ELSEVIER

Effects of Transition Zone Densification On Fiber/Cement Paste Bond Strength Improvement

Yin-Wen Chan and Victor C. Li
Advanced Civil Engineering Materials Research Laboratory, Department of Civil and Environmental Engineering, University of Michigan, Ann Arbor, Michigan

This paper investigates the effectiveness of transition zone densification on the fiber-cement paste system. By controlling the water: cement (w:c) ratio and the condensed silica fume content, environmental scanning electron microscopy studies confirm that transition zone densification can be achieved in all brass, steel, and polyethylene fiber-cement systems. However, single fiber pullout tests indicate that densification only enhances the brass-cement paste interface bond strength and not the other systems. Further microscopy investigation of the surface of fibers peeled off from a composite fracture surface and of the groove left by the fiber on the cement paste suggests that bond failure for the brass-cement system is of a cohesive type, whereas bond failure for the other two systems is of an adhesive type. It is concluded that the transition zone densification technique should be effective in fiber-cement systems in which bond strength is controlled by cohesive failure of the transition zone material. © 1997 Elsevier Science Ltd. ADVANCED CEMENT BASED MATERIALS 1997; 5, 8–17

KEY WORDS: Adhesion, Cohesion, Densification, Fiber bond, Interface, Transition zone

Fiber reinforcement is an effective way of improving the strength, ductility, and toughness of brittle materials. The primary benefit of fiber reinforcement arises from the fiber-bridging effect across matrix cracks. The efficiency of fiber bridging depends on the material properties of the fiber, matrix, and the transition zone between fiber and matrix [1–3]. The latest concept on composite material design has proven that a composite material with specific desired mechanical properties can be produced by properly tailoring the associate material parameters [4].

Among the various material parameters that influence the composite properties, the interfacial bond between cement paste and matrix is the most important one. Due to the porous nature of the transition zone between fibers and matrix in cementitious composites, it is generally assumed that the transition zone represents a weak link among constituents governing composite properties. Thus, it is expected that densification of the transition zone may serve to strengthen the transition zone and enhance the fiber-cement interfacial bond strength [5,6]. Indeed, as a common microfiller in refining the cement paste microstructure, silica fume has been suggested to increase fiber-cement bond strength in cement matrix [7,8].

For some material systems, the transition zone microstructure may not be the governing factor in interfacial bond strength. For example, the adhesion between fibers and matrix, if it is too low, could be responsible for the bond failure. It has been reported by Hild and Schwartz [9] that, for fibers and/or matrix made up of materials with low surface energy, such as polymer, the adhesion between fiber and matrix may be the controlling parameter for fiber-matrix debonding. For such material systems, densification of the fiber-cement transition zone may not be effective in bond improvement. Instead, fiber surface modification would be necessary to enhance the adhesion between fibers and cement matrix.

The purpose of this study is to investigate the effect of transition zone densification on the improvement of the bond strength between fibers and cement matrix of various fiber reinforced cementitious composites. An attempt is made to densify the cement matrix and, hence, the pore structure of the fiber-matrix transition zone. This is achieved by controlling the packing density of cement paste matrix through the reduction of w:c ratio and the use of silica fume. Bond strengths are measured based on single fiber pullout tests. Along with different cement matrices, various types of fibers are tested, including steel, brass, and polyethylene fibers. In addition to bond measurement using fiber pullout tests, the microstructures of the transition zone of different material systems are observed using environ-

---

Address correspondence to: Victor C. Li, Department of Civil and Environmental Engineering, University of Michigan, 2326 G.G. Brown Building, 2350 Hayward, Ann Arbor, Michigan 48109.

Received April 28, 1995; Accepted July 11, 1996

655 Avenue of the Americas, New York, NY 10010

ISSN 1065-7355/97/$17.00
PII S1065-7355(96)00077-6

mental scanning electron microscopy. The purpose of investigating the interfacial microstructure is to provide evidence of densification by cementitious composition control and to infer the path of interfacial bond failure in peel-off tests of different fiber-matrix systems. The results of fiber pullout tests and the environmental scanning electron microscopy study are then combined to address the effectiveness of the transition zone densification on interface bond strength.

From the environmental scanning electron microscopy study, it is found that the compactness of the microstructure of the transition zone is well improved by reducing w:c ratio and/or using condensed silica fume, regardless of fiber type. However, improvement in fiber-cement bond strength does depend on fiber type. This indicates that transition zone densification does not always result in bond strength improvement. Moreover, the environmental scanning electron microscopy observations reveal that bond failure between steel or polyethylene fibers and cement matrix is of the adhesive type, that is, debonding takes place at the contact surface between fiber and cement. On the other hand, the brass-cement bond strength increases with the density of cement matrix and the corresponding fiber debonding takes place mainly in the heterogeneous transition zone. These findings indicate that the debonding of the brass-cement system is related to failure in the transition zone. This is most likely a result of the stronger adhesive strength between the cement paste and the brass fiber in comparison with the cohesive strength of the transition zone material. Therefore, the brass-cement bond strength, which depends on the strength of the transition zone, is sensitive to matrix packing density. In contrast, the steel-cement and polyethylene-cement bond strengths apparently are dominated by the adhesion property between steel or polyethylene fibers and the adjacent cement material and are negligibly affected by the microstructure of the transition zone. As a result, transition zone densification may not be effective for bond improvement in the case of steel and polyethylene fiber reinforced cementitious composites.

Based on the present study, it is proposed that transition zone densification is an effective means of fiber-cement paste bond strength improvement for material systems in which the transition zone is weak relative to the adhesive strength between fiber and paste material. In systems where the adhesive strength is weak, densification of the transition zone may not increase the bond strength unless the adhesive strength is also improved in the same process of material modification. However, it should be noted that the slip-weakening effect observed in metal fiber-cement paste systems, which relates to damage of the cement paste material during fiber sliding, does appear to be alleviated by the densification of the transition zone.

Interfacial Microstructure

The bond strength of a cementitious composite is assumed to be strongly affected by the fiber-matrix interfacial zone. This transition zone consists of a phase distinct from the fiber and the bulk cement matrix [10–13]. Formation of the transition zone is usually attributed to local bleeding around fibers and the consequence of the "wall" effect [14,15]. The wall effect results from the inability of the cement grains, having an average particle size of typically 10 μm, to fill the space 10 to 20 μm from the fiber surface as densely as they do in the bulk matrix away from the fiber surface. Apart from the looser packing of matrix particles, the fiber surface provides more spacious nucleation sites for CH crystals and causes changes in the composition of the hydration products. As a result, a unique microstructure forms in the annular region of fibers much different from the bulk matrix in both porosity and composition of hydration product. Compared to the bulk matrix, the transition zone is more porous and is rich in CH crystals. The porosity and the density vary along the distance from the fiber surface.

Mechanical Properties of Transition Zone

The mechanical properties of the transition zone have been studied through measurement of microhardness of the cement materials along radial lines in the annular region around the fibers [16]. The result indicates that the microhardness of the matrix far from fiber surface is nearly constant. As it gets closer to the fiber surface, within a distance of typically 50 μm, the microhardness starts to decrease revealing a looser material structure. However, instead of monotonically decreasing, the microhardness rises when it gets close to the fiber surface. This is probably due to the abundant deposit of CH crystals near the fiber surface. A conceptual definition of the interfacial transition zone is given in Figure 1 based on a microhardness measurement. In general, microhardness can be related to microstructure as well as mechanical properties of materials such as tensile strength [17–19]. For cement paste, porosity and strength have been correlated [20]. Low microhardness usually suggests high porosity and low strength. The spatial variation in the microhardness measurement described earlier implies the existence of a weak point in the transition zone at the location of the lowest hardness. This weak point can be responsible for fiber-

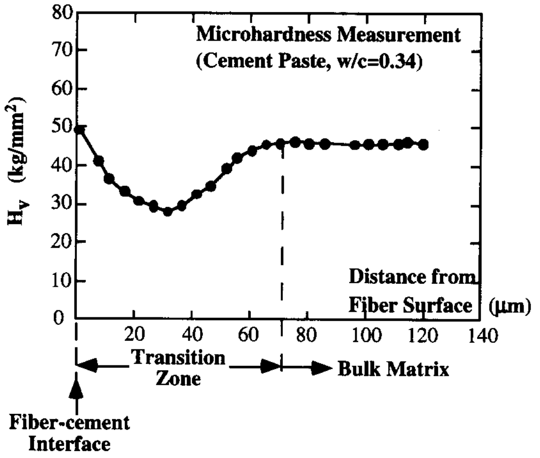


FIGURE 1. Schematic illustration of interfacial zone and microhardness measurement (after Wei, et al. [16]).

cement bond failure when debonding occurs in the transition zone.

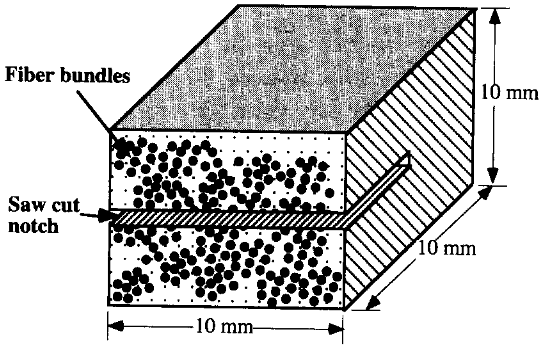
In summary, a less compact material structure of hydration products forms in the transition zone due to the increase in nucleation sites for CH crystals as well as the bleeding and wall effects. According to microstructure observations and the microhardness measurements, the interfacial zone is a weak phase in the composite structure and may dominate the failure mechanism of fiber debonding. Therefore, it is generally expected that densification in matrix microstructure may strengthen this transition zone and increase the interfacial bond strength. This hypothesis is tested by an experimental program of single fiber pullout tests and environmental scanning electron microscopy observations of fiber-matrix interface microstructures.

Test Program

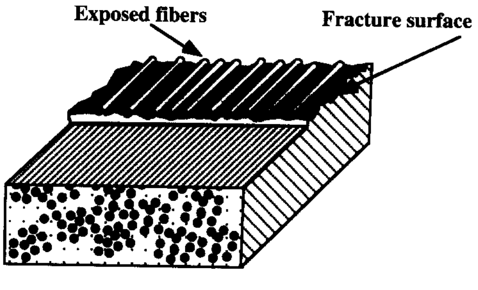
Conventional single fiber pullout tests were conducted in the test program to measure the bond strengths of various material systems. Steel, brass (both of 1.02-mm diameter), and two types of polyethylene fibers (Spectra, 38-µm diameter, and Snia, 20-µm diameter) were included in the experimental program. They were used in the "as received" condition. To alter the microstructure of the transition zone, three different types of cement matrices with various w:c ratios and silica fume (SF) contents were adopted, including plain cement paste with w:c = 0.40 (matrix I), plain cement paste with w:c = 0.27 (matrix II), and cement paste with silica fume (w:c = 0.27, SF:c = 0.20, and SP:c = 0.05, matrix III). In the case of polyethylene fiber, only matrices I and III were used. The fibers were arranged in the mold with an embedment length of 20 mm and a protruded portion to be held during the pullout process. The fiber pullout tests were conducted at the age of 28 days. At least three to six specimens were tested for each case. Pullout load and the corresponding fiber slip distance, the $P - u$ curves, were recorded for each specimen. Details of the fiber pullout experimental technique and data reduction procedure can be found in [21].

Microstructure Studies

Morphologies of microstructures of the transition zone material as well as the debonded surfaces on both matrix side and fiber side were observed by ElectroScan environmental scanning electron microscopy. Specimens for microstructure observation were prepared from fiber bundles cast in various cement matrices (Figure 2a). A saw cut was generated in the mid-surface of this specimen to create a notch. A fracture surface was created by extending a crack from the notch tip (Figure 2b). The fracture surface exposed various situations of fibers along the bundles, including partially exposed



(a) Specimen configuration



(b) After separation from extending notch

FIGURE 2. Preparation of the specimen for microstructure observation.

fiber surface, groove surface with fibers lifted away, and transition zone at the contact region of fiber to cement matrix. These features are desired for microstructure observation. A schematic of material samples prepared following these procedures for observation is shown in Figure 3. Specimens for microstructure studies are prepared this way instead of using debonded surfaces from fiber pullout tests, because interfacial features are likely to be destroyed during the pullout process.

The environmental scanning electron microscopy observations were conducted at the same age, 28 days, as the fiber pullout tests.

Results and Discussions

Transition Zone Densification

From the microstructure observations, we observed substantial differences, among the microstructures of the transition zones, of different matrix types. The morphologies of the microstructure of matrices I–III are shown in Figures 4a–4c, correspondingly. With the highest w:c ratio, matrix I results in a porous structure as can be seen clearly from Figure 4a. Figure 4b indicates that matrix II, with a slightly lower w:c ratio, has a less open microstructure than matrix I. On the other hand, due to the fine particle size of condensed silica fume (submicron in comparison to cement grain size of several to 10 μm) and the secondary pozzolanic reaction, matrix III gives the most compact microstructure, as shown in Figure 4c. The microstructure of matrix III is basically amorphous. No specific hydration product can be recognized. This is because the silica fume not only can fill in the originally open structure but also changes the morphology of the hydration product. With the drastic contrast among the material structure of different matrix types, the effects of reducing the w:c ratio and introducing microsilica fume on refining the matrix porosity are confirmed.

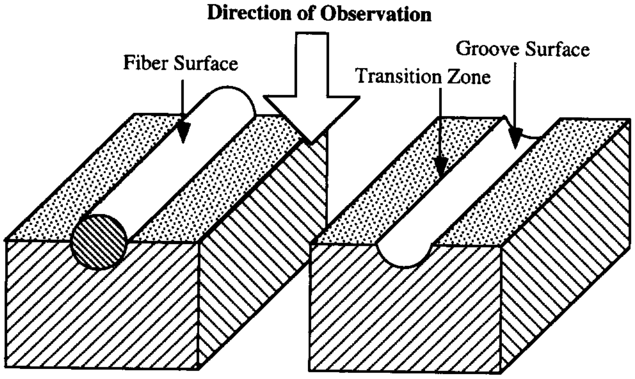


FIGURE 3. Schematic illustration of specimens for microstructure observation.

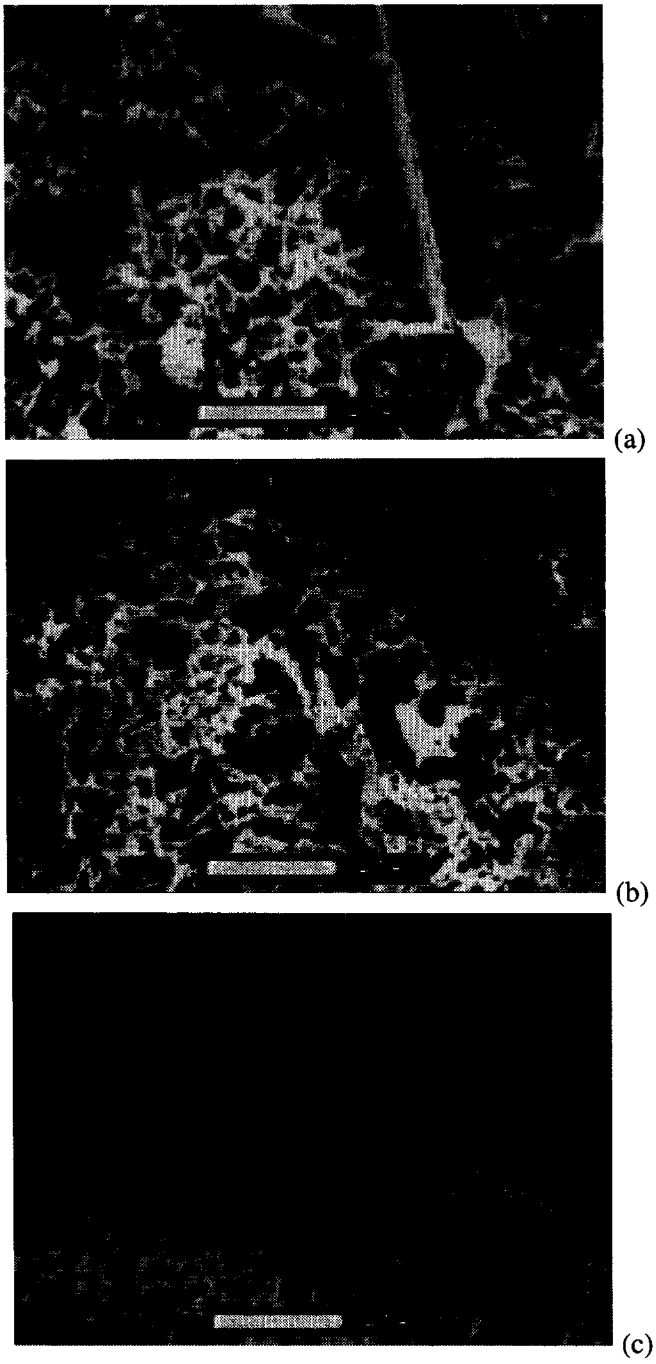


FIGURE 4. Microstructure of (a) matrix I (w:c = 0.40); (b) matrix II (w:c = 0.27); and (c) matrix III (w:c = 0.27, SF:c = 0.20, SP:c = 0.05). Scale = 20 μm.

Fiber-Matrix Bond Strength

Typical steel-cement pullout curves are shown in Figure 5. According to Leung and Li [22], this material system may be categorized as stable debonding, that is, no abrupt load drop following the peak load in the pullout curves. It is found that for the different fiber types tested in this program, the bond failures are all stable debonding. For these material systems, the elastic bond strength is identical to the frictional stress. Thus, only the interfacial friction τ is required for bond characterization of this particular material system. It can be interpreted from the peak load $P_{\max}$ using

$$\tau = \frac{P_{max}}{\pi d l} \qquad (1)$$

where d and l are the fiber diameter and embedment length, respectively.

It should be noted that τ in eq 1 measures only the initial frictional bond strength. In general, it has been found that τ is not constant during fiber pullout, but depends on slip distance. Slip-weakening is usually associated with metal fiber-cement paste interface systems, while slip-hardening is usually associated with polymeric fiber-cement paste interface systems [23]. The bond strength τ investigated here refers to the initial value prior to any significant amount of slip and is therefore dependent only on the peak load $P_{\max}$ and not on the subsequent slope of the descending branch of the pullout curve. The difference of this descending branch in the pullout curves (Figure 5) of the steel fiber derived for the three different matrices does suggest varying amount of slip-weakening during the sliding out process, presumably related to the different levels of damage of the matrices by the abrasive action of the fiber. Figure 5 indicates that the most dense matrix suffers the minimum damage and, therefore, the least slip-weakening effect.

The interpreted bond strength of steel and brass fibers embedded in various cement matrices tested at the age of 28 days is summarized in Figure 6. Besides the bond strength results from individual pullout specimens, Figure 6 also shows the effect of matrix type by the lines linking the averaged bond strength groups. The influence of cement matrix density on the interfacial bond strength of the steel-cement seems to be negligible. For various matrix types, the steel-cement bond strengths are about 4 MPa with only slight deviations. On the other hand, the bond strengths between brass fiber and cement matrix are noticeably affected by matrix types. The brass-cement bond strength apparently increases with the matrix packing density. Data scatter for the brass-cement system is probably due to the transition zone material failure, as will be further discussed in the following section. As shown in Figure 6, matrix I (w:c = 0.40) gives the lowest brass-cement bond strength followed by matrix II (w:c = 0.27). For the most compact matrix III (w:c = 0.27 and SF:c = 0.20), the brass-cement bond strength achieves an average of 4.7 MPa. In general, the bond strengths of brass-cement are higher than those of steel-cement.

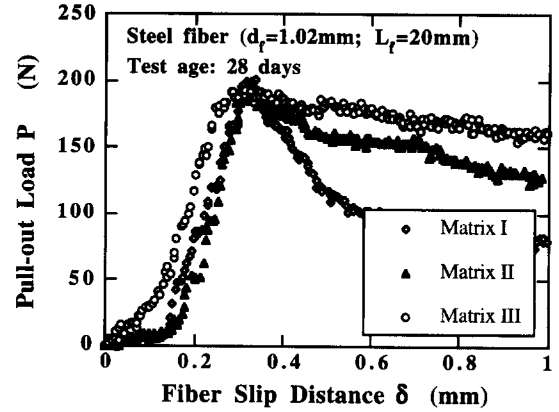


FIGURE 5. Typical steel-cement pullout curves.

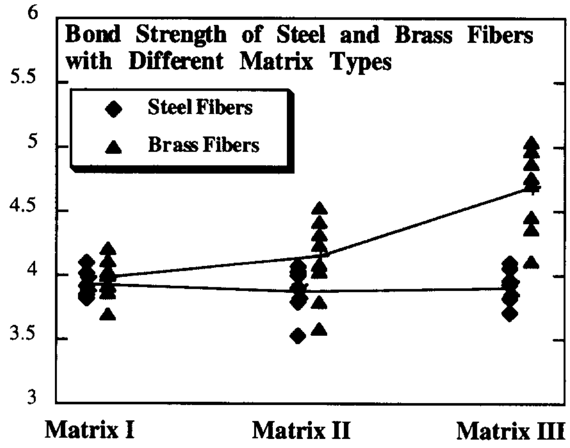


FIGURE 6. Steel and brass bond strengths with respect to various cement matrices.

The bond strength measurement of polyethylene is shown in Figure 7 in which the bond strengths of polyethylene fiber with matrices I and III are determined. Apparently, the effect of transition zone densification does not translate to bond strength enhancement in polyethylene-cement systems. The trend on bond measurement of various matrices of polyethylene-cement is similar to that of steel-cement.

In a study on the optimization on matrix packing density and its effect on the mechanical properties of cementitious composites, Glavind [24] conducted steel fiber pullout tests with various cement matrices. Silica fume or fine clay, with particle sizes on the order of submicron, was incorporated into the different matrices to densify the microstructure. The optimum dosages of silica fume and clay were 20% and 15% by weight of total cement content corresponding to maximum matrix

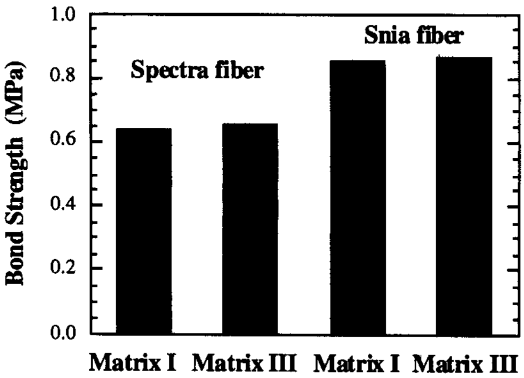


FIGURE 7. Effect of matrix type on polyethylene-cement bond strength.

packing densities (based on a geometric packing model). Compared with ordinary nonoptimized matrix, no increase in interfacial bond strengths for the matrices containing silica fume and clay were found. Some were even lower than ordinary matrix. The results indicate no correlation between packing density and interface bond strength. The present findings for interface densification effect on steel-cement (and polyethylene-cement) bond properties are consistent with Glavind's findings.

Microstructure Observation of Fiber Matrix Interface

Microstructure observations on virgin and peel-off fiber samples indicate that interfacial bond failures of the material systems tested in this experimental program occur at different locations: For steel or polyethylene fibers, bond failure occurs at the fiber-cement interface, whereas bond failure is found to take place in the porous transition zone between brass fiber and cement matrix.

Figure 8a–c shows the surface texture of virgin brass, steel, and polyethylene fibers. In general, the fiber surface is smooth except for some longitudinal markings caused by the manufacturing process. Figure 9a shows the surface morphology of the groove on the cement matrix left by peeling off a steel fiber. The groove surface is smooth, and the longitudinal markings resulting from contact with the steel fiber surface are well preserved. As a coupled specimen, the peel-off steel fiber is also observed. Figure 9b shows the surface of the steel fiber peeled off from cement matrix; it is comparable to the surface of the virgin fiber surface as shown in Figure 8b. This evidence indicates that, for the steel-cement system, bond failure during fiber peel-off occurs along the contact surface between steel fiber and cement matrix.

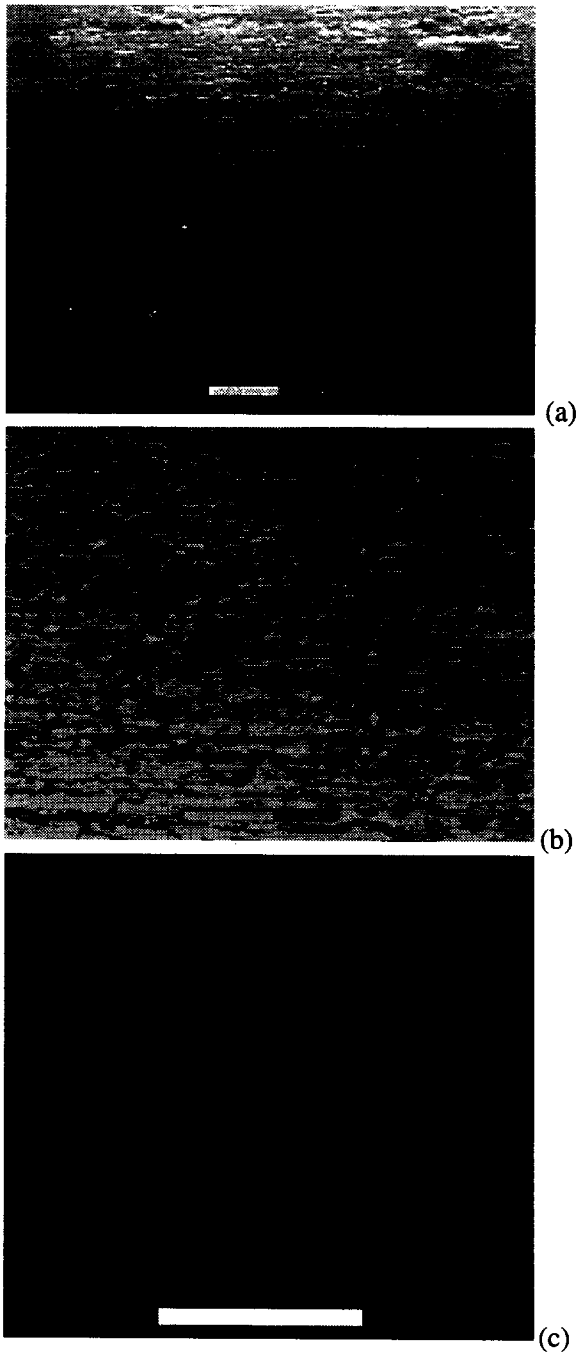


FIGURE 8. Virgin surface of (a) brass fiber (scale = 50 μm); (b) steel fiber (scale = 50 μm), and (c) Spectra fiber (scale = 30 μm).

As for polyethylene-cement system, (Figure 10a), the fiber-matrix surface is also well preserved without any evidence of transition zone material fracture due to fiber peel-off. The peel-off fiber surface (Figure 10b) is also found to be smooth and comparable to the virgin fiber.

For the brass-cement case, on the other hand, fiber debonding seems to be associated with fracture failure

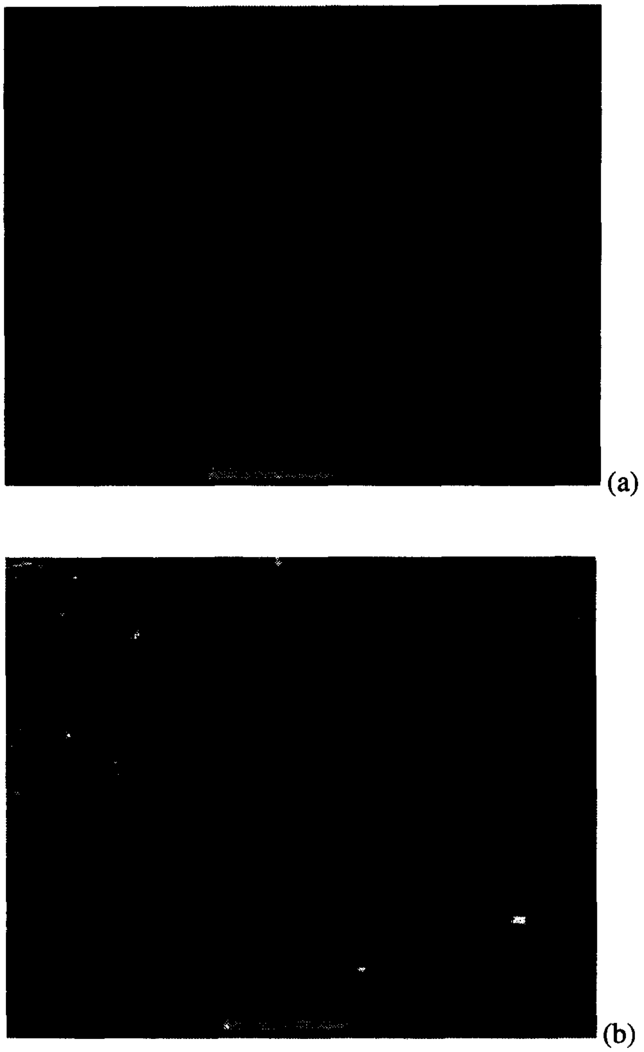


FIGURE 9. (a) Matrix groove and (b) steel fiber surface after peel-off (scale = 50 μm; matrix III).

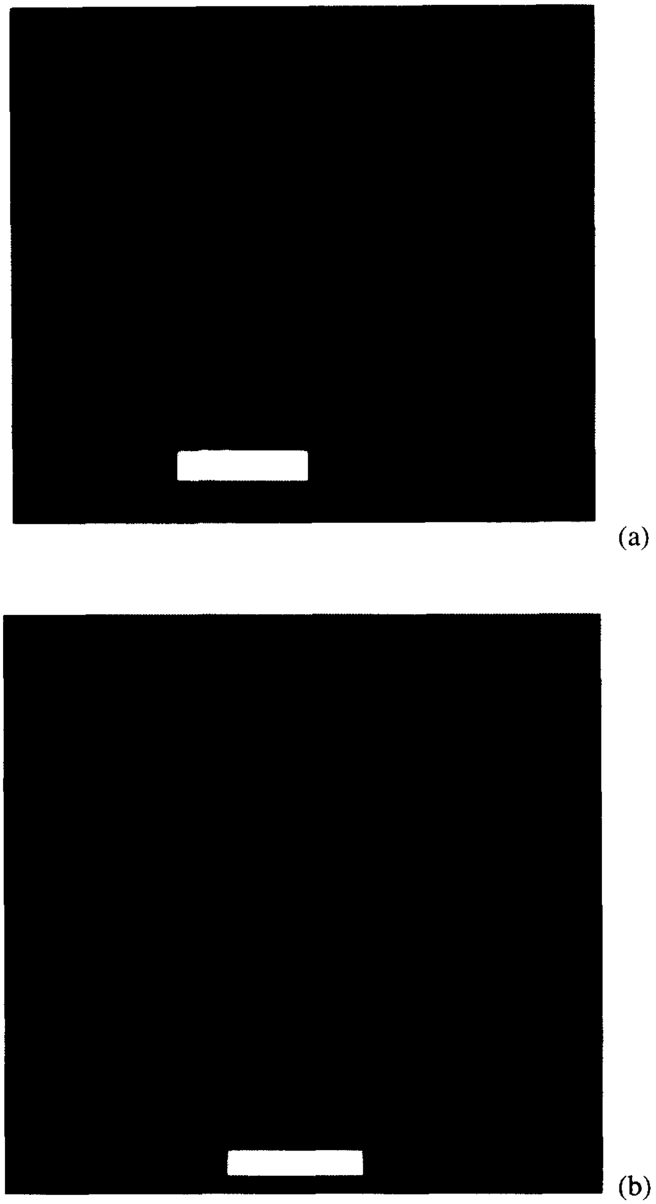


FIGURE 10. (a) Matrix groove and (b) Spectra fiber surface after peel-off (scale = 10 μm).

of the porous transition zone. Figure 11a shows a fracture surface along the groove on the matrix side left by peeling off the brass fiber. The separation between brass fiber and cement matrix apparently follows a tortuous path along the transition zone instead of the fiber-cement interface. The rough surface of the tortuous path suggests that the brass-cement bond failure has gone through the transition zone. Observations of the surface of the peel-off brass fibers also reveal consistent features, as shown in Figure 11b, where a layer of cement remnant can be recognized on the fiber surface.

Mechanisms of Interface Bond Failure

From the microstructure findings, two different patterns of bond failure are identified for different material systems. It is inferred that the adhesion between brass and cement is higher than that between steel and cement, because the bond failure occurs in the transition zone for the case of brass-cement but occurs at the fiber-cement contact surface for the case of steel-cement. This notion is supported by work of Khalaf and Page [25], who reported that, based on both SEM observation and experimental measurement of adhesion forces, brass possesses a much higher adhesion with cement than with mild steel. The higher adhesion of brass with cement is mainly due to the chemicophysical properties of brass that allows chemical reactions to occur while in contact with cement material [25].

With the scenario set forth by the pullout tests and microstructure studies described previously, it becomes

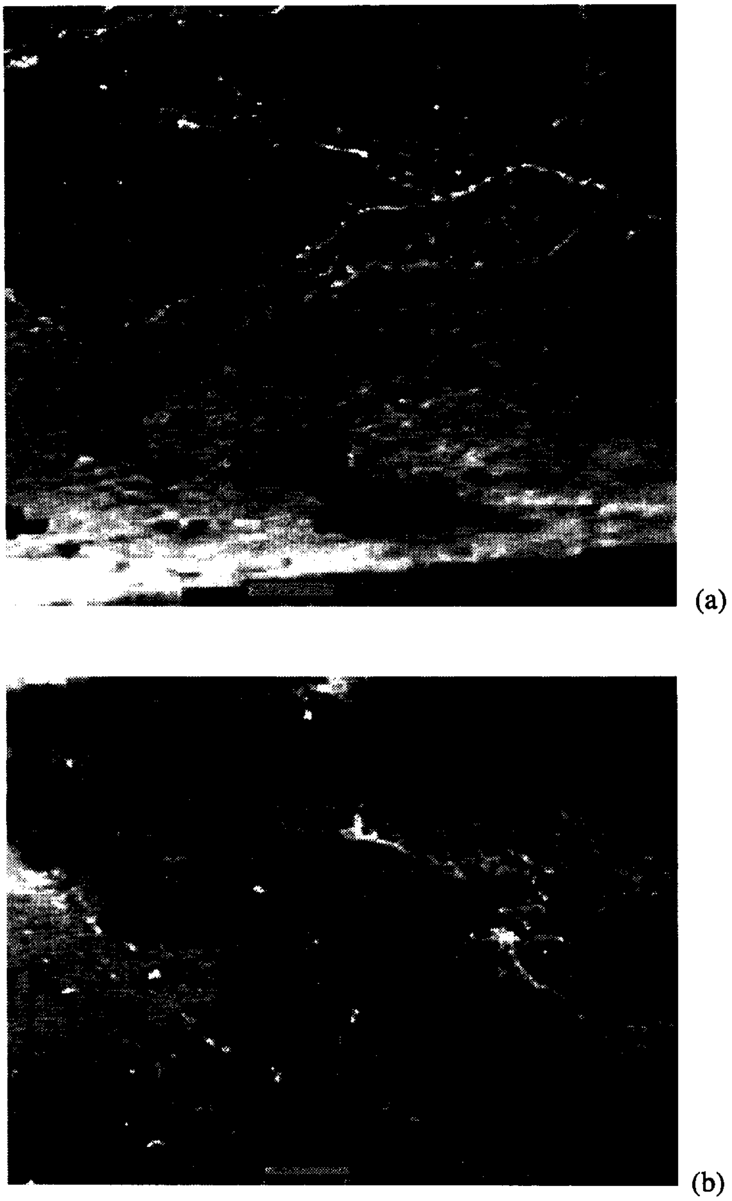


FIGURE 11. Matrix groove surface after brass fiber peeled off. Darker regions indicate transition zone materials lifted off and attached to fiber surface. (b) Brass fiber surface after separation from the cement matrix. Darker regions indicate transition zone materials. Bright spot on left shows a partially exposed fiber surface (scale = 50 μm; matrix III).

apparent that interface bond strength and debond mode are governed by the relative magnitude between fiber-matrix adhesion strength and transition zone material strength. To illustrate, if the adhesion strength (e.g., point B in Figure 12) is lower than the minimum strength (point C) in the transition zone, bond failure would follow along the fiber-matrix contact surface. This bond failure type can be categorized as adhesive type (as shown schematically in Figure 13a). For the current test result, the adhesion between steel or polyethylene fibers and cement can be so low that debonding occurs at the steel-cement or polyethylene-cement contact surface. For such material systems, strengthening the transition zone by a densification technique is not expected to improve the interfacial bond strength. This is consistent with the pullout test findings reported earlier. When fiber-matrix adhesive strength is low, it may be more effective to achieve bond strength improvement by fiber surface modification. A recent demonstration of this technique by means of plasma treatment on polyethylene fiber can be found in [26].

On the other hand, if the adhesion strength is higher (e.g., point A in Figure 12), fiber-cement bond failure would take place in the transition zone. This failure type can be categorized as cohesive type (Figure 13b). This scenario is consistent with the test results and microstructure observations in the brass-cement system reported earlier. For such material systems, strengthen-

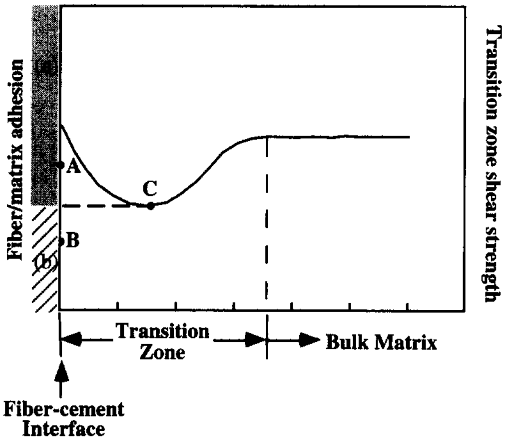


FIGURE 12. Schematic illustration of the conditions for different types of bond failures.

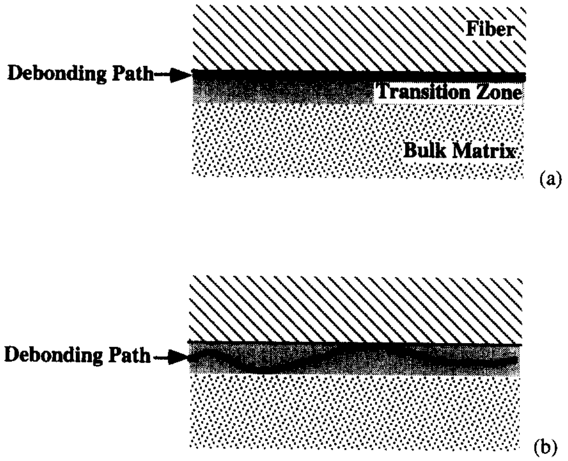


FIGURE 13. Schematic illustration of different types of bond failures: (a) adhesive type and (b) cohesive type.

ing the transition zone by the densification technique should be effective in enhancing the interfacial bond strength.

Conclusions

Based on the measurement of bond strength as well as the microstructure observation in steel-cement, polyethylene-cement, and brass-cement systems, interface bond failure was found to occur either at the fiber-cement contact surface or in the heterogeneous transition zone, depending on the relative magnitude between fiber-cement adhesive strength and transition zone material strength. It is concluded that adhesive bond failure occurs in the steel-cement and polyethylene-cement systems, and cohesive bond failure occurs in the brass-cement system studied.

Different interface bond failure mechanisms suggest different strategies of controlling fiber-cement bond strength. Transition zone microstructure densification may not be effective when the bond failure is governed by adhesion. This is confirmed by bond property measurement of steel-cement and polyethylene-cement systems, using different matrix types with controlled w:c and silica fume contents. On the other hand, when bond failure is governed by cohesive fracture in the transition zone, such as in the brass-cement system studied, bond strength improvement can indeed be achieved by the technique of transition zone densification.

It should be noted that in certain material systems, densification by particle packing may result in enhancement of contact surface area between fiber and transition zone material. This possible mechanism in improving adhesion, and therefore interfacial bond strength, even though not observed by the specific fiber-matrix material systems studied here, is not ruled out for other material systems.

This investigation has assumed that bond failure revealed by fiber peel-off is similar to that occurring in fiber pullout. This assumption is necessitated by the desire to observe the microstructure on the fiber surface and on the groove left by the peel-off fiber. It is expected that such microstructures will be masked by damage incurred in a fiber pullout process. While the previous conclusions are not entirely rested on this assumption, it should be pointed out that the details of interfacial failure may depend on the mode of loading (high shear in pullout, tensile opening in peel-off). Nonetheless, there is circumstantial evidence that in fiber pullout tests, cement remnants are more readily observed on brass fiber surfaces than on steel or polyethylene fiber surfaces, thus lending credence to the conclusions drawn from the present investigation.

Acknowledgments

The work presented here has been supported by grant No. MSS-9301949 from the National Science Foundation. We acknowledge helpful comments from three anonymous reviewers that led to improvements in the manuscript.

References

1. Marshall, D.B.; Cox, B.N.; Evans, A.G. *Acta Metallurgica,* **1989,** *15,* 2607–2619.
2. Majumdar, B.S.; Newaz, G.M.; Rosenfield, A.R. *Proc. 7th Int. Conf. on Fracture* **1989,** *4,* 2805–2814.
3. Budiansky, B.; Hutchinson, J.W.; Evans, A.G. *J. Mech. Phys. Solids* **1986,** *34,* 167–189.
4. Li, V.C.; Leung, C.K.Y. *J. Engin. Mech.* **1992,** *118,* 2246–2264.

5. Ping, X.; Beaudoin, J.J. *Cem. Concr. Res.* **1992,** *22,* 597–604.
6. Metha, P.K.; Gjorv, O.E. *Cem. Concr. Res.* **1982,** *12,* 585–595.
7. Bentur, A.; Cohen, M.D. *J. Amer. Ceram. Soc.* **1987,** *70,* 738–743.
8. Detwiler, R.J. *Cem. Concr. Res.* **1990,** *20,* 277–284.
9. Hild, D.N.; Schwartz, P. *J. Adhesion Sci. Tech.* **1992,** *6,* 897–921.
10. Stucke, M.S.; Majumdar, A.J. *J. Mater. Sci.* **1976,** *11,* 1019–1030.
11. Cohen, E.B.; Diamond, S. *Proc. RILEM Symp. on Fiber Reinforced Cement and Concrete;* Neville, A., Ed.; 1975, p, 315.
12. Bentur, A.; Diamond, S.; Mindess, S. *Cem. Concr. Res.* **1985,** *15,* 331–342.
13. Bentur, A.; Diamond, S.; Mindess, S. *J. Mater. Sci.* **1985,** *20,* 3610–3620.
14. Pinchin, D.J.; Tabor, D. *Cem. Concr. Res.* **1978,** *8,* 15–25.
15. Bentur, A.; Mindess, S. *Fiber Reinforced Cementitious Composites;* Elsevier Applied Science: New York, 1990, pp 22–30.
16. Wei, S.; Mandel, J.A.; Said, S. *ACI J.* **1986,** July-August, 597–605.
17. Sereda, P.J. *Cem. Concr. Res.* **1972,** *2,* 717–729.
18. Knudsen, F.P. *Dependence of Mechanical Strength of Brittle Polycrystalline Specimens on Porosity and Grain Size;* presented at the 59th Annual Meeting of the American Ceramic Society, Dallas, Texas, 1985.
19. Martin, R.B.; Haynes, R.R. *J. Amer. Ceram. Soc.* **1971,** *54,* 410–411.
20. Beaudoin, J.J.; Feldman, R.F.; Tumidajski, P.J. *Adv. Cem. Based Mater.* **1994,** *1,* 224–236.
21. Li, V.C.; Chan, Y.W. *J. Engin. Mech.* **1994,** *120,* 707–719.
22. Leung, C.K.Y.; Li, V.C. *J. Mater. Sci.* **1991,** *26,* 5996–6010.
23. Wang, Y.; Li, V.C.; Backer, S. *Int. J. Cem. Comp. Lightweight Concr.* **1988,** *10,* 143–149.
24. Glavind, M. *Ph. D. thesis;* Technical University of Denmark, Lyngby, 1992.
25. Al Khalaf, M.N.; Page, C.L. *Cem. Concr. Res.* **1979,** *9,* 197–208.
26. Li, V.C.; Wu, H.C.; Chan, Y.W. *J. Am. Ceram. Soc.* **1996,** *79,* 700–704.