Plasma Treatment of Polymeric Fibers for Improved Performance in Cement Matrices

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Received 4 June 1999; accepted 23 August 1999

ABSTRACT: The surfaces of collated fibrillated polypropylene fibers and monofilament polyolefin fibers were treated by low-temperature cascade arc plasma with different gases to study the effect of interface treatment on the mechanical performance and toughening in fiber-reinforced concrete composites. Results from static flexural tests conducted in a four-point configuration on 17 concrete mixes including one unreinforced control mix, 4 mixes with untreated fibers (two volume contents for each of two fiber types—fibrillated and monofilament), and 12 mixes with plasma-treated fibers (two volume contents, above two fiber types, and three plasma treatments) are presented and discussed. It is concluded that plasma treatment of polymeric fibers is effective in improving the flexural performance and toughness of fiber reinforced concrete composites. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 76: 1985–1996, 2000

Key words: fiber reinforced concrete; flexural toughness; interface properties; plasma treatment; polyolefin; polypropylene

INTRODUCTION

The incorporation of short discrete fibers in concrete results in improved resistance to fracture, fatigue, and impact loading.[1] Fibers contribute to the toughening of the resultant composite largely through interface stress transfer. Steel and synthetic fibers are among the common fiber types used in fiber-reinforced concrete. The interface characteristics of steel fibers are typically enhanced by providing mechanical anchorages such as hooked or enlarged ends, or by incorporating deformations/indentations along the fiber length.[1] Attempts have been made to enhance bond between polymeric fibers and cement matrices through mechanical means such as fibrillation and indentation with some success. Surface treatments offer alternate means of enhancing the interface characteristics of such fibers.[2–4] Use of plasma treatment to alter the characteristics of the fiber–matrix interface is among the relatively recent and potentially promising techniques to enhance the mechanical performance of fiber reinforced composite systems.[3,4] The ionized gas in the plasma chamber bonds to the fiber surface, thus altering the interface characteristics of the fiber. The effectiveness of plasma treatment in numerous scientific and engineering applications has been successfully demonstrated.[5]

Polymeric fibers are popular for reinforcing concrete matrices because of their low density (more number of fibers for a prescribed volume fraction), high tensile strength, ease of dispersion, relatively low cost compared to other fiber types,

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Contract grant sponsor: National Science Foundation; contract grant number: NSF-CTS-9400725.

Journal of Applied Polymer Science, Vol. 76, 1985–1996 (2000)

and their relative resistance to chemicals. Polypropylene and Polyolefin fibers are typically hydrophobic, resulting in a relatively poor bond with concrete matrices compared to some other types of fibers. Treatment of polypropylene with an aqueous dispersion of colloidal alumina or silica and chlorinated polypropylene enhances the affinity of these fibers towards cement particles. Treatment of polypropylene fibers with a surface active agent provides better dispersion of the fibers and a stronger bond between cement and fiber. The earlier attempts at surface treatments of polypropylene fibers have only had limited success, and have not been commercially attractive. The present investigation is aimed at studying the effectiveness of plasma treatment of polypropylene fibers in enhancing the flexural performance of polymeric fiber-reinforced concrete systems.

BACKGROUND INFORMATION

The effectiveness of polymeric fibers as concrete reinforcement depends upon the mechanical bond between the fiber and cement matrix. A mechanical bond or adhesion with calcium silicate hydrate for polypropylene fiber concrete has been reported.[6] The mechanical bond of collated fibrillated polypropylene is typically better than that of the monofilament polypropylene fibers, because cement matrix penetrating the fibrillated network anchors the network in the matrix.[7] The network structure of fibers leads to bidirectional action of fibers after the shearing action of aggregate particles during mixing. Fibrillated fibers have higher tensile strength and modulus of elasticity than films. The fiber geometry and type, fiber volume content, fiber configuration, fiber length, the modulus of elasticity and Poisson's ratio of the fiber each have a significant effect on the overall interface performance of the fiber in a cement-based matrix.

Denes et al.[3] have studied the influence of $SiCl_4$ plasma-activated polypropylene fibers and noted that regardless of the hydration period, composites made with plasma-treated polypropylene fibers have better flexural strength and toughness than those made with untreated fibers. Geometric details of the fibrillated polypropylene fibers used are not reported. Their test results show a reduction in flexural strength with increased fiber volume fraction for both the treated and untreated fiber composites. This perhaps can be attributed to difficulties in mixing larger quantities of fibers and a matrix-dominated flexural strength, both typical in such composites. If lower fiber volume fraction is used in conjunction with plasma treatment, it may be possible to limit fiber contents to levels that do not adversely affect mixing operations, and as a result, composite performance, as demonstrated later, based on the results from this investigation.

Li et al.[4] have reported results from tensile and pull-out tests on plasma-treated polyethylene fiber concrete composites ($l = 12.7$ mm, $d = 38$ μm, $V_f = 2\%$). They note that of the three treatments (NH_3, CO_2, and Ar) investigated by them, NH_3 plasma treatment provided the best improvement in bond strength (up to 35% over untreated fiber composites). From these earlier studies[3,4] and from the results of this investigation it appears that different treatments may be appropriate for different types of polymeric fibers.

Among the numerous ways to generate plasma, the cascade arc plasma technique is the most attractive one, largely because of its practical simplicity.[5] Cascade arc plasma comprises one or more sprays of controllable flux or beams of ionized gases, which are directed towards the surface of the polymeric fiber substrate. The low-temperature process that can be applied to many types of materials has a rapid deposit rate, even on a wide substrate. Operated at atmospheric or super atmospheric pressures the cascade arc was first used in a high-temperature mode. The plasma flux coming out from the cascade arc plasma column cools down upon expansion in a vacuum chamber, providing a low-temperature plasma source. Additional details on plasma polymerization are available in ref. 5.

EXPERIMENTAL PROGRAM

Details of the Experimental Program

A total of 17 mixes were tested, including one unreinforced concrete mix, four untreated fiber reinforced concrete mixes, and 12 treated fiber-reinforced concrete mixes, as detailed in Table I. A total of four replicate flexural specimens were tested for each mix. Closed-loop static flexural tests were conducted, with midspan specimen deflection as the feedback parameter used to control the test. Properties of the plain and untreated

Table I Details of the Experimental Program Showing Numbers of Flexural Specimens Tested for Each Series

Deatils of the Concrete Mix	V_f	Untreated	Ar	$CH_4 + O_2$	Air
Plain concrete mix	0%	4	—	—	—
Fibrillated polypropylene mix	0.25%	4	4	4	4
Fibrillated polypropylene mix	0.50%	4	4	4	4
Monofilament polyolefin mix	0.25%	4	4	4	4
Monofilament polyolefin mix	0.50%	4	4	4	4

fiber polypropylene reinforced concrete mixes were obtained from closed-loop circumferential strain-controlled cylindrical specimens.

Plasma Treatment

Fibrillated polypropylene and monofilament polyolefin fibers were plasma treated using a cascade arc plasma polymerization apparatus developed by Surface Science and Plasma Technology Center (SSPTC), University of Missouri–Columbia. Argon was used as the carrier gas. Methane, oxygen, and air were used as the reactive gases. In the first series, only Argon plasma (Ar) was used for the interface treatment. Methane and oxygen were added as reactive gases ($CH_4 + O_2$) in addition to the Argon (carrier gas) in the second series. In the last series, air was added as the reactive gas. Plasma treatment was undertaken in a steel chamber, which was first evacuated to less than 1 mTorr, by a combination pump. A power level of 240–280 W and an Argon flow rate of 1000 mL (STP)/min was used. The pressure in the steel chamber was maintained at 270 mTorr (36 Pa). Four grams of fibers were treated for 6 min on a rotating plastic plate (Fig. 1). Treated fibers were stored in water until they were mixed in concrete. This was necessary to ensure that there was no migration of hydrophilic moieties from the surface to the interior of the bulk fiber. Water absorption by the fibers was less than 0.02%, and did not adversely affect the concrete mix design. Methane and oxygen flow rates of 30 mL/min and 10 mL/min, respectively, were used in the second series. An air flow rate of 40 mL/min was used in the third series.

Contact Angle Measurements and Wettability of Fibers

The contact angle between a water droplet and fiber surfaces were measured before and after plasma treatment to investigate the wettability of the fibers subjected to the various surface treatments. Table II lists the contact angles measured in each case.

The contact angle test is a good measure of the hydrophobic or hydrophilic nature of substrate materials such as fibers. If the contact angle is smaller than 90°, the fiber is hydrophilic, while, if this angle is larger than 90°, then the fiber is hydrophobic (Fig. 2). As can be observed from Table II, the untreated fiber, for both fiber types, is hydrophobic. Plasma treatment in all cases make fibers of both types hydrophilic. The potential influence of the change in wettability on the mechanical performance of composites made using plasma-treated fibers is discussed later in the Results section.

Specimen Fabrication

Type I Portland cement, crushed limestone with a maximum aggregate size of 1″ (25.4 mm) and washed river sand meeting ASTM C 33 gradation specifications were used for fabricating the flexural specimens. Two-inch–long fibrillated polypropylene fibers (50 mm length, 25 μm film thickness—supplied by Fibermesh USA) and 2″-long (50 mm length, 0.63 mm diameter) polyolefin monofilament fibers (supplied by 3M Company) were used in the investigation.

Mix proportion used for all batches was: c : a : s : w : 1.0 : 2.55 : 1.35 : 0.38 (weight ratios). Fibers were added in the prescribed volume fractions detailed in Table I. All batches were mixed in horizontal pan mixer. Coarse and fine aggregates were dry mixed first. Half of the mixing water and cement were added next. The rest of the mixing water was added gradually with further mixing. Fibers were dispensed manually while the mixing continued. Uniform fiber dispersion with no significant balling was ensured. Concrete was poured into slab molds (26 × 16 × 4 in, 660 × 406

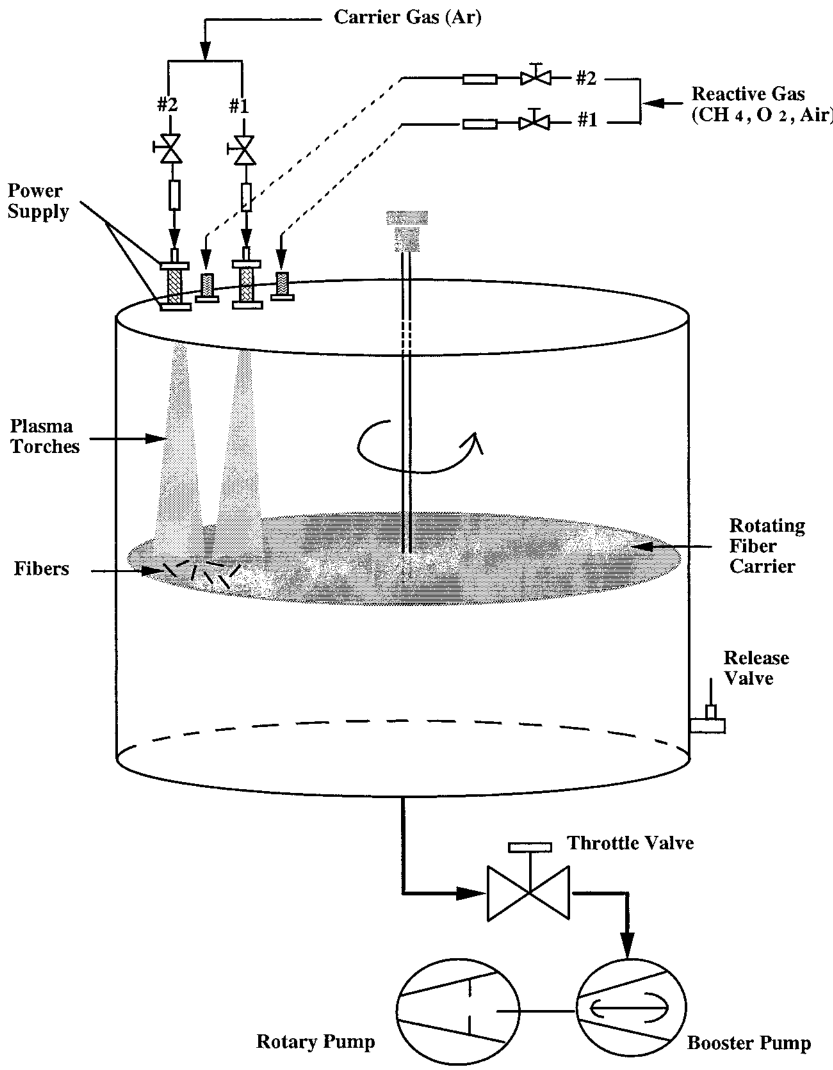


Figure 1 Schematic of the low-temperature cascade-arc plasma reactor.

× 102 mm), consolidated using a vibrating rod, finished, and covered with polyethylene sheets. The slabs were demolded after 24 h, at which time they were placed in a curing room (75°F, 98% RH). Individual flexural specimens were sawed just prior to testing at around 28 days.

Flexural Testing

The toughening resulting from incorporation of short randomly oriented fibers in concrete matrices[8] is characterized using a four-point flexural test (Fig. 3), such as the ones recommended by ASTM[9] and JCI.[10] The area under the load–deflection curve up to prescribed postcracking deflection limit represents the energy absorbed by the specimen, and is used to compute nondimensional toughness indices, residual strength indices, and equivalent flexural strength. Total load, P, net- and gross-deflection, δ_n and δ_g, respectively, at midspan were monitored and recorded for the flexural test.[8] Specimens were loaded at a midspan deflection (gross deflection) rate of 0.004 in/min, as recommended in ASTM C-1018.[9] Tests were conducted until a δ_g value of l/150 per the JCI toughness standard,[10] where L is the outer span in the four-point flexural test (Fig. 3). A PC-based data acquisi-

Table II Contact Angles Measured for Various Fiber and Treatment Combinations

Fiber Type	Untreated	Ar	$CH_4 + O_2$	Air
Fibrillated	98°	78°	58°	33°
Monofilament	108°	70°	62°	53°

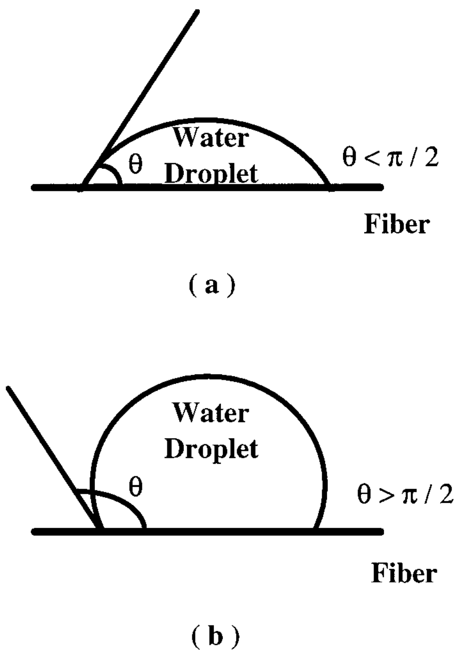


Figure 2 Schematic showing wettability of the fiber surface: (a) hydrophilic surface, and (b) hydrophobic surface.

tion system was used to acquire and store data from the flexural tests.

The ASTM C-1018[9] defines nondimensional toughness indices, I_5, I_{10}, I_{20}, and I_{30}, as ratios of the area under the load–deflection response up to 3 δ_f, 5.5 δ_f, 10.5 δ_f, and 15.5 δ_f, respectively, where δ_f, is the deflection at first-crack to the area under the load–deflection response up to δ_f (first-crack toughness, T_f).[8,9,11] For a material exhibiting elastic-ideally plastic behavior, indices I_5, I_{10}, I_{20}, and I_{30} take the values of 5, 10, 20, and 30, respectively. Values of these indices are useful in making qualitative judgments on the approximate shape of the postcracking load–deflection response of fiber–cement composites or in making relative comparisons. The JCI uses two toughness measures,[8,10,11] one based on absolute energy absorbed by a standard size specimen, T_{JCI}, and another based on equivalent flexural strength, σ_b. T_{JCI} is computed as the area under the load deflection response up to a limiting deflection of $L/150$ (δ_{limit}). σ_b represents the equivalent flexural strength at δ_{limit}, and is computed as T_{JCI} $L/\delta_{limit}bd^2$, where b, d, and L equal the width, the depth, and span of the test specimen. Even while δ_{limit} may be arbitrary and large for many practical applications, T_{JCI}, provides a sensitive toughness measure that can distinguish the influence of fiber reinforcing parameters and efficiency.[8,11]

RESULTS AND DISCUSSION

Results from compression tests performed on plain concrete and the untreated fiber mixes are presented in Table III. These results are useful for standard comparisons of the mixes. The effect of plasma treatment of fibers on the compressive properties was not studied. These tests were conducted on 3‘-diameter cylindrical specimens cored

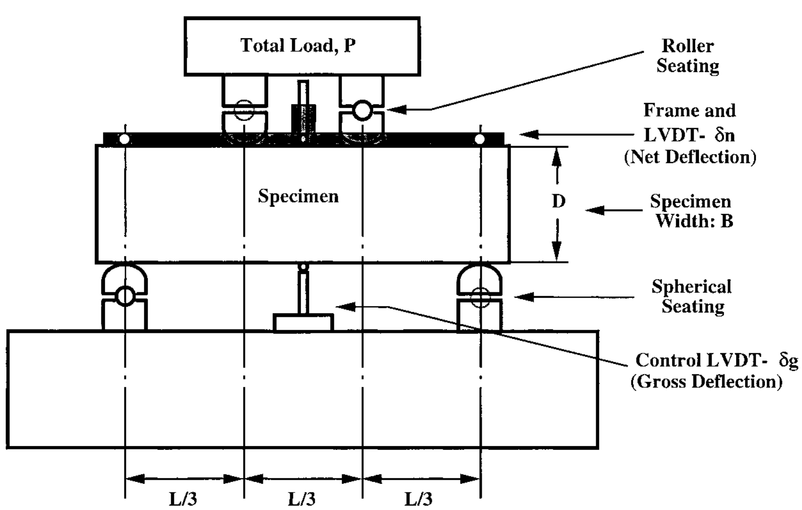


Figure 3 Closed-loop flexural test setup.

Table III Results from the Compression Test[a]

Mix Type	Compressive Strength f_c', (psi)[b]	Elastic Modulus, E_c, (ksi)[b]
Plain concrete	5536	3963
0.25% Fibrillated polypropylene	5120	4377
0.5% Fibrillated polypropylene	6157	4191

[a] Each entry represents an average of three or four specimens.
[b] 1 MPa = 145 psi, 1 Gpa = 145 ksi.

from slabs cast for the test program. The specimens were nominally 6‘ long. The tests were conducted using a closed-loop testing machine with circumferential strain as the feedback parameter used to control the tests. Results reported are for the fibrillated polypropylene fiber mixes. Similar tests were not conducted for the untreated monofilament polyolefin fiber mixes. Average compressive strength of the unreinforced mix, the mix with 0.25% fibers, and the 0.5% fiber mix had compressive strengths of 5536 psi (38.18 MPa), 5120 psi (35.31 MPa), and, 6157 psi (42.46 MPa), respectively.

Figure 4 includes flexural load–deflection responses of plain concrete and untreated fibrillated [Fig. 4(a)] and monofilament polyolefin [Fig. 4(b)] fiber mixes. The inset in each case includes plots of normalized load–deflection responses, where the load values are normalized with respect to corresponding peak load and the deflection values are normalized with respect to the deflection values at peak load. At the small fiber volume contents used (0.25 and 0.5%), composite flexural strength is obtained at first crack load. The flexural strength of the fiber composites is marginally smaller than that measured for the unreinforced concrete. This is attributed to the fact that mixing and consolidation of the fiber mixes is typically more difficult than that of the plain concrete mix. The insets clearly demonstrate that for the low fiber dosage rates used, the prepeak performance is matrix dominated, while the postpeak performance is influenced largely by the fiber parameters. Residual postcracking load capacity at any prescribed deflection level is larger for the mix with a higher fiber volume fraction. The fibrillated polypropylene fibers that have an effective aspect ratio approximately 1150 (length 50.8 mm, equivalent fibril diameter 44 μm) performed marginally better in the post-cracking regime compared to the monofilament polyolefin fibers, which had an aspect ratio of 81 (length 50.8 mm, fiber diameter 630 μm). It is also relevant to note that the mixing process opens up the fibrillated fibers to effectively provide significantly larger surface area for interface bonding. In automated BET absorption tests conducted on fibrillated fibers before mixing and after mixing (fibers sampled from the wet mix before final placement into the molds), a fourfold increase in fiber surface area was noted (from 14.7 m^2/g to 55.7 m^2/g). The same test on the monofilament polyolefin fibers showed no significant change in surface area due to mixing operations (0.005 m^2/g).

Because the fibers used have a elastic modulus value in the range 1–1.5 $\times$ 10^6 psi, and because only low fiber volumes were studied, matrix cracking is followed by an immediate drop in the load-carrying capacity. At deflections larger than at the peak, residual strength is realized through frictional pullout of fibers from the matrix. This action is fiber dominated, and can be observed from the inset to Figure 4, where larger fiber volumes sustain higher residual postcracking stresses.

Figures 5 and 6 show typical load–deflection plots for fibrillated polypropylene and monofilament polyolefin mixes, respectively, where the influence of plasma treatment is demonstrated. Load–deflection responses from untreated fibers as well as treated fibers suggest that, from among the various treatments investigated, plasma treatment with Argon (Ar) results in the best "overall" performance of the treated fiber mix. This is true for both fiber types used as well as the fiber volume fractions investigated. Treatments in air and in methane plus oxygen result in smaller residual strengths, in that order, compared to Argon. Figure 7 shows plots of the ranges of first crack strength obtained for all the specimens investigated. The scatter diagram for first crack strength, which also represents the peak load-carrying capacity of the specimen, suggests that although Argon is the best treatment for the fibrillated polypropylene fiber type, Ar treatment is marginally superior for the monofilament polyolefin fibers. These observations, along with relative values of the contact angles (Table II) characterizing the wettability of the fiber surface, suggests that

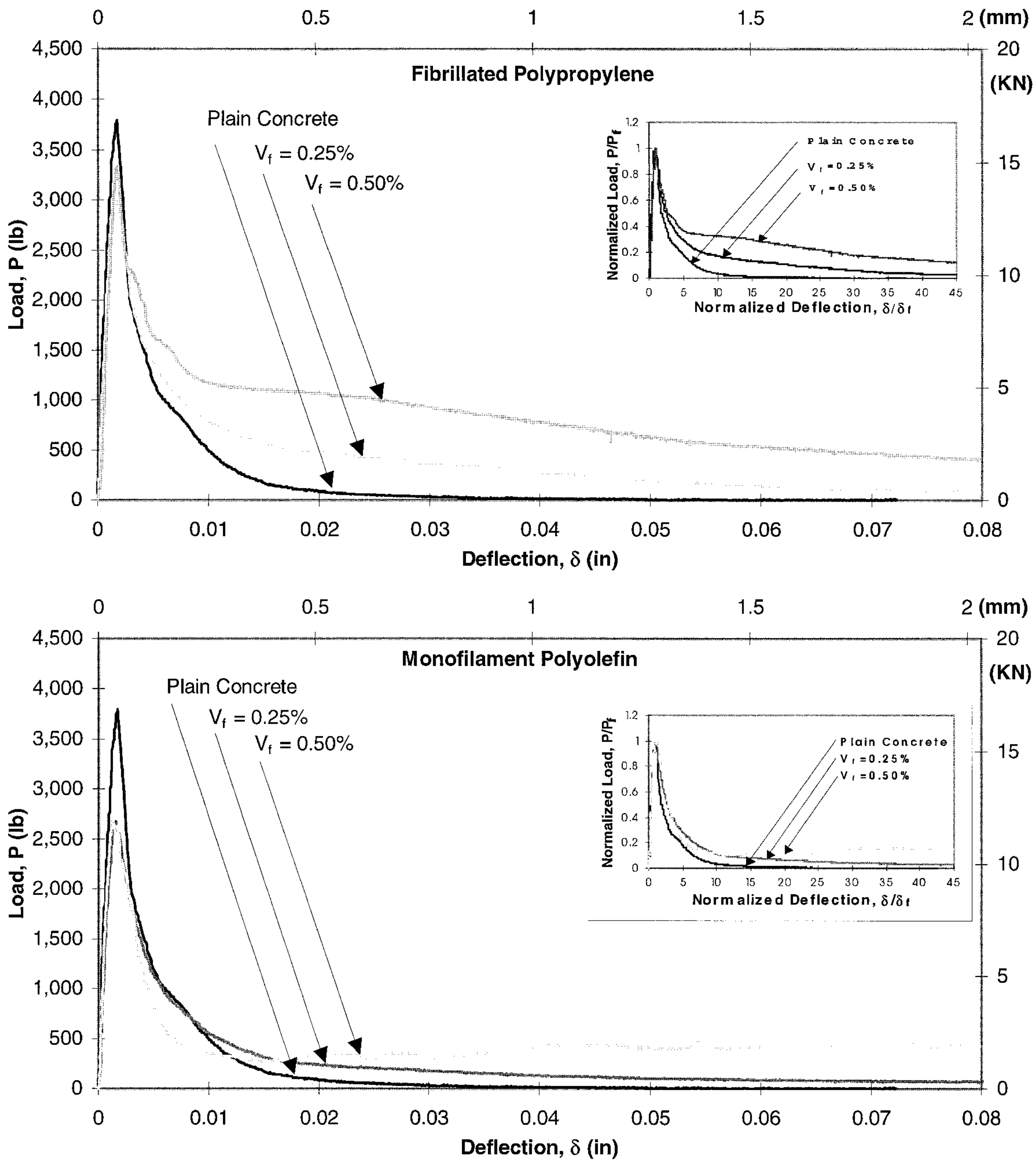
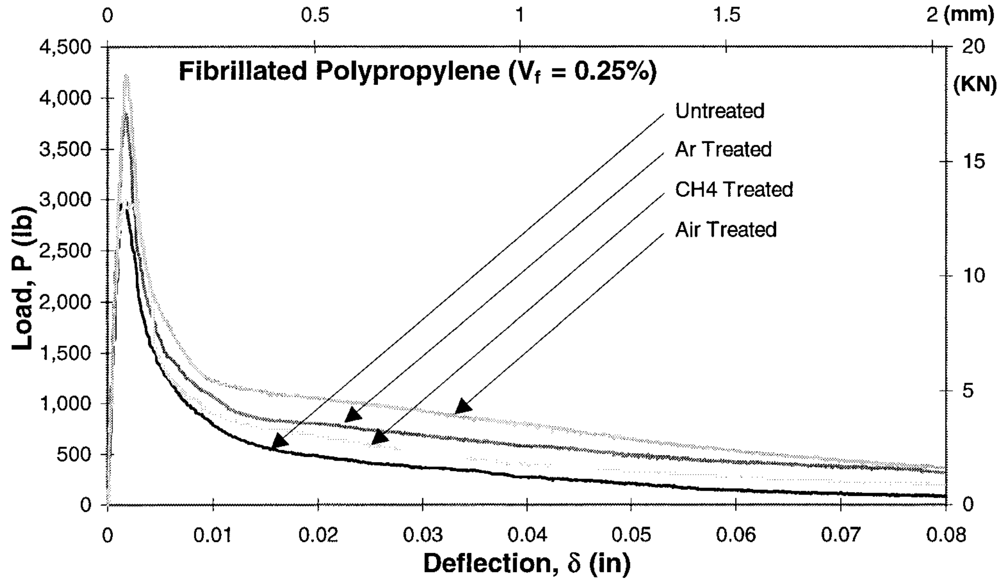
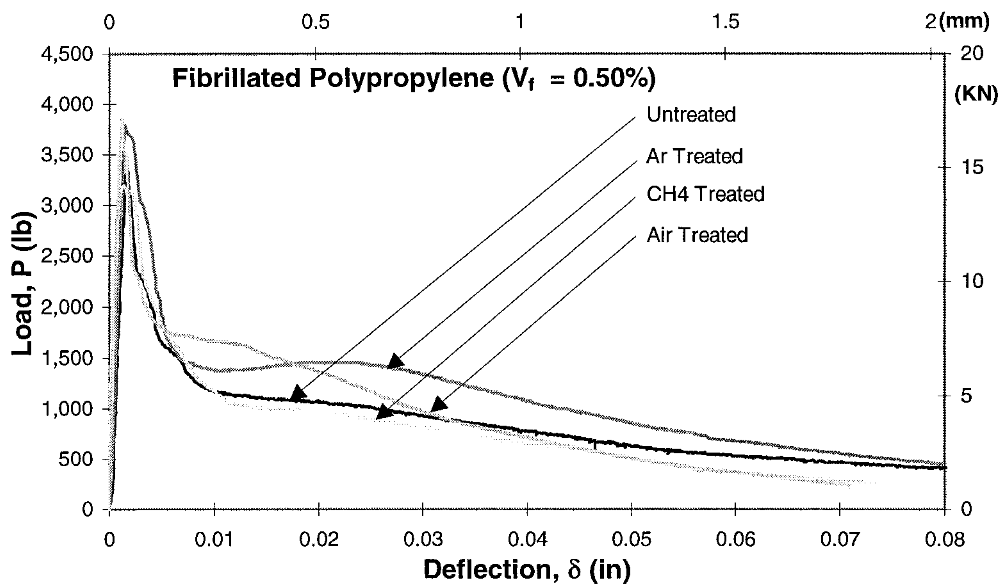


Figure 4 (a) Typical load–deflection responses of untreated fibrillated polypropylene fiber-reinforced composites. Inset shows load–deflection behavior where both load and deflection are normalized with respect to their values at the peak load. (b) Typical load–deflection responses of untreated monofilament polyolefin fiber-reinforced composites. Inset shows load–deflection behavior where both load and deflection are normalized with respect to their values at the peak load.

plasma treatment may be influencing the interface performance more than by merely altering the wettability of the interface. The limited studies on surface chemical alterations conducted during this investigation[12] were inconclusive. However, evidence of chemical modifications of the polypropylene fiber surface have been reported by Denes et al.[3]



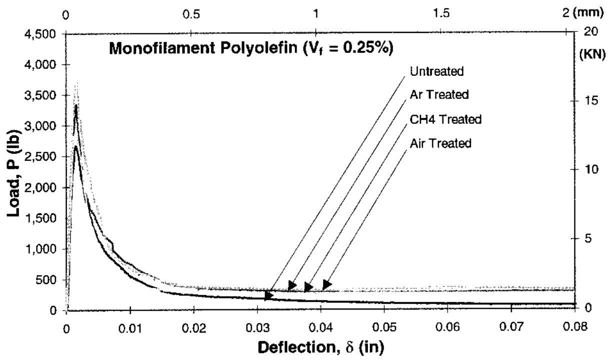
(a)



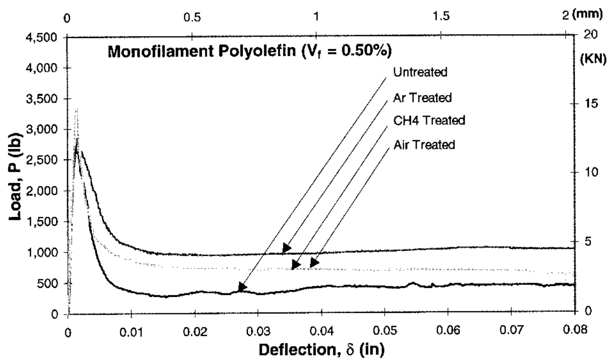
(b)

Figure 5 Load–deflection response of treated and untreated fibrillated polypropylene fiber composites showing influence of various fractions. (a) Fiber volume fraction = 0.25%, and (b) fiber volume fraction = 0.50%.

Toughness, which is generally measured as the area under the load–deflection response at prescribed values of deflection, is greater for all the treatment types compared to untreated fibers (Table IV). The Japanese toughness measure T_{JCI} and the ASTM toughness index I_{30}, both computed at relatively large limiting deflections, clearly are able to highlight the influence of plasma treatment in



(a)



(b)

Figure 6 Load–deflection response of treated and untreated monofilament polyolefin fiber composites showing influence of various fractions. (a) Fiber volume fraction = 0.25%, and (b) Fiber volume fraction = 0.50%.

enhancing the toughness of the fiber-reinforced composite system using treated fibers.

Figure 8 shows the energy absorbed per unit cross-sectional area (computed as area under the load–deflection curve up to various deflection limits divided by the cross-sectional area of the beam) as a function of specimen deflection for Argon-treated and untreated fiber concrete mixes. Clearly, in the fiber-dominated postcracking regime, the influence of plasma treatment becomes readily apparent, particularly at large deflections. Toughness enhancements can be achieved through improved fiber reinforcing parameters (use of higher fiber volume fraction, longer fibers, fibers with higher aspect ratio) and through fiber surface treatments (such as plasma treatment). The cost effectiveness of these alternate options of toughness enhancement presently favor plasma treatment. Additionally, another advantage is that the mixing problems due to balling and segregation often encountered while increasing the fiber volume fraction, fiber aspect ratio, and/or the fiber length do not exist for the case of plasma treatment.

This preliminary investigation was motivated by the need to demonstrate proof of concept and technical viability. Further work is needed to optimize the enhancement in flexural properties of plasma-treated fiber-reinforced concrete systems. Treatment variables include operating pressure, time of treatment, post treatment storage, gas flow rate, exposure distance, and type of gas. It is likely that different environmental and treatment conditions may be appropriate for different types of polymeric fibers.

CONCLUSIONS

1. Plasma treatment of polymeric fibers improved the flexural strength of fiber reinforced concrete composites made with these fibers. The enhancement of flexural toughness was, however, more significant than the improvement in flexural strength.
2. Among the limited treatment environments and conditions studied, it appears that Argon treatment improved the "overall" flexural performance the best. It is very likely, however, that different environments and different treatment conditions may provide the best enhancement in mechanical performance for the different types of synthetic fibers in use today. It is possible to optimize treatment environment (different gases and posttreatment storage conditions) and conditions (temperature, pressure, time, distance of exposure, etc.) for each fiber type.
3. Because plasma treatment of fibers results in composite toughness and strength enhancements akin to increased fiber volume contents, it is possible to use plasma treatment in situations where practical constraints may restrict the use of larger fiber volume contents (balling and segregation problems).
4. Enhancement of mechanical performance of synthetic fiber-reinforced concrete composites by plasma treatment of fibers can likely be attributed to a combination of two effects. The first is the modification

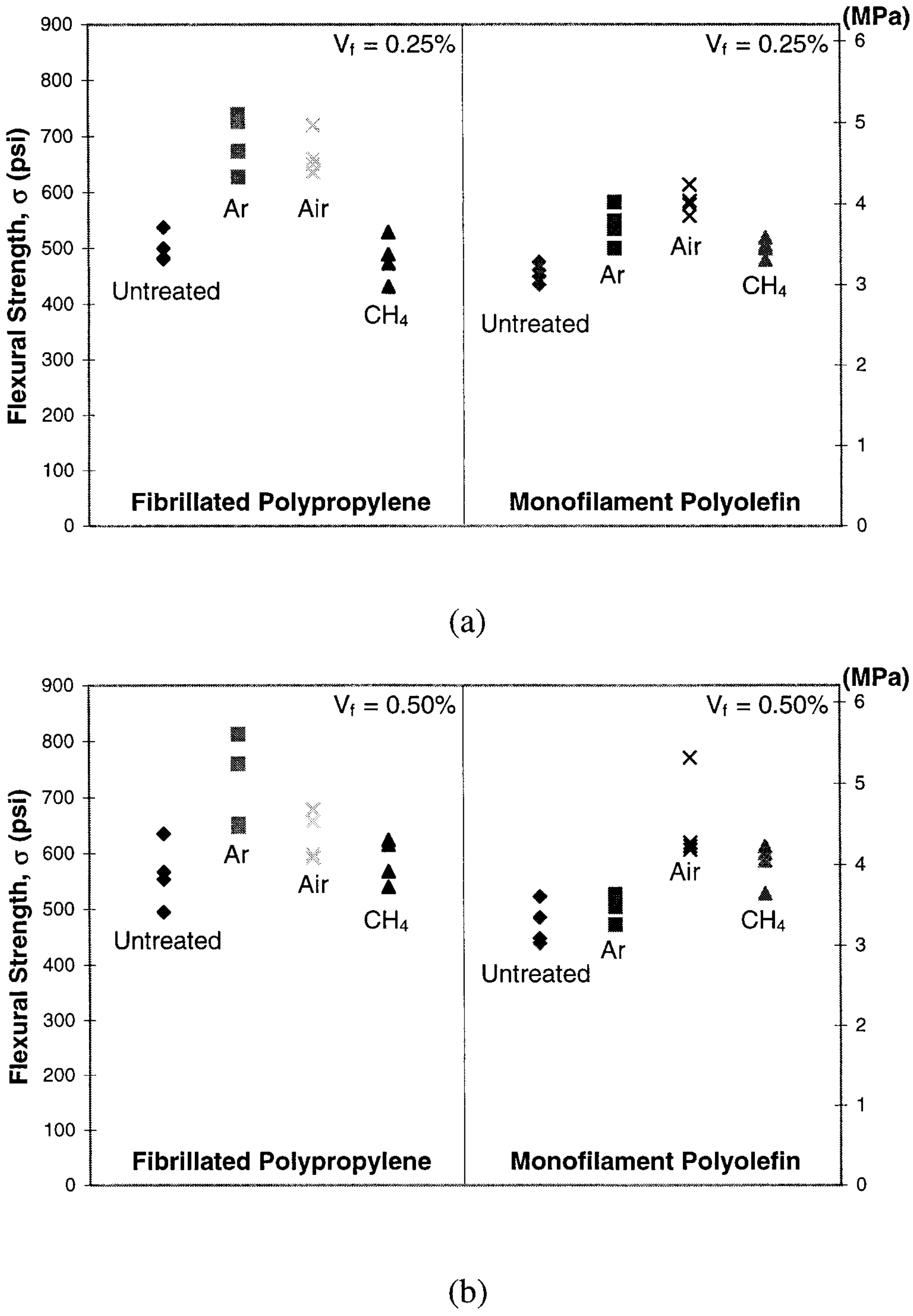


Figure 7 Flexural strength of treated and untreated fiber cement composites. (a) Fiber volume fraction = 0.25%, and (b) Fiber volume fraction = 0.50%.

of the weak boundary in the concrete mix near the fiber surface. Plasma treatment changes the hydrophobic nature of the fiber surface to hydrophilic. This change seems to modify the weak transition zone (water rich concrete layer caused by hydrophobic surface of the fiber) in the concrete matrix, which is identified as the

Table IV Summary of Results[a] from the Flexural Tests

Mix Details	Fiber Content and Treatment	First Crack Strength, π_f, (psi)[b]	First Crack Toughness, T_f, (lb-in)[b]	I_5	I_{10}	I_{20}	I_{30}	Toughness Index, T_{JCI} (lb.-in)[b]	Equivalent Flexural Strength, σ_b (psi)[b]
Plain Concrete	0.0%	616	3.68	2.78	3.70	4.25	4.39	16.56	35
Fibrillated	0.25% Untreated	500	3.84	2.99	4.32	5.89	7.02	38.94	81
Polypropylene	0.25% Ar	697	4.93	3.07	4.49	6.44	8.11	72.87	151
	0.25% $CH_4 + O_2$	481	2.91	3.41	4.85	6.88	8.52	40.25	87
	0.25% Air	667	3.99	3.07	4.38	6.34	8.05	58.61	122
	0.50% Untreated	563	3.58	3.57	5.57	8.90	11.83	82.13	175
	0.50% Ar	687	2.76	4.40	6.59	10.33	14.27	89.48	191
	0.50% $CH_4 + O_2$	586	4.11	3.86	6.04	9.50	12.76	104.68	203
	0.50% Air	632	3.99	3.78	6.14	10.49	14.46	111.32	215
Monofilament	0.25% Untreated	476	2.50	3.30	4.72	6.07	6.81	23.38	52
Polyolefin	0.25% Ar	535	2.95	3.23	4.69	6.27	7.27	37.20	74
	0.25% $CH_4 + O_2$	506	2.78	3.13	4.25	5.33	6.15	32.08	68
	0.25% Air	615	3.96	3.14	4.28	5.48	6.31	44.43	91
	0.50% Untreated	474	4.03	2.35	2.99	3.85	4.66	44.37	96
	0.50% Ar	503	3.54	2.85	4.49	6.83	8.88	84.78	173
	0.50% $CH_4 + O_2$	600	2.73	3.00	4.55	6.95	9.32	65.68	149
	0.50% Air	613	2.69	3.11	4.53	6.60	8.42	57.32	131

[a] ach entry in the table represents average values of three to four specimens.
[b] MPa = 145 psi, 1 N-m = 0.113 lb.-in.

potential failure zone in interfacial failures in fiber-reinforced concrete composites. The second is the modification of the fiber/concrete interface. The chemical changes on the fiber surface introduced by the plasma treatment enhance the compatibility of fibers with silica-rich inorganic matrix materials.

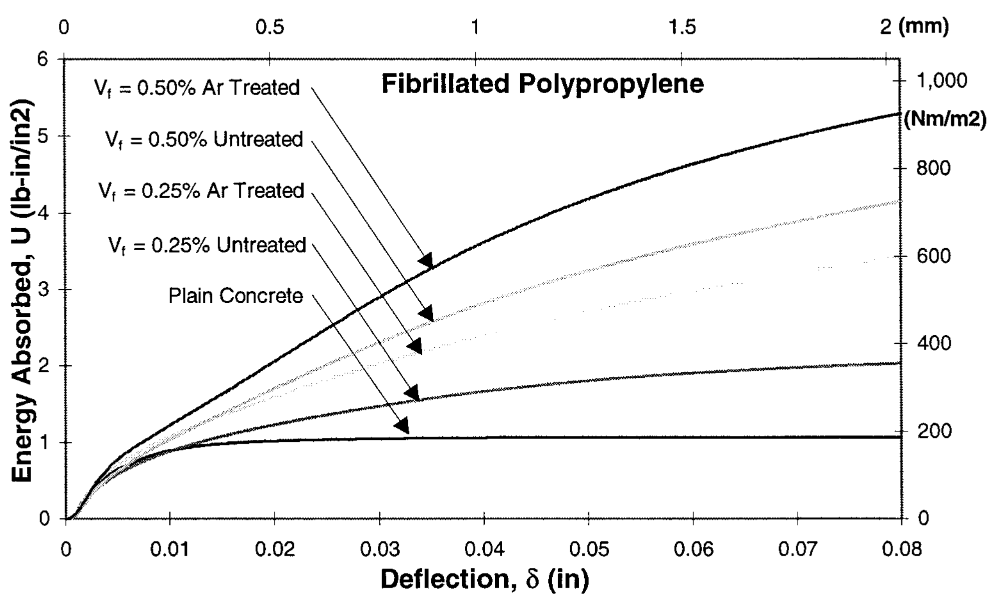


Figure 8 Energy absorbed versus deflection for plain concrete, untreated and Ar treated fiber cement composites.

This work was supported by a grant from the National Science Foundation (NSF-CTS-9400725). Thanks are also due to Mr. Mel Galinat, Fibermesh Company, and Mr. Cliff MacDonald, 3M, for providing the fibrillated polypropylene and monofilament polyolefin fibers, respectively. Instrumentation support in the CE Materials Laboratory was provided by Mr. Richard Wells and Mr. Richard Oberto.

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