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Effect of Plasma Treatment of Polyethylene Fibers on Interface and Cementitious Composite Properties

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It is well known that interfaces in composites play an important role in determining composite properties. In this paper, preliminary results of the improvement in tensile properties of a fiber-reinforced cementitious composite due to plasma treatment of the discontinuous polyethylene fibers are reported. Specific focus is placed on the pseudo strain-hardening composite properties induced by fiber reinforcements and associated load transfer from crack-bridging fibers to matrix. Single fiber pullout tests support that the composite property improvement is indeed derived from interfacial property enhancement of the plasma treatment process.

I. 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 failure by fast fracture. Fiber reinforcement in brittle matrix composites is therefore directed toward 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, pseudo strain hardening has been demonstrated in real material systems only with continuous aligned fiber reinforcement[1,2] or with large volume content of fibers.[3,4] Both requirements restrict practical applications, due to cost and fabrication obstacles. However, recent advancements in micromechanical theory have led to the design of pseudo strain-hardening cementitious composites with only a few percent by volume of discontinuous fibers.[5–7] The micromechanics are closely related to those for continuous aligned cement[8] and ceramic[9] systems. A variety of fiber types, including steel, polymer, and carbon, have been utilized to generate pseudo strain-hardening cementitious composites with discontinuous randomly oriented fibers. One of the most ductile composites has been designed with 2% of polyethylene fibers. This composite[10,11] has achieved a tensile ductility of 5.6% and a fracture energy of 27 kJ/m². This type of composite is now poised for a variety of applications in the construction and transportation industries.

As for many other composite properties, properties associated with pseudo strain hardening are strongly influenced by fiber/matrix interface characteristics. Recent progress in plasma science and economic plasma generation technology provides a

new tool for fiber surface modification. The use of the plasma treatment system in controlling the interface and therefore the cementitious matrix composite properties is explored in the present work. Preliminary results of mechanical properties on the fiber/matrix interface and composite levels are reported in this paper.

II. Role of Interface

Based on micromechanical theory of matrix crack extension and crack bridging by random discontinuous fibers, Li[7] shows that the fiber content must exceed a certain critical volume fraction, V_f^{crit}, in order for a brittle matrix composite to exhibit pseudo strain hardening under uniaxial tensile loading. This critical fiber volume fraction can be expressed in terms of fiber, matrix, and interface parameters as follows:

$$V_f^{crit} \equiv \frac{12 J_c}{g \tau (L_f/d_f) \delta_0} \tag{1}$$

where J_c is the composite crack tip toughness (and is approximately equal to the cementitious matrix toughness, for low fiber volume fraction), and L_f and d_f are fiber length and diameter, respectively. The snubbing factor g[12] and interface frictional bond strength τ are the parameters which describe the interaction between fiber and matrix. The snubbing factor can be interpreted physically as the increase in bridging force across a matrix crack when a fiber is pulled out at an inclined angle, analogous to a flexible rope passing over a friction pulley. Finally, δ_0 is the crack opening at which the fiber bridging stress σ_0 reaches a maximum, and is given by[12]

$$\delta_0 = \frac{\tau L_f^2}{E_f d_f (1 + \eta)} \tag{2}$$

where $\eta = (V_f E_f)/(V_m E_m)$, V_f, E_f are the fiber volume fraction and elastic modulus, respectively, and V_m, E_m are the matrix volume fraction and elastic modulus, respectively.

When a brittle matrix shows pseudo strain hardening, the ultimate strength of the composite σ_{cu} coincides with the maximum bridging stress σ_0, given by[5,13]

$$\sigma_{cu} = \frac{1}{2} g \tau V_f \left(\frac{L_f}{d_f} \right) \tag{3}$$

It is clear from (1) that increase of bond strength τ will lead directly to a reduction in the amount of fibers needed to create a composite which exhibits pseudo strain hardening. Furthermore, the ultimate strength of such a composite scales linearly with bond strength τ, according to (3). The ductility of the composite is associated with the inelastic strain generated as a result of multiple cracking.[8] This inelastic strain results from the multiple crack density and from the opening of each crack. The multiple crack density is expected to increase with the bond strength τ, which controls the rate of stress transfer from the bridging fiber into the matrix material. However, the "spring" stiffness of the bridging fibers will also increase, leading to a reduction in crack opening for a given bridging stress. Thus, the composite ductility is expected to increase less than

linearly with τ. All of the composite properties discussed above—the failure mode change from quasi-brittle to ductile, the ultimate strength, and the composite ductility—could therefore be modified through control of the interfacial bond strength, while keeping everything else the same. These concepts appear to be confirmed by the experimental results described below.

III. Interphase and Interface

In fiber-reinforced composites, there are two different interfacial failure modes when fibers are pulled out from the matrix. Adhesive failure occurs following the actual interface between fiber and matrix, whereas adherent failure takes place within the matrix. For fiber-reinforced cement-based composites, a distinctive layer of interphase or transition zone is typically observed.[14,15] This zone, extended from 50 to 100 μm away from the interface, 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.[15] 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 in removing the porous transition zone,[15,16] sometimes referred to as a process of "densification." Depending on fiber types and matrix constituents, either adhesive or adherent failure can occur in fiber-reinforced cementitious composites. Hence, proper strategy should be employed toward enhancing interfacial bonds or strengthening the interphase zone. In our previous investigation,[17] the polyethylene and steel fibers used in our tests were found to exhibit adhesion-type failure in the cement matrix. Clean fiber surfaces with little or no matrix residue after being pulled out are observed. This implies that interphase densification may not be effective in such composites.[17,18] For these composites, therefore, research should be directed toward improving bond strength through fiber surface modification to enhance adhesive strength.

IV. Fiber Surface Modification by Plasma Treatment

Interface property modification can be achieved by a variety of techniques. These include fiber deformation techniques such as twisting, crimping, pitting, or button-end creation, surface coating such as use of coupling agents, and fiber surface modification techniques such as chemical oxidation, corona treatment, and plasma treatment. These surface modification techniques are directed toward 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.[19]

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.[19] 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.[19,20] 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 suitable 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.[21–24] 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 provide further improvement. In addition to excellent adhesion, plasma-treated fibers also exhibit significantly enhanced pullout energy in single-fiber pullout tests.

Table I. Fiber Parameters

L_f (mm)	d_f (μm)	E_f (GPa)	V_f (%)	g
12.7	38	120	2	2

In the present research, two types of specimens were prepared. Coupon composite specimens were prepared to study uniaxial tensile response of the composite. Direct pullout specimens were prepared to study interfacial bond properties. A radio frequency discharge plasma surface treatment system is utilized to create the plasma for treatment of high-modulus polyethylene fibers. For the discontinuous fibers used in the composite test, the fiber bundles were treated in a Harrick PDC-3XG plasma cleaner. Limited by the small chamber size, only a small quantity (6.5 g) of fibers can be treated at a time. Three types of gases—Ar, CO_2 and NH_3—were employed at an initial pressure of 480 mtorr ($\approx 6.38 \times 10^2$ Pa) and a power level of 58 W. Fiber treatment time was 5 min and applied at flow rates of 50, 40, and 40 mL/min for the Ar, CO_2, and NH_3, respectively. Subsequently, the treated fibers were mixed with cement paste to form the composite in a manner identical to those without the plasma treatment.

For the continuous fibers used in the pullout specimens, a plasma treatment system manufactured by AIRCO/Plasma Science (Model PS 300) with a large chamber size (in comparison to the Harrick system) was used. Only Ar gas was employed, at a flow rate of 40 mL/min, initial pressure of 480 mtorr, and power level of 58 W. Treatment time was also 5 min. To ensure consistent treatment condition, a precise amount, 10 g in weight, of fibers was treated for each batch of process. The process of plasma treatment was followed immediately by specimen casting. The exposure of the treated fiber to air was controlled to be minimal to avoid possible reactions that the air might have to change the surface conditions of the treated fibers.
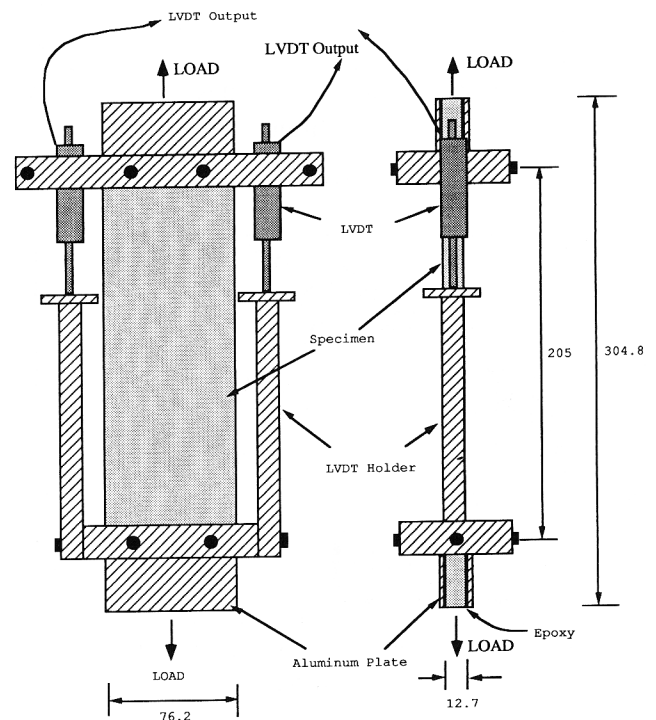
Fig. 1. Setup for direct tensile tests.

V. Composite Tensile Tests

The composites under investigation consist of Type I portland cement (c), silica fume (sf), water (w), and the treated and nontreated high-modulus polyethylene fibers. The mix proportions are c:sf:w = 1:0.2:0.27 by weight. Short fibers were mixed with other constituents to form a 3-D random reinforcement. Fiber parameters are given in Table I. The snubbing factor g has not been measured for the polyethylene fiber used in the present work, but is considered to be similar to that of other polymeric fibers. For example, Li et al.[12] published limited data on polypropylene ($g = 1.78$) and nylon fiber ($g = 2.31$), both in cement matrices. In the following, a value of $g = 2$ has been assumed. The bond strength deduced will change if the actual snubbing factor value is different. It has also been assumed that g is not affected by plasma treatment.

Direct tensile tests were performed separately, using coupon specimens of dimensions of 304.8 mm × 76.2 mm × 12.7 mm. Aluminum plates were glued onto the ends of the specimens to facilitate gripping. All specimens were 28 days old at testing. The detailed specimen preparation and testing procedure can be found elsewhere.[25] A schematic diagram of the uniaxial tensile specimen is shown in Fig. 1. The tensile behavior of composites reinforced with plasma-treated and nontreated polyethylene fibers can be determined from these direct tensile tests.

As shown in Fig. 2(a), a pseudo strain-hardening behavior is observed with composite ultimate strength and strain of 3.6—5.1 MPa and 5.5%. This behavior is distinctly different from brittle failure of plain cement matrix, or quasi-brittle failure of ordinary fiber-reinforced cement composites.[11,25] The plasma-treated fiber composites (Figs. 2(b–d)) exhibit even higher composite strength and strain. For Ar-plasma treatment, the composite ultimate strengths for three specimens are between 4.7 and 5.8 MPa. For CO_2-plasma treatment, the resulting strengths are from 5.2 to 5.9 MPa. For NH_3-plasma gas, the resulting strengths are 5.5 and 5.8 MPa for two specimens (Table II). The strain capacity at ultimate strength increases slightly from 5.5% for the nontreated fiber composite to 5.8%, 5.8%, and 6.75%, for the Ar, CO_2, and NH_3 plasma-treated fiber composites.

The theoretical relationship between composite ultimate strength and interface bond strength can be used to deduce the interfacial properties. Using Eq. (3) and information on fiber volume fraction, aspect ratio, and experimentally determined ultimate strength, the averaged bond strength was found to be 0.63 MPa in the composite with nontreated fibers, and 0.76, 0.83, and 0.85 MPa in the composite with Ar, CO_2, and NH_3 plasma-treated fibers, respectively. These represent bond strength improvement of 21%, 32%, and 35% for the Ar, CO_2, and NH_3 plasma-treated fibers over the nontreated fiber.

VI. Fiber Pullout Tests

To confirm that the composite properties are improved as a result of fiber/matrix interface bond improvement by the plasma
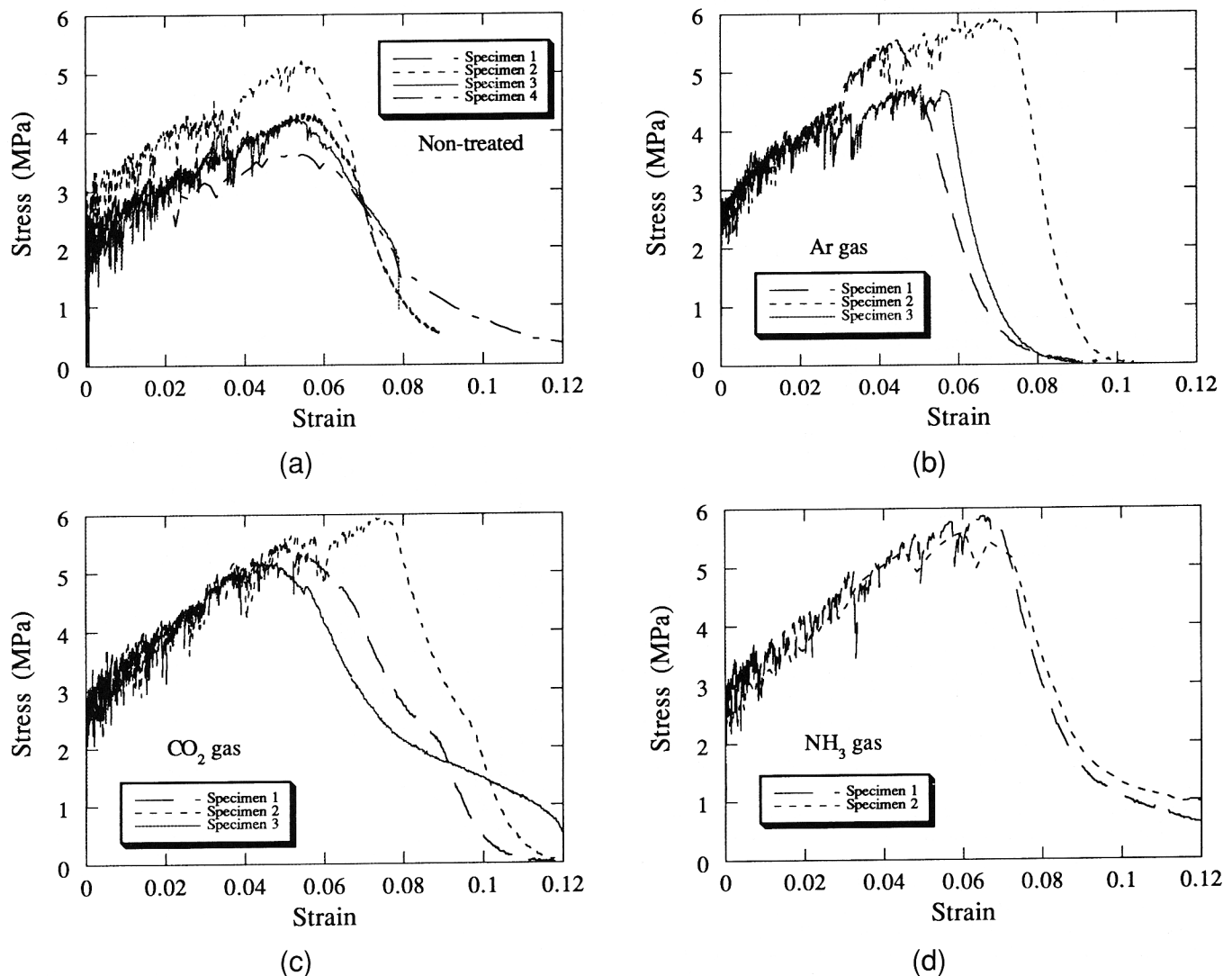

Fig. 2. Tensile stress/strain curves of cement paste reinforced with polyethylene fiber with (a) no treatment, (b) Ar plasma treatment, (c) CO_2 plasma treatment, and (d) NH_3 plasma treatment.

Table II. Composite Ultimate Strength and Deduced Bond Strength for Various Plasma-Gas Treatments

Plasma treatment	σ_{cu} (MPa)	τ (MPa)	Average τ (MPa)
Virgin	5.1	0.76	0.63
	4.1	0.61	
	4.2	0.63	
	3.6	0.54	
Ar	5.8	0.82	0.76
	4.7	0.70	
	4.8	0.72	
CO_2	5.5	0.82	0.83
	5.9	0.88	
	5.2	0.78	
NH_3	5.8	0.87	0.85
	5.5	0.82	

treatment, fiber pullout tests were conducted by pulling individual fibers out of cement matrix bases. The test setup and specimen configuration of the fiber pullout test are shown schematically in Fig. 3. A fiber sample is partially embedded in the dog-bone shaped 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 may occur between the grip and the fiber. The pullout 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 crosshead 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 crosshead 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 pullout tests.[26]

In the preparation of the test samples, specimens were demolded 24 h after casting and were cured in a water tank until testing. Fiber pullout tests were conducted at the age of 28 days, the same age as the composite specimens. The matrix used was identical to that used in the composite tensile specimens described above. Additional tests were carried out using a less
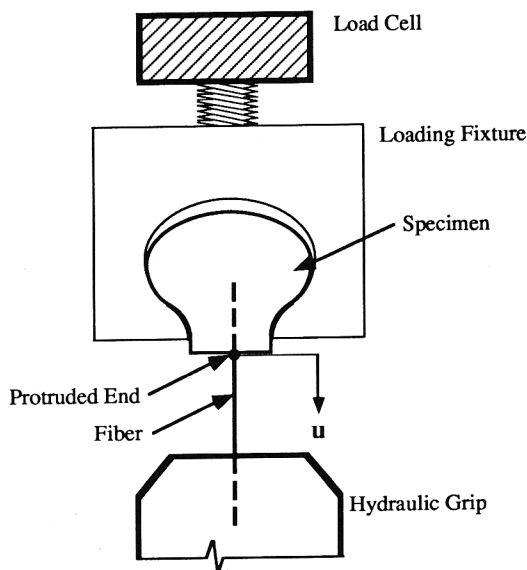
dense matrix (Type I portland cement with $w/c = 0.4$, with no silica fume). Test results were almost the same as for the denser matrix, suggesting that transition zone densification was not effective in improving the bond strength, which is controlled by adhesive failure in this material system.

Figure 4 shows typical P–u curves from pullout tests of plasma-treated and nontreated high-modulus polyethlene fibers. Fibers have an embedment length of 12 mm. Generally, the pullout curves include a near-linear portion corresponding to the debonding process at the very beginning and a highly nonlinear portion, which covers most of a pullout curve, representing the pullout process. The concave-downward shape of the nonlinear branch indicates a slip-hardening behavior of fiber pullout caused by the abrasion effect. Due to the abrasion effect between the fibers and cement matrix, the 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.[27,28] Due to this mechanism, the average frictional bond thus increases with the pullout distance. In addition, in the pullout curve of plasma-treated fibers, there is a slight load drop following the fully debonded stage. According to Leung and Li,[29] 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 nontreated fiber which does not exhibit such a load drop, the debonding process is basically frictional control.

Comparing the features of these pullout curves in Fig. 4, it is obvious that the fiber sample with plasma treatment has a much higher frictional bond and consumes significantly more energy during the pullout process than does the nontreated fiber. Figure 5 summarizes the initial frictional bonds from the result of a series of pullout 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 pullout curve divided by the 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 nontreated high-modulus polyethylene fiber has been enhanced by approximately 100% due to plasma treatment, increasing from 0.54 to 1.02 MPa. For comparison, the frictional bond strengths deduced from the composite ultimate tensile strength are also included in this figure.

VII. Further Discussions and Conclusions

According to the experimental results obtained from both single-fiber pullout tests and composite tensile tests, plasma treatment of high-modulus polyethylene fibers was found to be effective in improving interface bond properties between these fibers and a cementitious matrix. Plasma-treated fiber samples exhibit a much higher adhesion to cement material and profound slip-hardening characteristics of fiber debonding. It is

Fig. 3. Single-fiber pullout test setup.
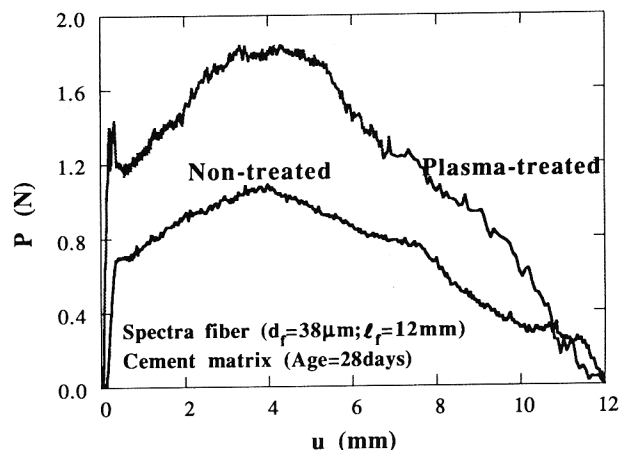
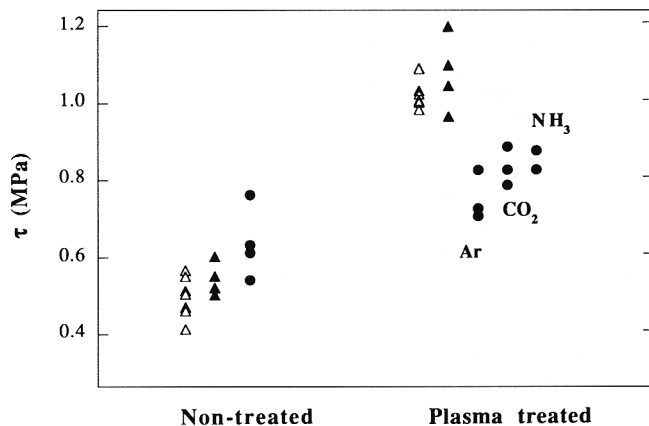**Fig. 4.** Pullout curves of plasma-treated and nontreated spectra fibers.

Fig. 5. Effect of plasma treatment on frictional bonds of spectra fiber/cement. Triangular symbols are bond strength deduced from pullout test; circular symbols are bond strength deduced from composite tensile test; open symbols indicate cement matrix without silica fume.

confirmed that enhancement in composite properties, namely, ultimate strength and strain in pseudo strain-hardening cementitious composites, is achievable from interface property modification as a result of 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.

In examining Fig. 5, among the three gas types used in this experimental program of the composite tests, it appears that the NH_3 plasma, with a 35% improvement in bond strength, provides the most effective treatment. Yet, in comparison to the data obtained from the direct pullout tests, this bond strength improvement for the treated discontinuous fiber bundle is only a fraction of that for the treated continuous fibers. Several possible sources for this discrepancy in magnitude change in bond strength can be traced. The continuous and discontinuous fibers were treated in different plasma treatment systems, even though conditions of treatment were kept to be as similar as possible. In particular, the small size of the chamber in the Harrick treatment system requires the fiber bundles to be packed together more tightly than the continuous fibers. Since the plasma penetration depth is limited, it is possible that the discontinuous fiber bundles were only partially treated, with only those fibers on the surface exposed to and receiving treatment from the plasma gas. In addition, the fiber bundles were used in the composite specimens with a general time delay after treatment of 30 days or more. In contrast, the continuous fibers were employed in the pullout test specimens immediately after plasma treatment. These considerations suggest that the composite test results (Figs. 2(b–d)) can be further improved, if plasma treatment conditions for the fiber bundles are more carefullly controlled.

Figure 5 also indicates that, for the untreated fibers, the bond strengths interpreted from the composite tests were higher than that from the pullout tests. The reason is not yet clear. The discrepancy may arise from nonuniform fiber dispersion in the composite or from improper value of the snubbing factor used in Eq. (3) to interprete bond property from composite ultimate tensile strength. For the latter case, and for consistency with the bond strength deduced from the pullout test, the snubbing factor g needs to be scaled up to 2.6 instead of 2. The bond strength interpreted from the composite tests for the treated fibers would correspondingly be scaled down by this same factor (2/2.6 = 0.77). However, the conclusion regarding the percentage improvement in bond strength due to plasma treatment with the different gas does not change.

This paper demonstrates, with preliminary data, that improvement of bond properties due to fiber surface modification by plasma treatment can be an effective means of achieving low-cost, low-fiber volume fraction, high-performance cementitious composites. The enhancement of composite ultimate strength and strain reported in this paper is expected to further promote the damage tolerance, which has been found to be superior for this brittle matrix composite.[10,11] However, further research in optimization of processing conditions in plasma treatment of the reinforcing fibers, now ongoing at the University of Michigan, is necessary to derive the full benefit of this technology.

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