Bond Properties of Carbon Fibers in Cementitious Matrix

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Abstract: The interfacial bond properties of two carbon fibers, having diameters of 10 μm and 46 μm, were tested for cement matrices of different water-to-binder (*w/b*) ratios and silica-fume contents. The pullout test was conducted using a special technique that prevented the brittle fiber from breaking during specimen handling. An environmental scanning electron microscope (ESEM) was used to determine the nature of the fiber-matrix bond. The results show a friction-based bond mechanism for the fiber of 10 μm, with a bond strength of 0.5 MPa for the high *w/b* matrix and without silica fume. Densifying the matrix by lowering the *w/b* ratio or by using silica fume improved the bond by 50–100%. For the larger-diameter fiber, silica fume had a very strong effect on the bond (an increase of 370–670%). It appears that long grooves along the fiber surface creates a mechanical anchorage, which is strongly affected by the presence of silica fume.

INTRODUCTION

Fiber-matrix interfacial properties are important in controlling macroscopic properties of composite materials. Generally speaking, the better the bond, the better the composite. Bentur and Mindess (1990) and Li et al. (1991) showed that increasing the bond leads to increase in both composite strength and toughness. However, beyond a certain bond strength, the toughness begins to decrease due to fiber rupture. In addition, recent works of Li (1992, 1993) and coworkers showed that the phenomenon of pseudo-strain-hardening is strongly influenced by the maximum bridging stress, which is directly proportional to the interface bond strength.

Most composite models are based on the assumption of a constant frictional bond between the fiber and the matrix, independent of slip. This implies a linear decaying line for the postpeak pullout load-versus-displacement (*P-u*) relationship. However, both slip-weakening behavior in pullout of steel fiber (Naaman et al. 1976) and slip-hardening behavior in pullout of nylon and polypropylene fiber (Wang et al. 1988a) from cement matrices have been observed. These various behaviors are shown schematically in Fig. 1. The prepeak *P-u* relationship typically shows a nonlinear behavior due to the debonding process with increasing load, regardless of slip dependencies.

Determining the direct pullout behavior of a single carbon fiber is a complicated task. Most carbon fibers have small diameter, typically between 6–15 μm. In addition, the strain capacity of carbon fiber is low, typically less than 2% [compare to 3–4% for steel and up to 20% for polypropylene (Ramachandran et al. 1982)]. A technique for interface bond determination was developed by Wang et al. (1988b) for fine flexible fibers, based on measuring the critical fiber length, the longest embedment length that enables fiber pullout rather than fiber rupture. This method was adopted by Larson et al. (1990) for carbon fibers.

In this work, both the bond properties and the complete *P-u* curve of carbon fibers were studied for different matrix compositions and fiber types, using a direct pullout procedure recently developed by Katz and Li (in press, 1995). This technique overcomes the fiber brittleness and alignment problems, and generates more complete information on the interface properties than the critical-fiber-length test procedure.

EXPERIMENTAL PROGRAM

Two carbon fiber types were tested in the experimental program: Fiber A, having a small diameter of d = 10 μm, and fiber B, having a large diameter of d = 46 μm. Fiber A is a commercially available fiber and fiber B is an experimental fiber prepared for bond property measurements. Fiber properties are presented in Table 1. Two water-binder (*w/b*) (cementitious materials) ratios were tested: low (*w/b* = 0.35) and high (*w/b* = 0.50). The effect of silica-fume, which improves the density of the matrix and possibly also the interface, was also studied in the experimental program. The composition of the different mixes is listed in Table 2. The two

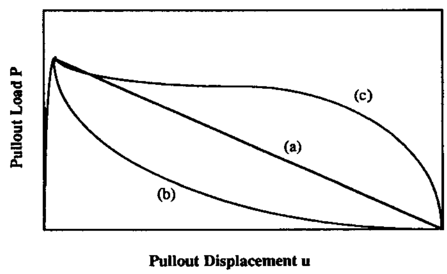

FIG. 1. Schematic Pullout Load-Versus-Displacement Curves from Single Fiber Pullout Test with: (a) Slip-Independent; (b) Slip-Weakening; and (c) Slip-Hardening Frictional Bond

TABLE 1. Properties of Carbon Fibers

(1)	Diameter (μm) (2)	Modulus of elasticity (GPa) (3)	Tensile strength (MPa) (4)	Strain capacity (%) (5)
Fiber A	10	240	1,970	0.8
Fiber B	46	175	930	0.5

TABLE 2. Matrix Composition

(1)	Mix 1 0.35 LD (2)	Mix 2 0.35 HD (3)	Mix 3 0.50 LD (4)	Mix 4 0.50 HD (5)
Portland cement type III	200 g	180 g	200 g	180 g
Water	70 g	46 g	100 g	76 g
Silica fume (slurry, 51% solids)	0 g	41 g	0 g	41 g
Superplasticizer	0 g	4 g	0 g	2 g
Water/binder	0.35	0.35	0.50	0.50
Silica fume/binder	0	0.10	0	0.10

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fiber types were tested for all matrices in Table 2 at the age of 14 days.

TEST PROCEDURE

The testing procedure used enables the preparation of specimens with high accuracy in fiber orientation and good control of the matrix around the fiber. By using this procedure, a specimen with five to 15 fibers is prepared, as shown in Fig. 2(a). The specimen is then sawn along the dashed line to provide a small specimen of a desire matrix thickness, L [Fig. 2(b)]. Using a sensitive load cell, a pullout test is then performed, yielding the complete P-u curve of the fiber. Details of this test setup are described in Katz and Li (in press, 1995).

The new setup was tested by the use of fine flexible polymeric fibers of known pullout behavior (Spectra 900; d = 42 μm). Test results showed that bond strength and the complete P-u curve are similar to data from literature (Li et al., in press, 1995), confirming the reliability of the new procedure.

Bond strength was calculated according to (1), assuming that bond mechanism is of friction, and that the fiber fully debonds prior to slippage and at the peak load P_{max}.

$$\tau = P_{max}/\pi d L \quad (1)$$

where P_{max} = maximum pullout load; d = fiber diameter; and L = fiber embedded length.

The fiber embedded length, L (between 5 and 6 mm), used in the tests was made as large as possible but still allowed a complete pullout of (most of) the fibers without rupture. The actual fiber diameter was measured under the microscope for better accuracy.

RESULTS AND COMMENT

Bond results of the pullout tests are presented in Table 3 and Fig. 3, the number in parentheses in the table is the coefficient of variation of the results. Test results are based on six specimens, but in particular cases, more specimens were tested as will be mentioned in the following. Because the results from the two fibers are basically different, the bond strength will be dealt with separately for the two fibers.

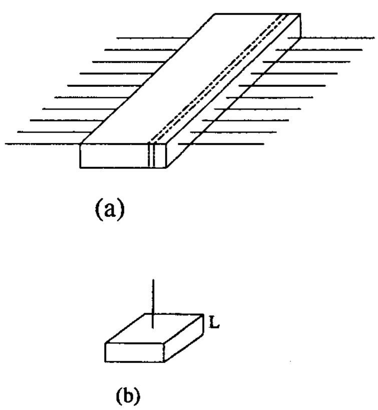

FIG. 2. Specimen: (a) After Demolding; (b) After Cutting

TABLE 3. Results of Bond Strength from Pullout Test

Cement mix (1)	Fiber A (2)	Fiber B (3)
0.35 LD	0.80 MPa (14%)	0.39 MPa (73%)
0.35 HD	1.29 MPa (14%)	>3.02 MPa (20%)
0.50 LD	0.52 MPa (31%)	0.52 MPa (49%)
0.50 HD	0.66 MPa (16%)	>2.44 MPa (23%)

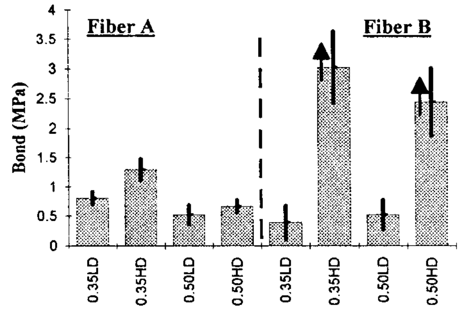

FIG. 3. Bond Strength of Fibers A and B, for Different Matrices

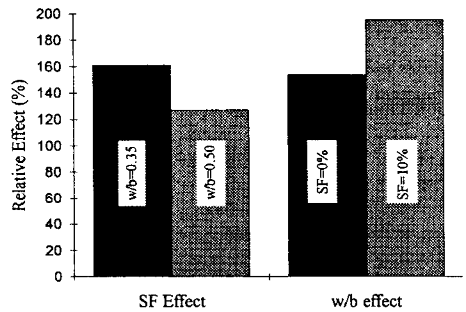

FIG. 4. Effect of Lowering w/b Ratio or Using Silica Fume for Fiber A

Fiber A

Bond values ranged from 0.52 MPa for the high w/b ratio matrix without silica fume to 1.29 MPa for the low w/b ratio matrix with addition of silica fume (Fig. 3). These values are somehow lower than the bond results of Larson et al. (1990). However, different carbon fibers are likely to have different fiber finish and sizing, which may affect the interfacial bond strength. The results here are similar to the conclusion from the work of Katz (1992) for carbon fibers of similar size.

When reducing the w/b ratio from 0.50 to 0.35 the bond strength was increased by 54% for composites of no silica fume and by 95% for the composites with 10% silica fume (see Fig. 4). Addition of silica fume leads to an increase of 27% and 61% for the high and low w/b ratios, respectively, on the bond strength as can be seen in Fig. 4.

These results indicate that densification of the matrix surrounding the fiber, either by reducing the w/b ratio or by using silica fume, has a positive effect on the bond strength. However, the use of silica fume appears to be more effective when used in low w/b ratio. Because the bond for this fiber is mainly of friction nature, densification of the matrix improves the fiber-matrix interfacial zone, thus leading to a better bond.

Fiber B

The wide range of bond strength as shown in Fig. 3 indicates a more complex mechanism of bond of this fiber. Matrices of plain cement exhibit low bond strength of ~0.4–0.5 MPa with large coefficient of variation (50–60%). The matrix of the low w/b ratio showed slightly lower bond, which could not be identified clearly due to the high coefficient of variation. The effect of lowering the w/b ratio was not very strong, ~25% for the matrices containing silica fume (Fig. 5), and it is slightly lower than the same effect for fiber A. Matrices containing silica fume showed much better bond strength of 2.44 and 3.02 MPa for the high and low w/b ratios, respectively. The strong effect of the use of silica fume relative to plain cement matrix can be seen in Fig. 5. Many fibers broke in the silica-fume matrices due to the high bond, even for

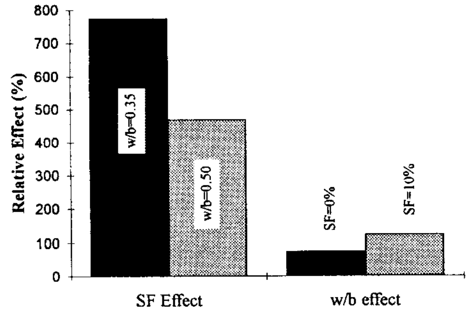

FIG. 5. Effect of Lowering w/b Ratio or Using Silica Fume for Fiber B

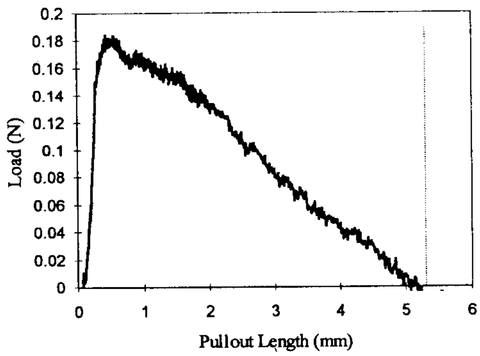

FIG. 6. Typical *P-u* Curves for Fiber A

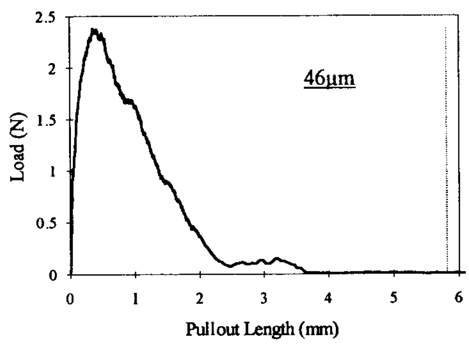

FIG. 7. Typical *P-u* Curves for Fiber B

very short embedded length. Therefore, the bond determined for these fibers is only a minimum value calculated based on the results of the fibers that successfully pulled out and those fibers that broke at the minimum embedded length. Higher bond values are possible, indicated by the arrows in Fig. 3. However, the low coefficient of variation (~20%) indicates that these values are close to the real ones which are presumably just slightly higher.

Typical load-displacement (*P-u*) curves for fiber A and fiber B are presented in Figs. 6 and 7, respectively. For fiber A it can be seen that the load reaches a peak after a short pullout displacement (which includes also setting of the system and stretching of the fiber) and drops almost linearly until a complete pullout was achieved. Complete fiber-embedment length is marked by a dashed line in the figures. This form of the *P-u* curve was observed for all the matrices, regardless of matrix composition. This linear decay of the pullout curve indicates a friction mechanism of bond that is slip-independent.

Close examination of the fiber surface by an environmental scanning electron microscope (ESEM) as shown in Fig. 8 shows very smooth surface clean from cement particles, indicating poor adhesion of the matrix to the fiber. It seems

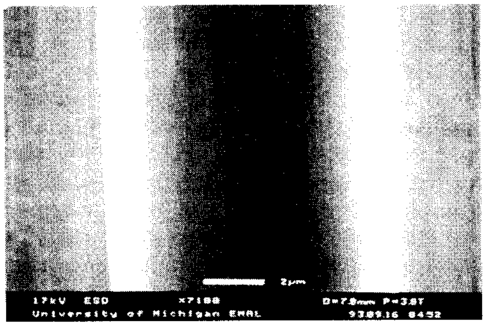

FIG. 8. ESEM Observation on Surface of Fiber A

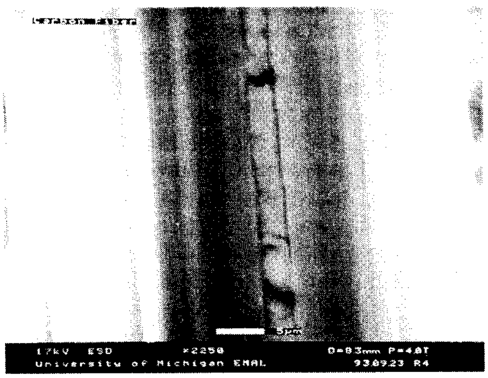

FIG. 9. ESEM Observation on Surface of Fiber B

that the small diameter of the fiber relative to the cement particles, accompanied by very smooth and hard fiber surface leads to a pure friction bond, which is kept constant during fiber pullout. No fiber surface damage is observed. When the matrix becomes denser, as a result of low w/b ratio or addition of silica fume, the contact between the fiber and the matrix is improved, leading to a better bond, as can be seen in the results in Table 3 and Fig. 3.

The pullout behavior of fiber B is very different from that of fiber A. The *P-u* curve of this fiber (Fig. 7) is characterized by a severe drop of the load after the peak, more likely of an elastic bond. However, for an elastic bond, immediate drop of the load is expected, accompanied by a linear reduction until the whole fiber is pulled out (Bartos 1980). For this fiber the *P-u* curve concave downward, indicating more-severe damage to the interface layer as the stiff fiber is pulled out. The interface might be completely deteriorated as the stiff fiber is pulled out through it, even for a short distance, leading to a rapid drop of the friction load between the fiber and the matrix.

Close examination of the fiber surface by ESEM (Fig. 9) reveals typical two to four long grooves along the fiber filled with matrix material. These grooves are relatively narrow, with width of a few micrometers (<4 μm) only. It seems that penetration of cement products into this groove plays an important role in the mechanical connection between the fiber and matrix. Good penetration of the matrix into these grooves can provide a good mechanical anchor between the fiber and the matrix. The drop of the pullout load after the peak, observed for this type of fiber, can be explained by breakage of the matrix penetrated into the grooves at the fiber surface, which leads to a severe reduction in the loads.

When using matrices of plain cement, with cement grain dimension comparable to or larger than groove width, the penetration of the matrix into the small grooves is poor, due to size effect and probably also due to the formation of a porous transition zone around the fiber, as know for larger steel fibers (Bentur et al. 1985; Wei et al. 1986) or aggregates

(Goldman and Bentur 1993). This leads to poor and inhomogeneous mechanical bond, as can be seen in Table 3 by the low bond and high coefficient of variation. (Due to the large variation in the results of the plain cement matrices, the experiment was repeated. However, similar results were observed. While other results in the table are based on at least six specimens, more than 20 specimens were considered for fiber B in plain cement matrix.) In contrast, the use of ultrafine particles such as silica fume (size <0.1 μm) is expected to cause densification of the transition zone as well as penetration into the grooves. These effects may explain the remarkable increase in bond strength when silica fume is used with fiber B, described earlier.

CONCLUSIONS

The interfacial properties of carbon fiber in a cement matrix can be characterized by a pullout test using a special specimen preparation procedure employed in the present study.

The densification of the matrix surrounding the fiber plays an important role in the interface bond strength and pullout behavior of carbon fiber. Densification may lead to enhanced contact between the fiber surface and adjacent transition zone material and therefore improved frictional strength.

For small-diameter and smooth-surface fiber, the bond between the fiber and the matrix is basically of a friction nature, and values of 0.5–1.3 MPa were observed. The higher bond value was achieved with densification of the matrix by silica fume combined with a low *w/b* ratio.

Long and narrow grooves along the surface of the large diameter carbon fibers may play an important role in its bond mechanism. It seems that penetration of the cement matrix into the grooves produces a mechanical anchorage that is very sensitive to its quality. When using plain cement matrix, the penetration of the matrix into the narrow grooves is limited, leading to a low bond value (0.4–0.5 MPa) with large data scatter. However, when silica fume was added to the matrix, a better penetration of the matrix into the groove was achieved, leading to a higher bond value of 2.4–3.0 MPa.

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