# INFLUENCE OF REINFORCEMENT MORPHOLOGY ON THE MECHANICAL PROPERTIES OF SHORT-FIBER COMPOSITES

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Abstract

A major problem of short-fiber composites is that the interfaces between the fiber and matrix become a limiting factor in improving mechanical properties such as strength. For a short fiber, a strong interface is desired to effectively transfer load from matrix to fiber, thus reducing the ineffective fiber length. However, a strong interface will make it difficult to relieve fiber stress concentration in front of an approaching crack. Stress concentrations result in fiber breakage. We report in this paper an innovative approach to overcome this problem: reinforcement morphology design. Short-fibers with enlarged ends are processed and used to reinforce a polyester matrix. The initial results show that the bone-shaped short-fibers produce a composite with significantly higher strength than can be attained with conventional short, straight fibers.

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## Introduction

These 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 the composites (1-4). For a short fiber, a strong interface is desired to effectively transfer load from matrix to 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 (5-9). 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 (10, 11). 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 an interface may still cause successive adjacent fiber breakage and subsequently reduce composite toughness (1, 11). On the other hand, a weak interface significantly decreases the fiber load-carrying length due to ineffective load transfer and *complete* fiber interfacial debonding and pullout during loading. A compromise in the interfacial bond strength will also compromise the load-carrying potential of short fibers.

The aforementioned problems are intrinsic with the conventional short straight-fiber composites, and cannot be solved by modifying the fiber/matrix interfacial property. 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. This can be achieved by modifying the morphology of short fibers. For example, a bone-shaped short fiber with two enlarged ends can effectively transfer load from matrix onto the 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 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 is transferred through mechanical interlocking between the enlarged fiber ends and the matrix, the load-carrying potential of short fibers can be better utilized. These advantages should translate into significantly higher strength for this class of bone-shaped short-fiber composites having weak-to-moderate interfaces.

To prove the above innovative concept, we have initiated a project to make a prototype bone-shaped short-fiber composite. The initial results show that the bone-shaped short-fiber composite has a significantly higher strength than the conventional short straight fiber composite.

## Experimental Procedure

### Fabrication of fibers

Commercially pure Ni 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 designed in our laboratory. The machine automatically feeds and swings the Ni filament through a flame from a mini hydrogen torch. The flame cuts the Ni filament by melting it, and the melted Ni forms two balls on the two cut ends for each cutting due to surface tension of the melted Ni. The average ball size is determined by the size of the torch. The bone-shaped Ni fibers used in this study have an average ball diameter of 183 μm and a length of 2.5 mm. The short straight Ni fibers with a length of 2.5 mm were obtained by cutting the Ni filament using scissors.

## Fabrication of Composite Samples

After the Ni bone-shaped and straight fibers were fabricated, they were coated with a bond-weakening agent named n-octadecyltrichlorosilane (n-OTS). The coating procedure was as follows: The Ni fibers were washed in a 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% n-OTS and put into an oven at 50°C for 60 minutes. The final step of the fiber coating process was to pour out the ethanol/n-OTS solution and place the fibers back into the oven at 100°C for 120 minutes to bake on the n- coating.

Polyester was chosen as matrix material. To suspend the Ni fibers in the uncured polyester during the fabrication so as to obtain a spatially-random distribution of Ni fibers, 0.39 g of amorphous fumed silica (Cab-O-Sil, Cabot Corporation, Tuscola, Illinoise) was added to 10 ml of polyester as a thickening agent before mixing with Ni fibers. The mixture of polyester and Cab-O-Sil was then placed through repeated cycles of vacuum/ambient pressure to remove the air bubbles introduced during the mixing. 0.6 % volume of Methyl Ethyl Ketone Peroxide (MERK hardener) and 1.5 g of Ni fiber was then added. The new mixture was again placed through vacuum/ambient pressure cycles to remove air bubbles. Finally, the mixture was extruded into a sample mold through a syringe. The sample mold produces a net-shaped sample for mechanical testing. The extrusion process aligns Ni fibers to some extent. Further alignment was obtained using the principle of elongation flow, which was achieved by sliding two mold parts against each other, 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 fibers from settling down to mold bottom. Afterward, the samples were allowed to cure at room temperature for seven days before mechanical testing. Excellent fiber alignment and random fiber spatial-distribution were achieved using the above procedure.

Both bone-shaped short-fiber composite samples and straight short-fiber composite samples were fabricated using the above procedure. They all have a fiber length of 2.5 mm and fiber volume fraction of 1.7%. The only difference between the bone-shaped short-fiber composite samples and the straight short-fiber composite samples is the fiber morphology, thus allowing us to examine the effect of fiber morphology on strength of composites. Fiber-free blank matrix samples were also fabricated for comparison.

## Characterization

The sample dimension is shown in Fig. 1. Tensile testing was performed using a Model 1125 Instron testing machine. An extensometer was used to measure 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.

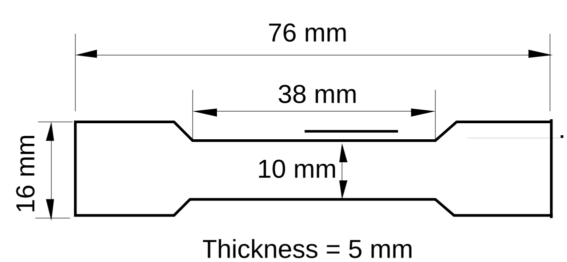


Fig. 1. The dimension of composite sample for tensile testing

## Results and Discussion

The stress-strain curves of both bone-shaped short-fiber composites and conventional straight short-fiber composites are shown in Fig. 2. It is obvious that the strength of composite samples reinforced with bone-shaped short fibers are 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 is higher than that of straight short fiber composite. 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 I are the yielding strength of each sample tested. The average strength of the bone-shaped short fiber composite samples improve by 10.2% over that of conventional straight short-fiber composite samples.

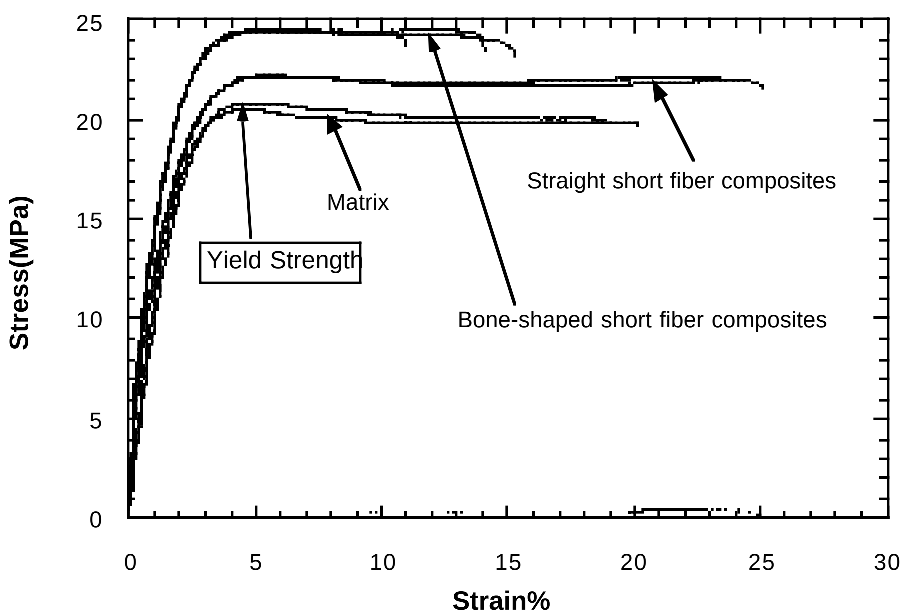


Fig. 2 Stress-strain curves of bone-shaped and straight short Ni fiber composites and polyester matrix. 0.6% MEKP hardener was used to harden polyester matrix for all samples. Ni fiber length = 2.5 mm, diameter = 76.2 µm. Fiber volume fraction = 1.7%.

Table I. The yielding strengths of the bone-shaped and straight short fiber composites and the polyester matrix.

| Sample # | Bone-shaped (MPa) | Straight-fiber (MPa) | Matrix (MPa) |
|---|---|---|---|
| 1 | 24.33 | 22.13 | 20.45 |
| 2 | 24.48 | 22.21 | 20.84 |
| 3 | 24.49 | ___ | ___ |

| | | | |
|---|---|---|---|
| Average | 24.43 | 22.17 | 20.65 |

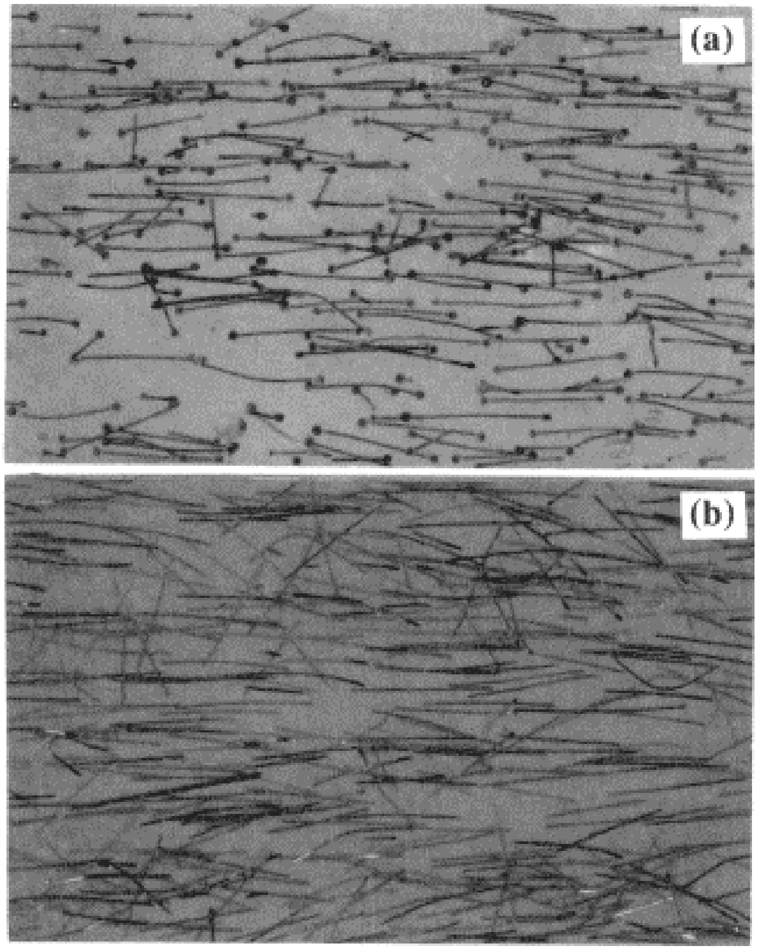


Fig. 3. Short fibers are well aligned in (a) a bone-shaped and (b) straight short fiber composite samples.

Note that the fiber contents in both the bone-shaped short-fiber composites and straight short-fiber composites are only 1.7%. The 10.2% improvement in the yielding strength of bone-shaped short-fiber composites with a 1.7% fiber volume fraction indicates that the bone-shaped short fibers is much more effective in strengthening the polyester matrix than the straight short fibers. Figure 3 shows that the short fibers are well aligned in both the bone-shaped and the straight short-fiber composites, and therefore the composite samples can be considered as reinforced by unidirectional fibers. Using a simple rule of mixture, the composite strength can be described as

$$\sigma_c = V_f \sigma_{ef} + \left(1 - V_f\right)\sigma_m \tag{1}$$

where $\sigma_c$ is the yielding strength of composites, $V_f$ is the fiber volume fraction, $\sigma_m$ is the average yielding stress in matrix, $\sigma_{ef}$ is the effective fiber stress at the composite yielding. Note that the stress distribution along the fiber length is not uniform (12). 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 its enlarged ends. The effective stress for the bone-shaped and straight short fibers at composite yielding can be calculated from Eq. 1 as

$$\sigma_{ef}^{b} = \frac{\sigma_c^b - \left(1 - V_f\right)\sigma_m}{V_f} \tag{2}$$

and

$$\sigma_{ef}^{s} = \frac{\sigma_c^s - \left(1 - V_f\right)\sigma_m}{V_f} \tag{3}$$

where $\sigma_{ef}^{b}$ and $\sigma_{ef}^{s}$ are the effective stress at yielding for bone-shaped and straight fibers, respectively; $\sigma_c^b$ and $\sigma_c^s$ 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 I that $\sigma_{ef}^{b}$ = 243.65 MPa, and $\sigma_{ef}^{s}$ = 110.06 MPa. It is obvious that the effective stress of bone-shaped fibers at yielding is more than twice of that of the straight fibers, i.e. the bone-shaped short fibers is more than twice as effective as the straight short fibers in reinforcing the composites.

As mentioned in the introduction, 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 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

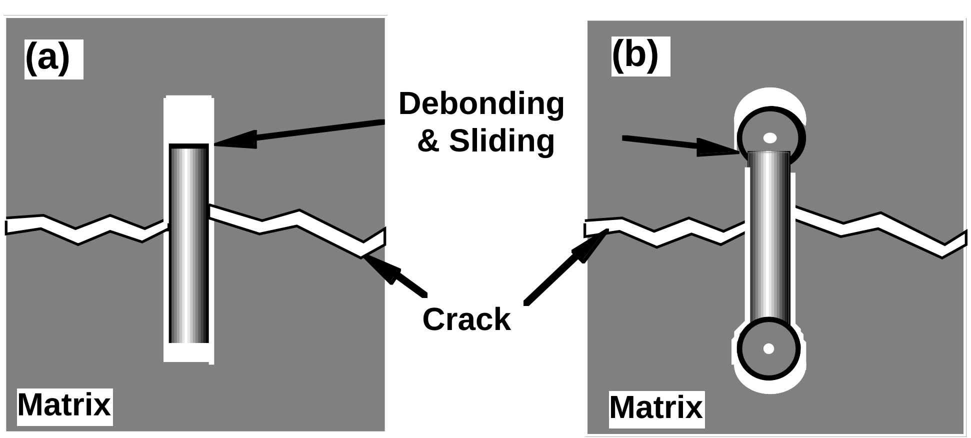


Fig. 4 A schematic drawing of crack bridging by (a) a straight fibers and (b) a bone-shaped fiber. For a weak interface, the straight fiber will debond, and lose the load carried in it. However, the load transfer is not affected for the bone-shaped fiber, which enables effective crack bridging.

crack pass through a straight fiber weakly bonded to the matrix (Fig. 4a), 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 [13, 14]. This will result in a very low effective fiber stress, $\sigma_{ef}^{s}$. In contrast, the bone-shaped short fiber does not depend on 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 (Fig. 4b), which results in a high effective fiber stress, $\sigma_{ef}^{\upsilon}$.

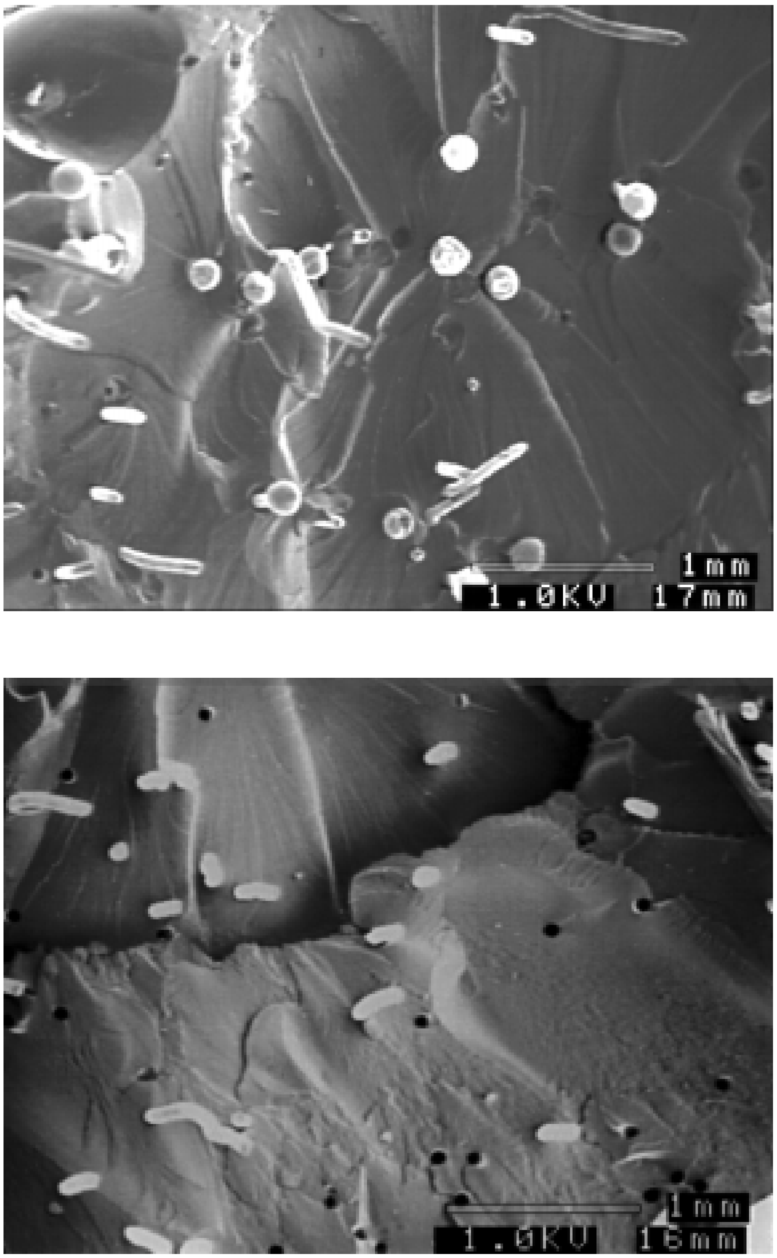


Fig. 5. Fracture surface of a) bone-shaped fiber reinforced composites and b) straight short fiber composites.

SEM micrographs of fracture surfaces are shown in Fig. 5. It can be seen from Fig. 5a 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 has a smaller final strain because the fiber were broken instead of being pulled out with the balls. If the balls are made smaller, it might be possible to allow the balls to be pulled out with the fiber. The balls at the fiber ends will make it consume more energy to pull out a fiber, which will increase both the strength and toughness of short fiber composites.

The fracture surface of a straight short-fiber composite sample is shown in Fig. 3b, which clearly shows the pulled-out fibers and holes. Due to the weak interface, the fibers were easily pulled out, which result in a lower yielding strength and Young's modulus. The straight short fiber composites has a larger final strain than the bone shaped short fiber composites because the weak interface allows straight fibers to be easily pulled out without damaging the matrix.

## Conclusions

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.

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## References

1. A. Crasto, S. H. Own, and R. V. Subramanian, Composite Interfaces, H. Ishida and J. L. Koenig, eds., (New York, NY: Elsevier Science Publishers, 1986), 133.

2. M. Fishkis, J. R. Yeh, and K. Wefers, J. Materials Science, 29 (1994) 110.

3. E. A. Feest, Composites, 25 (1994), 75.

4. S. M. Kunz, K. Chia, C. H. McMurtry, and W. D. G. Boecker, Composite Applications, T. J. Drozda, ed., (Dearborn, Michigan: Society of manufacture Engineers, 1989), 38.

5. C. M. Friend, J. Materials Science, 22 (1987) 3005.

6. C. M. Friend, Scripta Metall., 23 (1989) 33.

7. W. H. Bowyer and M. G. Bader, J. Materials Science, 7 (1972) 1315.

8. K. Xia and T. G. Langdon, J. Materials Science, 29 (1994) 5219.

9. Y. T. Zhu, G. Zong, A. Manthiram, and E. Eliezer, J. Materials Science, 29 (1994) 6281.

10. E. Yasuda, T. Akatsu, Y. Tanabe, and Y. Matsuo, Composite Materials, A. T. Benedetto, L. Nicolais, and R. Watanabe, eds., (New York, NY: Elsevier Science Publishers, 1992) 167.

11. T. Norita, J. Matsui, and H. S. Matsuda, Composite Interfaces, H. Ishida and J. L. Koenig, eds., (New York, NY: Elsevier Science Publishers, 1986) 123.

12. Y. T. Zhu, W. R. Blumenthal, and T. C. Lowe, J. Materials Science, 32 (1997) 2037-43.

13. N. Sato, T. Kurauchi, S. Sato, and O. Kamigaito, J. Materials Science, 26 (1991) 3891.

14. S. R. Nutt and J. M. Duva, Scripta Metall., 20 (1986) 1055.