Effects of Poly(vinyl alcohol) on Fiber Cement Interfaces. Part I: Bond Stress–Slip Response

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This is the first part of a two-part article describing the effects of adding poly(vinyl alcohol) (PVA) to a cement based matrix to improve the bond at the fiber–matrix interface. Two types of fibers were used, steel and brass fibers (simulating brass-coated steel fibers) in a series of pull-out tests where the load versus global slip up to complete pull-out was recorded. The measured slip was that at the section where the fiber penetrates the matrix. The first article describes the mechanical effects of the addition of PVA, while the second article presents the microscopic observations. Correlation between the two studies is pointed out in the second part and conclusions are drawn. In particular, it is observed that the addition of PVA in the amount of 1.4% by weight of cement matrix leads to a significant improvement in the bond strength as well as in the frictional resistance, thus pull-out work, after the peak load. ADVANCED CEMENT BASED MATERIALS 1994, 1, 115–121

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The nature of bond in today's fiber-reinforced cementitious composites is very complex because of the presence and the combined action of several bond components. These include: (1) physical and chemical adhesion between fiber and matrix, (2) the mechanical component of bond such as in deformed, crimped, and hooked fibers, (3) fiber-to-fiber interlock, or entanglement, due to high fiber content, and (4) friction which is greatly influenced by confinement. Several recent analytical and experimental studies have addressed this problem [1–9].

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The addition of polymers to a cement matrix can add significantly to the magnitude of the "adhesion" component of bond at the fiber-matrix interface. For example, Wei et al. [7,8] found that the pull-out force of a fiber was almost quadrupled by adding 15% by weight of cement of a water-dispersion of acrylic polymer particles. This improvement was suggested to arise from the small size (50 to 100 nm) of the acrylic polymer particles which, being smaller than the cement particles, are able to fill in the porous zone that typically surrounds the fibers. In the work described here, a water-soluble polymer, poly(vinyl alcohol) (PVA), was used. This polymer is dispersed as molecules which are orders of magnitude smaller than particles, and the question to be answered is whether a much smaller amount of this polymer could be as effective as the acrylic particles, such as used by Wei et al.

In the first part of this study pull-out tests are carried out to investigate the effects of adding PVA to the cement matrix on the pull-out load versus slip response of plain smooth steel and brass (simulating brass-coated steel) fibers. The second part of this study deals with the microscopic observations of the effect of PVA on the interface zone between the fibers and the cementitious matrix, and provides some correlation with results of the first part.

Experimental

Six series of pull-out tests were undertaken to correlate with the microscopic observations. Each series had four pull-out fibers. The polymer used was PVA (Airvol-203). The tests were designed to investigate the effects of the polymer addition to the slurry matrix on the fiber–matrix interfacial bond. Two types of fibers

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were used: steel and brass. Because available brass-coated steel fibers were not long enough to work with the pull-out test set-up, brass fibers were used instead. The difference between the Poisson's ratio of brass fibers (ν = 0.35) and that of brass-coated steel fibers (ν = 0.30) was assumed sufficiently close to have little effect on the pull-out load versus slip response curve. The fibers used were smooth fibers, with two different diameters for the steel fibers, namely 0.03 in. (0.75 mm) and 0.016 in. (0.4 mm), and one diameter for the brass fibers, that is, 0.03 in. (0.75 mm).

The matrix used was a cement based slurry with type I cement. The water-to-cement ratio was 0.35. Most specimens were about 28 to 30 days old at time of testing. When PVA was used, its proportion was 1.4% by weight of cement (equivalent to 4% by weight of water). The PVA was dissolved in the water prior to its addition to the mix. The specimens were cured in water for 1 week, then left in laboratory air environment until testing (about 70% relative humidity and 20°C). The 28 days compressive strength of the plain slurry matrix, as measured from tests on 4 × 8 in. (100 × 200 mm) cylinders was about 5 ksi (35 MPa). This value may seem too small for a water-to-cement ratio of 0.35. However, it is justifiable because (1) the matrix was a paste without sand, (2) shrinkage cracks were certainly existent, and (3) the specimen size was relatively large for a paste. Moreover, the air entrainment induced by the addition of PVA may have also contributed to a smaller compressive strength. For the pull-out specimens, the pull-out fiber was embedded 1 in. (25 mm) in the matrix and the fiber did not extend below the bottom surface of the specimen; thus the embeddment length decreases during pull-out.

Mixing PVA with Cement Based Slurry

PVA solids were mixed with water before the solution was added to the cement. It was observed that about 30 minutes are needed to dissolve the total amount of PVA in the water, otherwise some of the undissolved PVA could be observed at the bottom of the container. It was also observed that when the water solution is added to the cement in the mixer, air bubbles appear on the surface of the fresh mix, especially when the mixing speed was relatively high. One trial PVA solution was prepared in a food blender at high speed. A large volume of foam accumulated in the blender. The foam was left to settle for about 3 hours before adding the solution to the cement. In this case fewer bubbles were observed when the solution was added to the mortar mix compared to the solutions mixed by hand. Also, the density increased by almost 15% compared to that obtained when PVA was hand mixed. In the experiments described here, the hand mixing method was followed.

Test Set-Up and Testing Procedure

Each test series consists of a block of matrix 7 × 4 × 1 in. (17.5 × 10 × 2.5 cm) crossed by four pull-out fibers (Figure 1). The test set-up used is illustrated in Figures 2 and 3. The pull-out test procedure is applied to one fiber at a time. As can be seen in Figure 2, the top end of the fiber is held by a specially designed grip attached to the load cell of an INSTRON machine. The displacement of the top end of the fiber is monitored by a miniature LVDT installed inside the grip. The end of the core of this LVDT touches the top surface of the specimen. A gap of about 0.5 in. (12.5 mm) exists between the top surface of the specimen and the bottom surface of the grip. The upward movement of the crosshead of the testing machine applies a pull-out force on the top end of the fiber. The cross-head speed was 0.075 mm/min for the initial portion of the curve up to a slip about three times that of the peak load. To complete each test in a reasonable time, the crosshead speed was increased to 0.5 mm/min in the descending branch of the curve during frictional pull-out. The value of the force, as well as the global slip at the end of the fiber, were recorded by a data acquisition system and stored in a computer file. The pull-out load versus end slip relationship was then developed and plotted using such data files. Additional details about the pull-out load versus slip test procedure and data interpretation can be found in ref 1.

Curve Averaging Procedure

Each series yielded four pull-out load versus slip curves, one for each fiber; for comparative purposes, a representative average curve was needed for each series tested. The criteria used to chose the proper averaging method were that the average curve have a peak pull-out load, P_p, equal to the average of the peak pull-out loads recorded for the individual pull-out tests, and an end slip at peak, Δ_p, equal to the average end

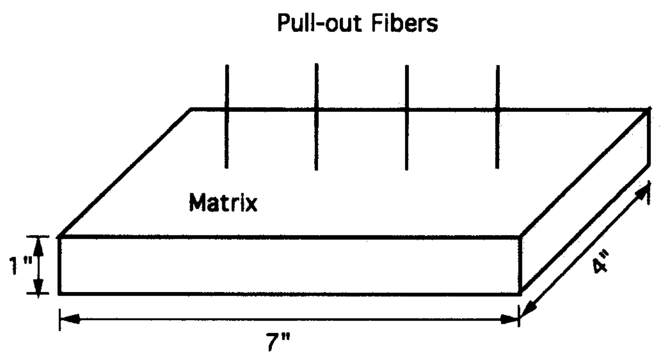

FIGURE 1. Typical pull-out specimen (1 in. = 25.4 mm).

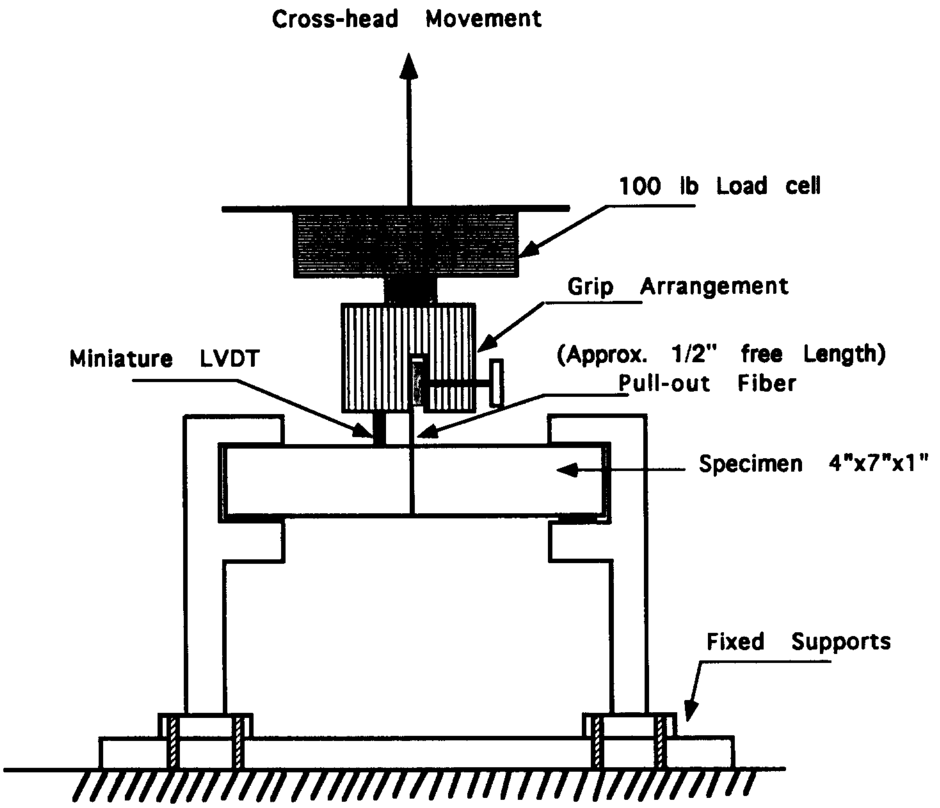

FIGURE 2. Pull-out test fixture (1 in. = 25.4 mm).

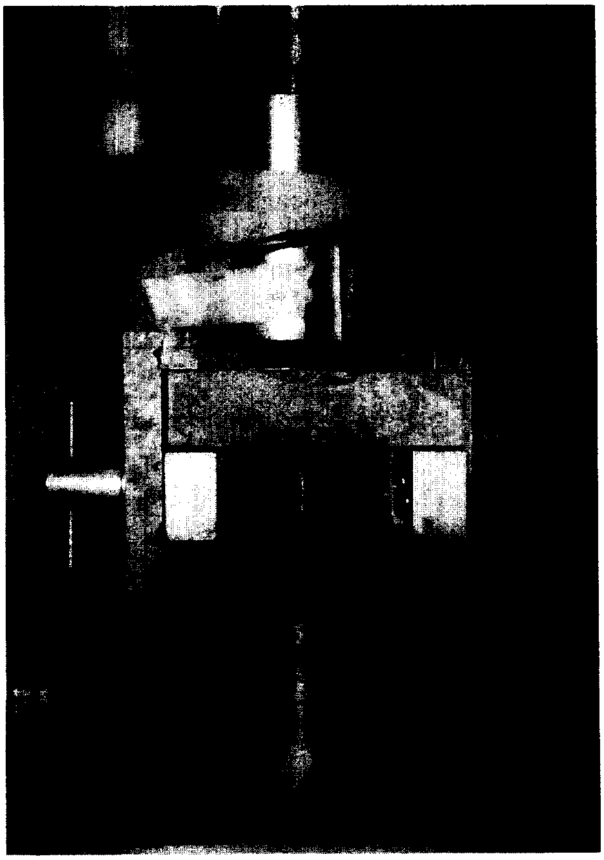

FIGURE 3. Photograph of specimen and test setup.

slips at peak for the individual tests. Equal weights were assigned to all curves used in averaging. Then the pull-out loads on the ascending (prepeak) branch of each pull-out curve were evaluated at 50 equal end slip intervals. The interval was selected in terms of each curve's own peak displacement. The average of these loads at each interval was evaluated, and taken as the average load at the corresponding end slip interval based on the peak point of the average curve. The same interval was used for the descending (post-

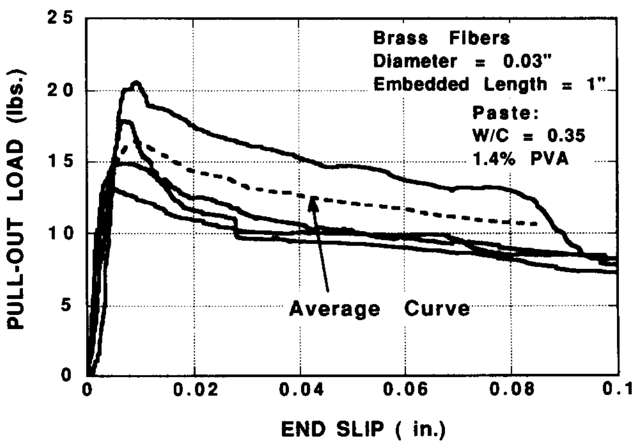

FIGURE 4. Typical pull-out curves of brass fiber series and the average curve (1 lb = 4.448 N, 1 in. = 25.4 mm).

peak) branch of the average curve. An example of a series of four pull-out versus slip tests and the average curve (in dotted line) is shown in Figure 4. It is noted that, although the measured slip at peak load is very small, the elastic strain contribution to the measured slip can be significant, especially for small fiber diameters. This contribution to slip is due to the fiber strain under load over the free length of the fiber. A prior investigation has indicated that the net slip at the peak load can be several times smaller than the measured slip [1]. However, the influence of the elastic strain becomes insignificant in the descending branch of the pull-out curve. Note that the slip reported in the figures of this article is the displacement observed at the free tip of the fiber without correction for the elastic strain effect.

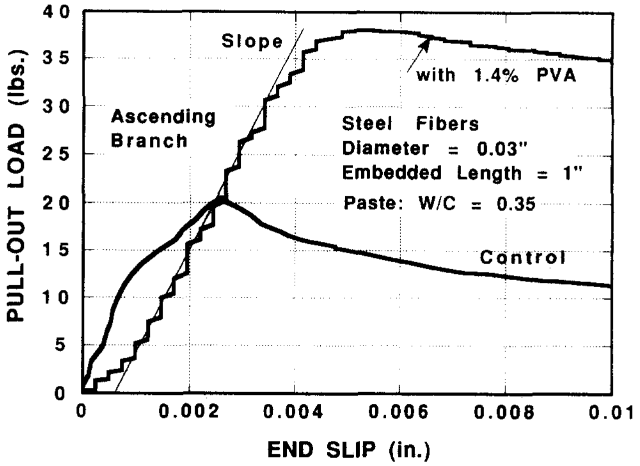

FIGURE 6. Magnified ascending branch and corresponding slope for 0.03 in. diameter steel fiber (1 lb = 4.448 N, 1 in. = 25.4 mm).

Pull-Out Load Versus End Slip Response

A schematic pull-out load versus slip curve for smooth steel fibers is shown in Figure 5 [1,2]. In the ascending branch, it is observed that the curve remains linear up to a certain load P_{crit} smaller or equal to the peak load. When the fiber fully debonds, the load drops quickly and the resistance to pull-out is mainly provided by friction between the fiber and the matrix.

Figures 6, 7, and 8 are plotted on three different scales to illustrate the details of the various portions of actual pull-out load versus slip curves. Figure 6 shows the ascending branch of the pull-out load versus slip curve for smooth steel fibers with and without PVA. Here the total slip scale is 0.25 mm (0.01 in.). It can be seen that not only is the peak load almost doubled by the presence of the PVA, but also after the peak, the load does not drop as quickly as for the case of the plain slurry. Figures 7 and 8 give a better idea of the overall response up to a total slip of 2.5 and 10 mm (0.1 and 0.4 in.), respectively. They show that, in the descending branch, the area under the load-slip curve, hence the pull-out work, with PVA is about two to three times that of the control curve. Similar trends were also observed for steel fibers with smaller diameter, as shown in Figure 9.

The effect of polymer addition on brass fibers was less pronounced than for steel fibers (Figure 10). Compared to the control, the increase in the maximum pull-out load as well as the area under the curve due to PVA addition was about 40%.

The pull-out load versus slip curves for steel and brass fibers of the same diameter with PVA addition are compared in Figure 11. It can be observed that the peak load for steel fibers is approximately twice that of

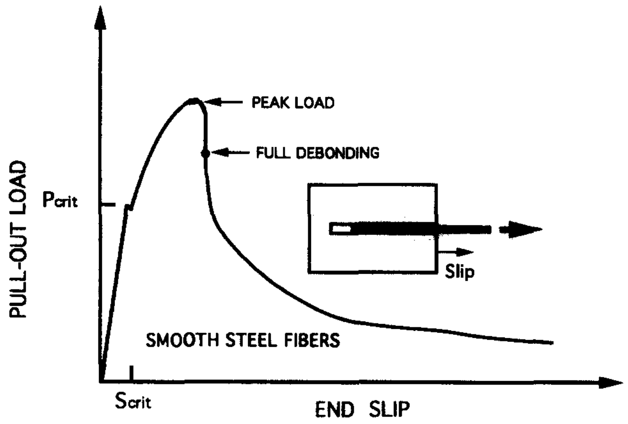

FIGURE 5. Schematic representation of typical pull-out curve of smooth fibers (1 lb = 4.448 N, 1 in. = 25.4 mm).

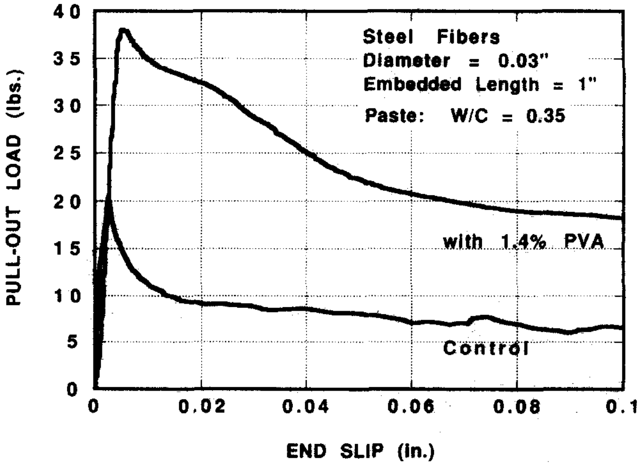

FIGURE 7. Effect of PVA on the pull-out curve of 0.03 in. diameter steel fiber up to 0.1 in. slip (1 lb = 4.448 N, 1 in. = 25.4 mm).

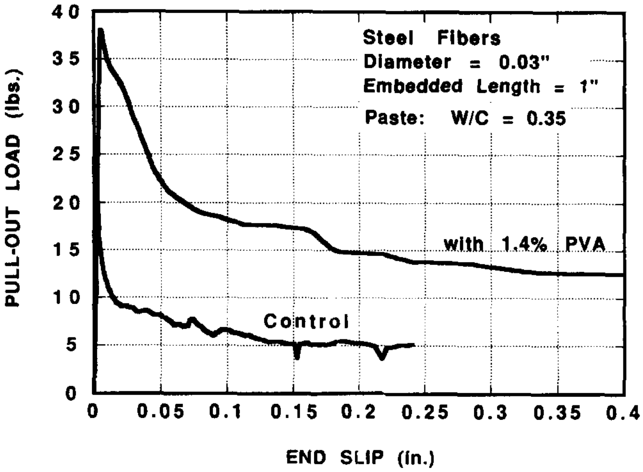

FIGURE 8. Effect of PVA on the pull-out curve of 0.03 in. diameter steel fiber up to 0.4 in. slip (1 lb = 4.448 N, 1 in. = 25.4 mm).

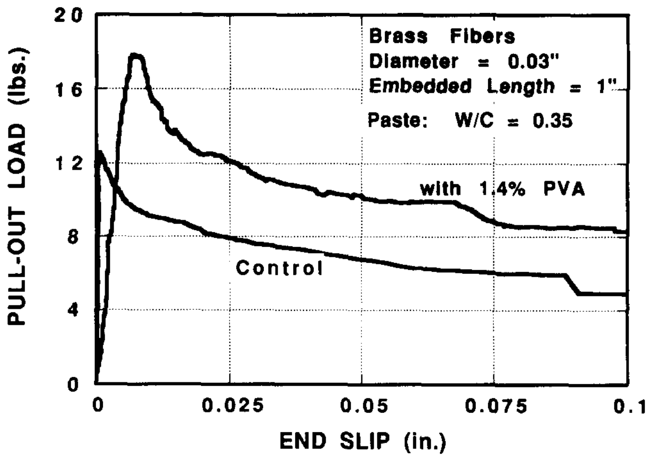

FIGURE 10. Effect of PVA on the pull-out curve of 0.03 in. diameter steel fiber up to 0.4 in. slip (1 lb = 4.448 N, 1 in. = 25.4 mm).

brass fibers. Moreover, in the descending branch, the steel fiber requires more than twice the amount of work needed to pull out a brass fiber of the same diameter.

In general, whether steel or brass fibers are used, the addition of PVA increased the frictional resistance to pull-out; however, this increase was larger for steel than that for brass fibers. This behavior was different from that reported for other additives, such as latex, which did not lead to any increase in the frictional resistance to pull-out [1].

A summary of the observed maximum values of the pull-out loads and the corresponding end slips with and without PVA are shown in Table 1. Because the elastic strain contribution of the free portion of the fiber to the measured end slip is significant, two values of slip at the peak load are given: one as measured (i.e., including the elastic strain contribution) and one corrected (i.e., net slip of the embedded portion of fiber). The contribution of the elastic strain was assumed acting over a length of 0.75 in. (19 mm), for an actual free length of 0.5 in. (12.5 mm) to account for the portion of fiber within the jaws of the gripping device. Two observations can be made, namely, the net slip can be significantly smaller than the measured slip, and either value is relatively small in comparison to the slips measured in the postpeak regime.

Pull-Out Work

The pull-out work, or the dissipated bond energy, is defined as the area under the pull-out load versus slip curve. Examples of pull-out work values calculated for the steel and brass fibers are shown in Table 2. The

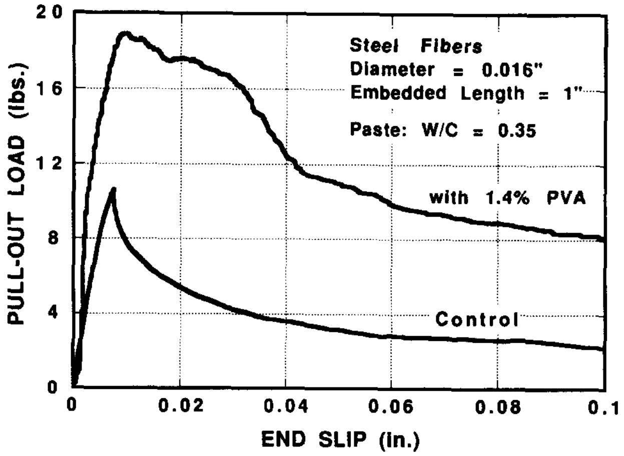

FIGURE 9. Effect of PVA on the pull-out curve of 0.016 in. diameter steel fiber up to 0.4 in. slip (1 lb = 4.448 N, 1 in. = 25.4 mm).

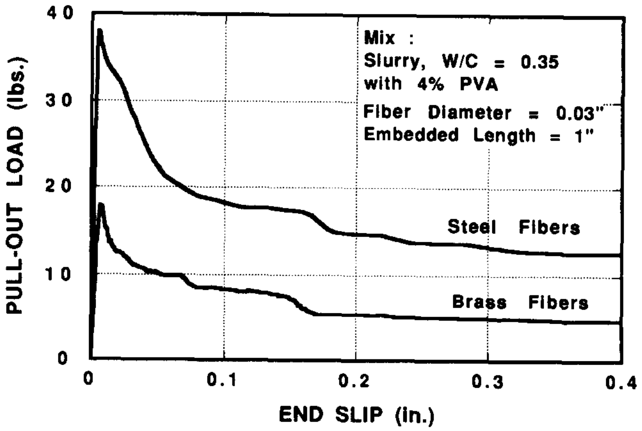

FIGURE 11. Typical comparison of pull-out load versus slip curves of steel and brass fibers with PVA (1 lb = 4.448 N, 1 in. = 25.4 mm).

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TABLE 1. Typical average pull-out data for steel and brass fibers with and without PVA*

Fiber Type	Diameter (in.)	P_{peak} With PVA (lb)	Δ_{peak} With PVA (in.)	P_{peak} Without PVA (lb)	Δ_{peak} Without PVA (in.)	Δ_{peak} With PVA Net (in)	Δ_{peak} Without PVA Net (in)
Steel	0.016	17.5	0.00732	10.8	0.0075	0.00432	0.00564
Steel	0.030	37.6	0.00541	20.4	0.00277	0.00357	0.00177
Brass	0.030	18.1	0.00767	12.7	0.00266	0.00590	0.00144

*Values in columns 4 and 6 do not account for fiber elastic strain; values in columns 7 and 8 are net, excluding elastic strain contribution (1 lb = 4.448 N, 1 in. = 25 mm).

work was calculated for three values of slip, namely, the slip at peak load, the slip at 0.2 in. (5 mm), and the slip at 0.4 in. (10 mm). The pull-out work up to 5 mm for steel fibers with PVA was three to four times that without PVA. This ratio became two to three for an end slip of 10 mm. In the case of brass fibers, the pull-out work up to 5 mm end slip for specimens with PVA was 50% more than that for specimens without PVA. Comparing steel and brass fibers with PVA, the pull-out work of steel fibers up to an end slip of 5 mm was two to three times that of brass fibers. This ratio remained approximately the same for a 10 mm end slip. Table 2 also shows the pull-out work of steel and brass fibers up to the peak loads. The numerical values given are net values and do not include the effect of fiber elastic strains. Theoretical considerations indicate that the peak load either corresponds to the end of the debonding process or to a point close to it [2]. Alwan et al. [3] used the term debonding pull-out work to describe this component of the total pull-out work. From Table 2 and the pull-out curves, in general, it can be observed that the debonding pull-out work of smooth fibers up to their peak loads can be one to two orders of magnitude smaller than the pull-out work of these fibers in the descending branch. This confirms previous observations that the main contributor to the pull-out work of fibers from cement matrices is friction, not adhesion, and that the total pull-out work can be, as a first approximation, obtained from the postpeak response of the curve. Thus, an additive that improves the frictional response can be much more effective in increasing the toughness of the composite than an additive that improves adhesion.

General Observations and Conclusions

From the results of this experimental study on the pull-out load versus slip response of fibers embedded in a cement matrix modified by PVA addition, the following conclusions can be drawn:

1. The addition of 1.4% by weight of PVA to the plain matrix leads to an increase in both the maximum pull-out load and the frictional resistance to pull-out for both steel and brass fibers. This increase was more significant for steel fibers (60% to 80%) than for brass fibers (42%).
2. The drop in the pull-out load after the peak for specimens with PVA was less drastic than that for the plain matrix or the matrix with a latex additive [1]. With PVA, the drop in the pull-out load was gradual, hence leading to an increase in the pull-out work.
3. The addition of the PVA water solution (about 4% PVA by weight of water) to the matrix induced air voids in the matrix during mixing. Because air entrainment leads to a decrease in the density of the matrix, the bond characteristics at the fiber–matrix interface could be affected should the proportion of PVA be increased. This point deserves further in-depth investigation.
4. For the range of parameters tested in this study, particularly the fineness of the cement binder, the effect of fiber diameter on the characteristics of the pull-out load versus end slip relationship with or without PVA was not significant.
5. The use of brass coating to protect steel fibers

TABLE 2. Typical pull-out work of smooth steel and brass fibers*

	Work Values Without PVA			Work Values With PVA		
Fiber Type	W_{peak} (lb-in.)	$W_{0.2''}$ (lb-in.)	$W_{0.4''}$ (lb-in.)	W_{peak} (lb-in.)	$W_{0.2''}$ (lb-in.)	$W_{0.4''}$ (lb-in.)
Steel, d = 0.016"	0.0251	0.408	1.06	0.113	1.95	3.08
Steel, d = 0.03"	0.0183	1.41	2.54	0.0952	4.13	6.80
Brass, d = 0.03"	0.0344	1.21	2.10	0.0702	1.75	2.73

*Work values are in (lb-in.) up to the slip indicated in subscript. W_{peak} is net value, excluding contribution of fiber elastic strain (1 lb = 4.448 N, 1 in. = 25 mm; 1 lb-in. = 0.113 J).

from corrosion during storage can be detrimental to their bond properties. Indeed, the average peak pull-out load for brass fibers with and without PVA was only 62% and 48% of that of steel fibers, respectively.

6. The addition of PVA to the matrix increased the work of pull-out measured at different slips. The ratio of debonding pull-out work with PVA to that without PVA, measured at the peak load, varied between four to five for steel fibers and was about two for brass fibers. The ratio of frictional pull-out work was about 2.75 for steel fibers and 1.3 for brass fibers.
7. Because frictional pull-out work is one to two orders of magnitude larger than debonding pull-out work, improving frictional resistance through an additive, such as PVA, can be much more effective than improving adhesion only.

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