INTERFACE PROPERTY TAILORING FOR PSEUDO STRAIN-HARDENING CEMENTITIOUS COMPOSITES

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It is well known that interfaces in composites play an important role in determining composite properties. In this paper, the role of interface properties on pseudo strain-hardening properties of brittle cement matrix composites is briefly summarized. An example of interface tailoring by plasma treatment on polyethylene fibers and its effect on composite behavior is described.

1. Introduction

Materials like cement are considered brittle. Brittle materials typically have tensile strength much lower than their compressive strength. With low ductility and fracture toughness, they are also prone to fail by fast fracture. Fiber reinforcement in brittle matrix composites are therefore directed towards enhancing tensile strength and strain, and fracture resistance.

Many studies have been conducted on the fracture resistance of fiber reinforced cement and concrete, and this property has been enhanced steadily over the years. The increase in tensile strain capacity, essentially achieved by modifying the failure mode from quasi-brittle to ductile, however, has remained a theoretical possibility until recent years. In the past, this concept of pseudo strain-hardening has been demonstrated in real material systems only with continuous aligned fiber reinforcement, or with large volume content of fibers. Both requirements restrict practical applications due to cost and fabrication obstacles.

However, recent advancements in micromechanical theory has led to the design of pseudo strain-hardening cementitious composites with only a few percent by volume of discontinuous fibers [1,2]. A variety of fibers, including steel, polymer and carbon, can be utilized. One of the most ductile composites has been designed with 2% of polyethylene fibers. This composite has achieved a ductility of 4% and a fracture energy of 35 kJ/m^2. This type of composite is now poised for a variety of applications in the building, construction and transportation industries.

2. Role of Interface

With respect to fracture resistance, fiber/matrix interfaces are designed for high energy absorption by frictional sliding. Excessive bond tends to reduce the composite fracture energy because of fiber rupture [3-5]. For composite ductility via pseudo strain-hardening, fiber/matrix interfaces are designed for high load transfer across matrix cracks. For this reason, good bonding property is the objective.

Good bonding property leads to the following composite enhancements:

- Low fiber volume fraction to reduce cost and improve processability

- **High** strain capacity or ductility

- **High** tensile strength

The theoretical treatment of these subjects can be found in [1,2,6].

3.   Interphase and Interface

In fiber reinforced composites, there are two different failure modes when fibers are pulled out from matrix. Adhesive failure occurs following the exact interface between fiber and matrix, whereas adherend failure takes place within the matrix. For fiber reinforced cement-based composites, a distinctive layer of interphase zone is typically observed [7,8]. This zone is considerably weaker than the bulk matrix due to large CH crystals and higher porosity as reflected in microhardness tests which show that the weakest point in the interphase zone is about 30 μm away from the fiber surface [8]. This weak zone may be diminished or even removed by control of packing density and hydration around fiber interface. Silica fume and superplasticizer are found to be very effective [8,9]. Depending on fiber types and matrix constituents, either adhesive or adherend failure can occur in fiber reinforced cementitious composites. Hence proper strategy should be employed towards enhancing interfacial bonds or strengthening the interphase zone. In our previous investigation, polyethylene and steel fiber composites all exhibit adhesion-type failure. Clean fiber surface with little or no matrix residue after being pulled out are observed. This implies that interphase densification may not be effective in such composites [10,11]. For these composites, therefore, research should be directed towards improving bond strength through interface modification.

4.   Interface Property Modification by Plasma Treatment

Interface property modification can be achieved by a variety of techniques. These include, e.g., fiber deformation techniques such as twisting, crimping, pitting or button-end creation, surface coating such as use of coupling agents, and surface modification techniques such as chemical oxidation, corona treatment and plasma treatment. These surface modification techniques are directed towards improving interfacial strength, and have been employed with various degrees of success in polyethylene fiber reinforced epoxy and PMMA composites. The most significant improvement in adhesion has been accomplished with cold gas plasma [12].

A plasma is generated by exciting gas molecules with a source of electrical energy. When this energy is applied to the gas, electrons are stripped from the molecules, producing a mix of highly reactive disassociated molecules [12]. Hence the mechanism for surface modification of polymer fibers in a gas plasma is the removal of hydrogen atoms from the polymer backbone followed by their replacement with polar groups. The presence of polar or functional chemical groups on the fiber surface enhances reactivity with the resin matrix, thus promoting excellent adhesion [12,13]. The selection of reaction gases and process conditions such as generator power and reactor pressure provides opportunities for tailoring fiber surface chemistry and reactivity most adequate for a given matrix.

Various gases, ammonia, air, nitrogen, argon, and carbon dioxide have been employed in production of polyethylene/epoxy or polyethylene/PMMA composites [14-17]. In general, it is found that the interfacial bond strength can be readily doubled with only a few minutes' fiber exposure to plasma. Prolonged exposure does not improve further. In addition to excellent adhesion, plasma treated fibers also exhibit significantly enhanced pull-out energy in single fiber pull-out test.

In the present research, a radio frequency discharge plasma surface treatment system is utilized to create the plasma for fiber treatment. High modulus polyethylene fibers were plasma treated with argon gas at a flow rate of 40 ml/min and power level of 58 W for 5 minutes. Subsequently, the treated fibers were mixed with cement paste to form the composite in an identical manner as previous polyethylene fiber reinforced cementitious composites without the plasma treatment. Two types of specimens were prepared. Direct pull-out specimens were prepared to study interfacial bond properties. Uniaxial tension coupon specimens were prepared to study uniaxial tensile response of the composite.

5. Interface Properties

Fiber pull-out tests were conducted by pulling individual fibers out of cement matrix bases. The test setup and specimen configuration of the fiber pull-out test are shown in Figure 1. A fiber sample is partially embedded in the dog-bone shape
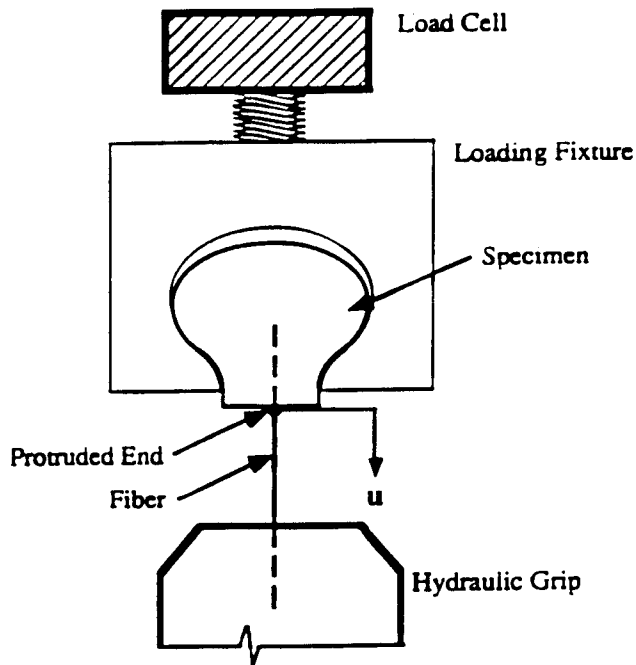

Figure 1 - Single fiber pull-out test setup.

specimen. The dog-bone shaped specimen is held by the loading fixture connected to a load cell. On the other end of the specimen, a hydraulic grip is used to hold the protruding fiber such that no slip between the grip and the fiber may happen. The pull-out test is conducted using a uniaxial hydraulic MTS testing machine which applies a constant displacement rate to the fiber grip. A computer data acquisition system is employed to collect data during the tests, including the applied load P obtained from the load cell and the displacement of the fiber grip by measuring the cross-head movement. The displacement of the fiber protruded end u is obtained by subtracting the elastic stretch of the fiber free length between the matrix base and the fiber grip from the measured cross-head displacement. The elastic stretch of the fiber free length

at any given applied load, in turn, is calculated based on the initial fiber free length, fiber cross-sectional area, and fiber elastic modulus. In general, the interfacial bond properties are interpreted based on these P-u curves obtained from the pull-out tests [18].

In preparation of the test samples, specimens were demolded 24 hours after casting and were cured in a water tank till testing. Fiber pull-out tests were conducted at the age of 28 days. The matrix was composed of Type I Portland Cement with a water/cement ratio by weight of 0.4. At least 6 specimens were tested for plasma treated and non-treated samples.

Figure 2 shows typical P-u curves from pull-out tests of plasma treated and non-treated spectra fibers. Fibers have embedment length of 12 mm. Generally, the pull-
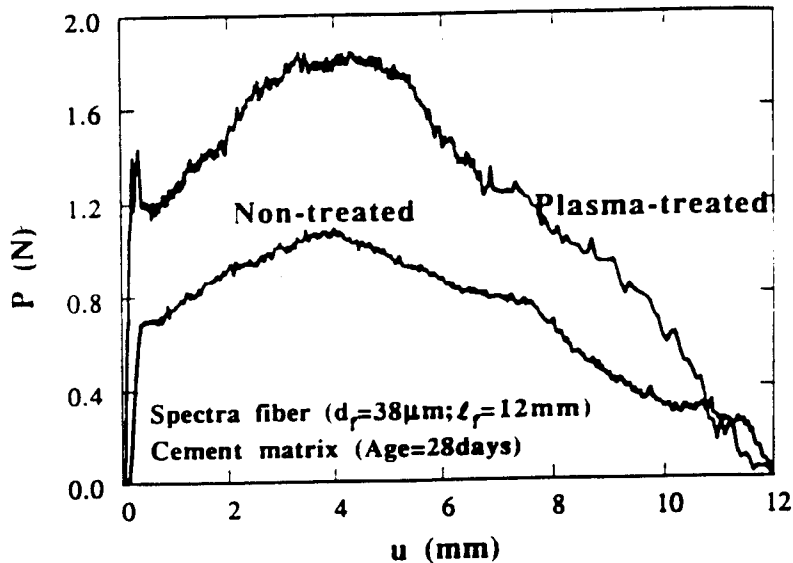

Figure 2 - Pull-out curves of plasma-treated and untreated spectra fibers.

out curves include a linear portion corresponding to the debonding process at the very beginning and a non-linear portion, which covers most of a pull-out curve, representing the pull-out process. The concave-downward shape of the nonlinear branch indicates a slip-hardening behavior of fiber pull-out caused by the abrasion effect. Due to the abrasion effect between fibers and cement matrix, fiber surface is damaged and stripped into small fibrils. These small fibrils in turn contribute to the resistance against the fiber from being pulled out [19,20]. Due to this mechanism, the average frictional bond thus increases with the pull-out distance.

Comparing the features of these pull-out curves in Figure 2, it is obvious that the fiber sample with plasma treatment has a much higher frictional bond and consumes much more energy during the pull-out process than does the non-treated fiber. Figure 3 summarizes the initial frictional bonds from the result of a series of pull-out tests. The average initial frictional bonds are calculated from the load at full debonding which, in this case, refers to the onset of the nonlinear branch of a pull-out curve divided by the
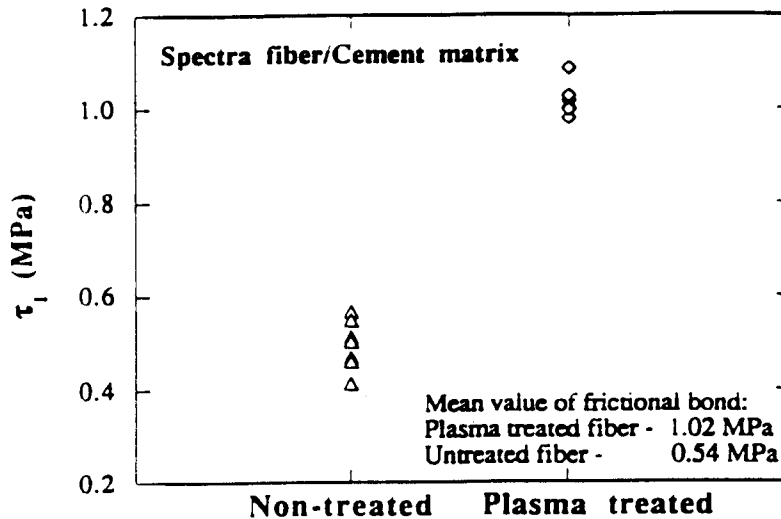
Figure 3 - Effect of plasma treatment on frictional bonds of spectra/cement.

initial fiber/matrix contact area $\pi d_f \ell_f$, where ℓ_f and d_f are the fiber embedment length and fiber diameter, respectively. As shown in the figure, the mean of the frictional bond of non-treated spectra fiber has been enhanced by approximately 100% due to plasma treatment, increasing from 0.54 MPa to 1.02 MPa.

Besides, in the pull-out curve of plasma treated fibers, there is a slight load drop following the fully debonded stage. According to Leung and Li [21], this load drop implies that, in the debonding stage, this particular fiber/cement system has a higher elastic bond strength than the frictional bond, whereas, for the non-treated fiber which does not exhibit such a load drop, the debonding process is basically frictional-control.

According to the result obtained from fiber pull-out tests, the plasma treatment has a definite effect in enhancing the bond property between spectra fiber and cement matrix. Due to the increase in surface reactivity, plasma treated fiber samples exhibit a much higher adhesion to cement material and alters the characteristics of fiber debonding. The frictional bond in the pull-out process is found to be doubled.

6. Composite Properties

The composites under investigation consist of Type I cement (c), silica fume (sf), water (w), and high modulus polyethylene fibers. The mix proportions are as follows: c:sf:w = 1:0.2:0.27 by weight. Short fibers were mixed with other constituents together to form a 3-D reinforcement. The length L_f and diameter of such fibers are 12.7 mm and 38 μm respectively.

Direct tensile tests were performed using coupon specimens of dimensions of 304.8 x 76.2 x 12.7 mm separately. Aluminum plates were glued onto the ends of the

specimens to facilitate gripping. All specimens were four weeks old at testing. The detailed specimen preparation and testing procedure can be found elsewhere [6]. The tensile behavior of composites reinforced with plasma treated and non-treated polyethylene fibers can be determined from these direct tensile tests.

As shown in Figure 4, the effect of plasma treatment on composite tensile properties is clearly demonstrated. For a composite with two volume percent fiber
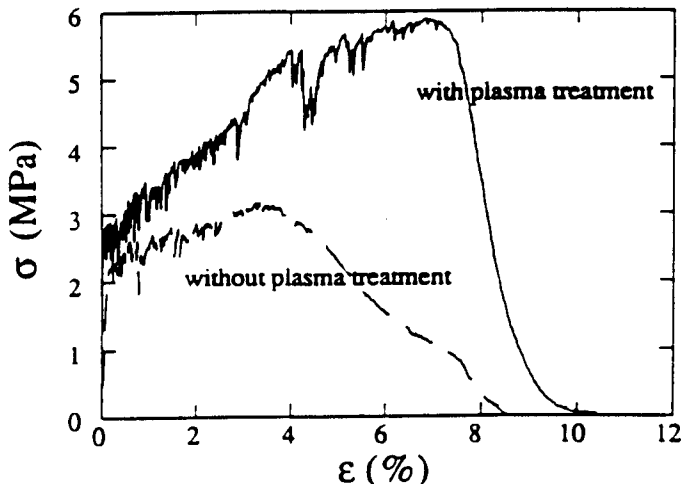

Figure 4 - Tensile stress/strain curves of cement paste reinforced with polyethylene fiber with and without prior argon plasma treatment ($V_f = 2\%$).

reinforcement (non-plasma treated), a pseudo strain-hardening behavior is observed with composite ultimate strength and strain of 3.2 MPa and 3.5%. This behavior is distinctly different from brittle failure of plain cement marix, or quasi-brittle of ordinary fiber reinforced cement composites [6]. The plasma treated fiber composites exhibit even higher composite strength and strain, namely 5.8 MPa and 7%. These represent 81% and 100% increase in strength and strain respectively. The composite ultimate strength is found theoretically proportional to interfacial bond strength [1,22]. Using this theoretical relationship, and information on fiber volume fraction, aspect ratio and experimentally determined ultimate strength, the bond strength was found to be 0.57 MPa in the composite with untreated fibers, and 1.10 MPa in the composite with plasma treated fibers. These numbers are derived from the average of 2 and 5 tests for plasma treated and non-treated fiber composites, respectively. In addition, higher interfacial bond strength contributes to more efficient stress transfer leading to high cracking density (number of cracks per unit length). Hence high composite ultimate strain due to higher bond strength can be expected [23], and is found in this study as shown in Figure 4.

7. Conclusions

In both single fiber pull-out tests and composite tests, plasma treatment of polyethylene fibers was found to be effective in improving interface bond properties between these fibers and a cementitious matrix. In both cases, the plasma treatment leads to an increase of approximately 100% in frictional bond strength. It can therefore

be confirmed that the enhancement in composite properties, namely ultimate strength and strain in such pseudo strain-hardening cementitious composites, derives from plasma treatment of fibers. This is in lieu of using higher fiber volume fraction, or using longer fiber length, which leads to processing difficulties of the composite.

Optimization of bond properties by interface modification, such as the present plasma treatment, can lead to low cost low fiber volume fraction high performance cementitious composites.

Acknowledgements

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