphology has been found to significantly affect the mechanical properties of short fiber composites. The bone-shaped short fibers are much more effective in reinforcing the composite matrix due to more effective crack bridging, which is attested by the high yielding strength and Young’s modulus of bone-shaped short fiber composites. An optimized bone morphology, coupled with a weak interface, has the potential to significantly improve both the strength and fracture toughness of short fiber composites.
EXAMPLE 2

Commercial polyethylene (Micro Dyneema™) filament with a diameter of 181 μm was used as the precursor fiber. Bone-shaped short fibers were fabricated using a jig assembly and a mini-hydrogen torch. The jig assembly consisted of three matching multiple slotted metal plates. A continuous fiber was wrapped multiple times around one of the plates, which was subsequently sandwiched between the other two matching plates. Then a precision hydrogen flame was passed through the length of the slots thereby cutting the exposed filaments and leaving enlarged ends on each individual fiber.

Composite samples were fabricated with a matrix material of polyester and the polyethylene bone-shaped short fibers prepared as described above. Suspension of the polyethylene fibers in the uncured polyester during fabrication to obtain a spatially-random distribution of the polyethylene fibers was accomplished by addition of 0.39 g of amorphous fumed silica (Cab-O-Sil) to 10 ml of polyester as a thickening agent before mixing with the polyethylene fibers. The mixture of polyester and Cab-O-Sil was then placed through repeated cycles of vacuum/ambient pressure to remove any air bubbles introduced during mixing. A hardening agent of methyl ethyl ketone peroxide (0.6 percent by volume) and 0.5 g of polyethylene short fibers were then added. The new mixture was again placed through vacuum/ambient pressure cycles to remove any air bubbles. Finally, the mixture was extruded into a sample mold through a syringe. The sample mold produced a net-shaped sample for mechanical testing. The extrusion process aligns the polyethylene fibers to some extent. Further alignment was obtained using the principle of elongation flow, which was achieved by sliding two mold parts against one another, forcing the mixture to flow in the longitudinal direction of the sample. The sample mold was then mounted onto a slowly rotating machine for four hours to prevent the fibers from settling down to the mold bottom. Afterward, the samples were allowed to cure at room temperature for seven days before mechanical
testing. Excellent fiber alignment and random spatial-distribution were achieved using the above procedures.

Both bone-shaped short-fiber composite samples and straight short-fiber composite samples were fabricated using the above procedure. They all had a fiber length of either 3.6 mm or 4.9 mm and a fiber volume fraction of 5 percent. The only difference between the bone-shaped short-fiber composite samples and straight short-fiber composite samples was the fiber morphology, thus allowing examination of the effect of fiber morphology on the strength of composites. Fiber-free blank matrix samples were also fabricated for comparison.

The sample dimension is shown in Figure 1.

The stress-strain curves of both bone-shaped short-fiber composites and conventional straight short-fiber composites are shown in Figure 4. It can be seen that the strength of composite samples reinforced with the bone-shaped short fibers was significantly higher than that of composite samples reinforced with straight short fibers. It can also be seen that the Young’s modulus of the bone-shaped short fiber composite was higher than that of the straight short fiber composite. The determination of yielding strength as shown in Fig. 4 is according to the ASTM standard D 638M-91a, which is for tensile properties of plastics. Listed in Table 2 is the yielding strength for each sample tested. The average strength of the bone-shaped short fiber composite samples improved over that of conventional straight short fiber composite samples by 16.6 percent and 17.0 percent, respectively, for samples with fiber lengths of 3.6 mm and 4.9 mm.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Bone-shaped (MPa)</th>
<th>Conventional straight fibers (MPa)</th>
<th>Matrix (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Yielding Strengths of bone-shaped and conventional straight short-fiber composites (fiber length, l=3.6 or 4.9 mm) and the polyester matrix.
Listed in Table 3 are the Young's modulus of these samples. The Young's modulus was measured as the slope of the strain-stress curve in the strain range of 0.2 to 1.1 percent. Such a range was chosen because curves are linear within it. On average, the Young's modulus of the bone-shaped short-fiber composites is 18.4 percent higher than the conventional short-fiber composites for samples with fiber length of 3.6 mm and 12.1 percent higher for samples with fiber length of 4.9 mm.

Table 3. Young's modulus of bone-shaped and conventional straight short-fiber composites (fiber length, l=3.6 or 4.9 mm) and the polyester matrix.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Bone-shaped (MPa)</th>
<th>Conventional straight fibers (MPa)</th>
<th>Matrix (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>l = 3.6 mm</td>
<td>l = 4.9 mm</td>
<td>l = 3.6 mm</td>
</tr>
<tr>
<td>1</td>
<td>778</td>
<td>668</td>
<td>670</td>
</tr>
<tr>
<td>2</td>
<td>853</td>
<td>1002</td>
<td>819</td>
</tr>
<tr>
<td>Average</td>
<td>816</td>
<td>835</td>
<td>745</td>
</tr>
</tbody>
</table>

Note that the fiber content in these composite samples was only about 5 percent. The effective fiber stress at the composite yielding (σ_e) was calculated using the equations shown in Example 1 and from the average strength data in Table 2. The results are shown in Table 4.
Table 4. The effective fiber stress $\sigma_{ef}^b$ and $\sigma_{ef}^s$, for bone-shaped and conventional straight short-fiber composites

<table>
<thead>
<tr>
<th>Fiber length</th>
<th>$\sigma_{ef}^b$</th>
<th>$\sigma_{ef}^s$</th>
<th>$\sigma_{ef}^s / \sigma_{ef}^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 = 3.6 mm</td>
<td>79.4</td>
<td>30.8</td>
<td>2.6</td>
</tr>
<tr>
<td>1 = 4.9 mm</td>
<td>106.0</td>
<td>52.4</td>
<td>2.0</td>
</tr>
</tbody>
</table>

Toughness (energy consumed per unit volume of sample before failure) was calculated as the area under the stress-strain curve using the equation

$$\frac{\text{Energy}}{\text{Volume}} = \int_0^{\varepsilon_f} \sigma d\varepsilon$$

where $\varepsilon_f$ is the failure strain. Listed in Table 5 is the toughness of the tested samples.

Note that curve B2 in Fig 4, which is from a bone-shaped short-fiber composite, shows an unloading phenomenon before final failure because the sample didn’t break in the gauged section. Otherwise, curve B2 should look like curve B1, which is from the other bone-shaped short-fiber composite sample in Fig. 4. As a result, the toughness cannot be calculated from curve B2. It can be seen from Table IV that when the fiber length is 3.6 mm, the toughness of bone-shaped short-fiber composite is 25 percent higher that that of the conventional straight short fiber composite. However, when the fiber length is 4.9 mm, the toughness of bone-shaped short-fiber composite was 25 percent lower that that of the conventional straight short fiber composite. As discussed previously, if the fiber morphology can be optimized in such a way that much more energy would be consumed during the crack propagation due too difficult fiber pull-out, both strength and toughness of composite material will be significantly improved. This has been evidenced by the strain-stress curves of the bone-shaped short-fiber composite in which the fiber length is 3.6 mm. It is evident from curve B1 and B2 in Fig 4 that the bone-shaped short fibers were effectively bridging cracks before sample failure, preventing an abrupt failure as in conventional straight short fiber composite. Note that the toughness referred above is
different from the fracture toughness, which is a measure of material resistance to crack propagation. It is expected that the fracture toughness of bone-shaped short-fiber composite will be significantly higher than that of conventional straight short fiber composite because the bone-shaped short fibers can bridge cracks more effectively.

Table 5. Toughness of bone-shaped and conventional straight short-fiber composites and the polyester matrix.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Bone-shaped (KJ/m$^2$)</th>
<th>Conventional straight fibers (KJ/m$^2$)</th>
<th>Matrix (KJ/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$l = 3.6$ mm</td>
<td>$l = 4.9$ mm</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>2763</td>
<td>1737</td>
<td>2197</td>
</tr>
<tr>
<td>2</td>
<td>--</td>
<td>1741</td>
<td>--</td>
</tr>
<tr>
<td>Average</td>
<td>2763</td>
<td>1739</td>
<td>2197</td>
</tr>
</tbody>
</table>

SEM micrographs of fracture surfaces showed hackle marks radiating from fiber’s ball ends or craters where the ball ends were before composite failure. This indicated that cracks were initiated at the balls, radiated out and later coalesced, resulting in composite failure. Bone-shaped fibers extruding out of the fracture surface bridged the cracks before finally being pulled out. A closer examination revealed that the fiber ends had the shape of a disk with relatively sharp edges. During the tensile testing, these edges created tensile stress concentration in the matrix that leads to crack formation. A smaller, elliptical fiber end with its long axis parallel to the fiber longitudinal direction will help to relieve the stress concentration and consequently further improve composite toughness. The fracture surface of a conventional straight short fiber composite sample clearly showed pulled out fibers and holes. Due to the weak interface, the fibers were easily pulled out, which resulted in a lower yielding strength and Young’s modulus.
Another series of composite samples were fabricated with a matrix material of polyester and the polyethylene bone-shaped short fibers as above and the samples were tested for fracture toughness. In order to investigate the fracture toughness of the straight short fiber composites and the bone-shaped short-fiber composites and crack-bridging effectiveness of the bone-shaped short-fibers, double cantilever beam samples were fabricated and tested. Figure 7 shows the curves of normalized load, $P$, against crack length, $a$, for both bone-shaped short-fiber composites (solid marks) and straight short-fiber composites (open marks) with a fiber length of 3 mm. The normalized load was calculated as $P = P/w$, where $P$ is the measured load and $w$ is the crack width. The crack length, $a$, was measured in situ as the distance between the loading line and the crack tip (Fig. 1b). Fig. 7 demonstrates that higher load is required to propagate cracks in the bone-shaped short-fiber specimens than in the straight short-fiber specimens. This further proves that bone-shaped short-fibers bridge cracks more effectively than straight short-fibers.

The total normalized energy consumed for a crack to propagate from the initial crack length $a_0$ to crack length $a$ can be calculated as

$$E(a) = \int_0^{a_0} P(v) dv$$

where $P(v)$ is the normalized load as a function of crack opening displacement, $v(a)$, along the loading line. Each measured crack length, $a$, corresponds to a displacement, $v(a)$. Shown in Fig. 8a are the total normalized energies, $E(a)$, calculated by the above equation, as a function of crack length for double cantilever beam tests of both bone-shaped short-fiber and straight short-fiber composites. As shown, $E(a)$ is higher for bone-shaped short-fiber composites than for straight short-fiber composites.
The supplied energy for a crack to propagate by a unit length can be calculated by
\[ \sigma(a) = \frac{dE(a)}{da} \]
This can be done by first fitting the curves in Fig. 8a of \( E(a) \) versus \( a \) with a polynomial function and then obtaining its derivative. As shown in Fig. 8a, \( E(a) \) values from an individual double cantilever beam testing is not a smooth function of crack length and vary substantially from specimen to specimen, as a result of the random distribution of fibers bridging the crack. Therefore, for fitting purposes, we combined all three data sets of \( E(a) \) for each type, bone-shaped short-fiber and straight short-fiber composites, to fit them to a third order polynomial function (see Fig. 8a).

Using the above equation, \( \sigma(a) \) was calculated from this polynomial function and is shown in Fig. 8b. For both types of double cantilever beam specimens, \( \sigma(a) \) is an increasing function of \( a \). Also note that \( \sigma(a) \) includes energies consumed both by crack propagation and by further deformation in the two beams of the double cantilever beam specimen, which makes it larger than the crack resistance, \( R \), or the energy consumption in the formation of a unit length of crack. As shown, the bone-shaped short-fiber double cantilever beam specimens require significantly more energy for crack propagation than the straight short-fiber double cantilever beam specimens. Since the fibers did not fracture, this enhanced crack resistance, or indirectly, higher fracture toughness of the bone-shaped short-fiber composites, is due solely to their enhanced ability to bridge matrix cracks and to resist pull-out.

**EXAMPLE 3**

Three categories of samples, blank concrete beams, straight steel fiber (wire) reinforced concrete beams and bone-head steel fiber (wire) reinforced concrete beams were prepared for 4-point bending tests, intended to identify and compare the roles played
by discrete straight wires and bone-head wires on strengthening concrete. The sizes of the samples are approximately 9.5" x 1.5" x 1.0". The diameter of the short fibers (wires) was about 0.82 mm and the length of the short fibers (wires) was about 2.5 cm. Concrete matrix was prepared by blending 210 ml water and 600 g Portland cement until a uniform mixture was obtained. This water/concrete ratio was determined by the fact that in this ratio the viscosity of the concrete matrix is moderate, rendering optimal attributes to the concrete in limiting the size and amount of bubbles in the sample and supporting the wires from sinking as well. The steel-wire content was about 1 volume percent for the bone-shaped wires and about 0.85 volume percent for the straight shaped wires.

In the case of blank concrete samples, concrete matrix was directly added to the mould, into which the concrete filled automatically due to its fluidity. Appropriate shaking of the mould was followed to reduce trapped air bubbles. In the cases of wire reinforced composite samples, a similar approach was adapted. However, four layers of wires were incorporated into the concrete samples, equally dividing the thickness. The incorporation of wires was achieved by hand lay-up of steel wires in concrete.

The curing of the samples was carried out initially in plastic bags for 12 hours, and then in air covered with wet clothes for a few days prior to testing.

Testing yielded the results shown in Table 6 and Figure 6.
Table 6: Bending Test Results for Concrete Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>S1 (MPa)</th>
<th>Su (MPa)</th>
<th>Disp (0.8)</th>
<th>D (0.8)</th>
<th>A and A_{5\text{mm}}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>1.12</td>
<td>1.12</td>
<td>0.12 mm</td>
<td>0.16 mm</td>
<td>0.02135</td>
</tr>
<tr>
<td>Blank</td>
<td>1.34</td>
<td>1.34</td>
<td>0.19 mm</td>
<td>0.26 mm</td>
<td>0.03755</td>
</tr>
<tr>
<td>With straight fibers</td>
<td>3.53</td>
<td>3.53</td>
<td>1.92 mm</td>
<td>2.65 mm</td>
<td>2.6110</td>
</tr>
<tr>
<td>With bone-shaped fibers</td>
<td>6.18</td>
<td>6.18</td>
<td>1.75 mm</td>
<td>2.41 mm</td>
<td>3.4769</td>
</tr>
<tr>
<td>With bone-shaped fibers</td>
<td>6.79</td>
<td>7.58</td>
<td>4.50 mm</td>
<td>6.19 mm</td>
<td>7.5974</td>
</tr>
</tbody>
</table>

S1: flexural strength at first crack (at the onset of nonlinearity)

Su: maximum flexural strength (at the maximum load)

Disp (0.8): cross-head displacement at the point where the load drops to 90 percent maximum load (N*M=j)

D(0.8): deflection at the center of the beam at the point where the load drops to 90 percent maximum load, (D(0.8)=Disp.(0.8) x 11/8

A: area under the curve for concrete blank

A_{5\text{mm}}: area under the curve up to 5 mm cross-head displacement


Although the present invention has been described with reference to specific details, it is not intended that such details should be regarded as limitations upon the scope of the invention, except as and to the extent that they are included in the accompanying claims.
WHAT IS CLAIMED IS:

1. A short fiber-reinforced composite comprising:
   a matrix material selected from the group consisting of metals, ceramics, polymers, and concretes; and,
   a minor percentage of non-metallic short fibers having a fiber morphology wherein said short fibers have one or more fiber regions of a greater cross-sectional diameter than a predominant cross-sectional diameter of said short fibers at majority portions of said short fibers, said cross-sectional diameter measured perpendicular across a main axis of said short fibers.

2. The short fiber-reinforced composite of claim 1 wherein said short fibers have a fiber morphology wherein said short fibers include said greater cross-sectional diameters at ends of said short fibers.

3. The short fiber-reinforced composite of claim 1 wherein said short-fibers are of a material selected from the group consisting of glass, polyesters, polyamides, polycryllics, polyolefins, cellulose, cellulose derivatives, alumina, aluminum nitride, aluminum silicate, boron carbide, carbon, graphite, magnesium oxide, mullite, silicon carbide, silicon dioxide, silicon nitride, titanium nitride, and zirconium dioxide.

4. The short fiber-reinforced composite of claim 1 wherein said short fibers are substantially aligned parallel within said short-fiber reinforced composite.

5. The short fiber-reinforced composite of claim 1 wherein said minor percentage of short fibers is up to about 30 percent by volume.

6. The short fiber-reinforced composite of claim 1 wherein said short fibers have an average fiber length of from about 0.1 mm to about 10 cm.

7. The short fiber-reinforced composite of claim 1 wherein said matrix material is selected from the group consisting of metals, ceramics, and polymers.
8. A short fiber of a material selected from the group consisting of glass, polyesters, polyamides, polyacrylics, polyolefins, cellulose, cellulose derivatives, alumina, aluminum nitride, aluminum silicate, boron carbide, carbon, graphite, magnesium oxide, mullite, silicon carbide, silicon dioxide, silicon nitride, titanium nitride, zirconium dioxide, aluminum, iron, nickel, niobium, titanium and tungsten, said short fiber characterized as having a fiber morphology wherein said short fibers have one or more fiber regions of a greater cross-sectional diameter than a predominant cross-sectional diameter of said short fibers at majority portions of said short fibers, said cross-sectional diameter measured perpendicular across a main axis of said short fibers.

9. The short fiber of claim 8 wherein said short fiber has a fiber length of from about 0.1 mm to about 10 cm.

10. The short fiber of claim 8 wherein said short fibers include said greater cross-sectional diameters at ends of said short fiber.

11. A short fiber-reinforced composite comprising:

a matrix material selected from the group consisting of ceramics, polymers, and concretes; and,

a minor percentage of short fibers having a fiber morphology wherein said short fibers have one or more fiber regions of a greater cross-sectional diameter than a predominant cross-sectional diameter of said short fibers at majority portions of said short fibers, said cross-sectional diameter measured perpendicular across a main axis of said short fibers.

12. The short fiber-reinforced composite of claim 11 wherein said short fibers have a fiber morphology wherein said short fibers include said greater cross-sectional diameters at ends of said short fibers.

13. The short fiber-reinforced composite of claim 12 wherein said short-fibers are of a material selected from the group consisting of glass, polyesters, polyamides, polyacrylics, polyolefins, cellulose, cellulose derivatives, alumina, aluminum nitride,
aluminum silicate, boron carbide, carbon, graphite, magnesium oxide, mullite, silicon carbide, silicon dioxide, silicon nitride, titanium nitride, zirconium dioxide, aluminum, iron, nickel, niobium, titanium and tungsten.

14. The short fiber-reinforced composite of claim 12 wherein said minor percentage of short fibers is up to about 30 percent by volume.

15. The short fiber-reinforced composite of claim 12 wherein said short fibers have an average fiber length of from about 0.1 mm to about 10 cm.

16. The short fiber-reinforced composite of claim 11 wherein said matrix material is selected from the group consisting of ceramics and polymers.
**Fig. 1** The dimension of composite sample for tensile testing.

**Fig. 2** Stress-strain curves of bone-shaped and straight short Ni fiber composites and polyester matrix. 0.6% MEKP hardener was used to harden polyester matrix for all samples. Ni fiber length = 2.5 mm, diameter = 76.2 μm. Fiber volume fraction = 1.7%.
Fig. 3 A schematic drawing of crack bridging by (a) a straight fiber and (b) a bone-shaped fiber. For a weak interface, the straight fiber will debond, and lose the load carried in it. However, the load transfer is not affected for the bone-shaped fiber, which enables effective crack bridging.

Fig. 4 Strain-stress curves of polyester matrix and composites reinforced with bone-shaped and conventional short fibers.
FIG. 6

![Graph showing load (N) vs. displacement (mm) for three types of concrete: BSS-fiber reinforced concrete, CSS-fiber reinforced concrete, and unreinforced concrete.](image-url)
FIG. 7
FIG. 8(a)

FIG. 8(b)
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER
   IPC(6) : D02G 3/00
   US CL : 428/359
   According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED
   Minimum documentation searched (classification system followed by classification symbols)
   U.S. : 428/359

   Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

   Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<tr>
<td>A</td>
<td>US 5,149,573 A (KOBE et al.) 22 September 1992, see the Figures.</td>
<td>1</td>
</tr>
<tr>
<td>A</td>
<td>US 4,454,183 A (WOLLMAN) 12 June 1984, see the Figures.</td>
<td>1</td>
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<tr>
<td>A</td>
<td>US 3,849,840 A (YAMADA et al.) 26 November 1974, see the figures.</td>
<td>1</td>
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</tbody>
</table>

Further documents are listed in the continuation of Box C. See patent family annex.

Date of the actual completion of the international search: 07 APRIL 1999
Date of mailing of the international search report: 05 MAY 1999

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