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Silane-treated carbon fiber for reinforcing cement

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Abstract

The tensile strength of cement paste was increased by 56% and the modulus and ductility were increased by 39% by using silane-treated carbon fibers and silane-treated silica fume, relative to the values for cement paste with as-received carbon fibers and as-received silica fume. Silane treatment of fibers and silica fume contributed about equally to the strengthening. Silane treatment of fibers and silica fume also decreased the air void content and the drying shrinkage. The strengthening, air void content reduction and drying shrinkage reduction were less when the fiber treatment involved either potassium dichromate or ozone. Silane's effectiveness is due to its hydrophilic nature.

Keywords: A. Carbon fibers, Carbon composites; B. Surface treatment; C. X-ray photoelectron spectroscopy; D. Mechanical properties

1. Introduction

Carbon fiber cement–matrix composites [1] are structural materials that are gaining in importance quite rapidly due to the decrease in carbon fiber cost and the increasing demand of superior structural and functional properties. These composites contain short carbon fibers, typically 5 mm in length, as the short fibers can be used as an admixture in concrete (whereas continuous fibers cannot be simply added to the concrete mix) and short fibers are less expensive than continuous fibers. However, due to the weak bond between carbon fiber and the cement matrix, continuous fibers [2–4] are much more effective than short fibers in reinforcing concrete. Surface-treatment of carbon fiber (e.g., by heating [5] or by using ozone [6,7], silane [8], SiO_2 particles [9], or hot NaOH solution [10]) is useful for improving the bond between fiber and matrix, thereby improving the properties of the composite. In the case of surface treatment by ozone or silane, the improved bond is due to the enhanced wettability by water. Admixtures such as latex [6,11], methylcellulose [6], and silica fume [12] also help the bond.

The effect of carbon fiber addition on the properties of concrete increases with fiber volume fraction [13], unless the fiber volume fraction is so high that the air void content becomes excessively high [14]. (The air void content increases with fiber content and air voids tend to have a negative effect on many properties, such as the compressive strength.) In addition, the workability of the mix decreases with fiber content [13]. Moreover, the cost increases with fiber content. Therefore, a rather low volume fraction of fibers is desirable. A fiber content as low as 0.2 vol.% is effective [15], although fiber contents exceeding 1 vol.% are more common [16,20]. The required fiber content increases with the particle size of the aggregate, as the flexural strength decreases with increasing particle size [21].

Effective use of the carbon fibers in concrete requires dispersion of the fibers in the mix. The dispersion is enhanced by using silica fume (a fine particulate) as an admixture [14,22–24]. A typical silica fume content is 15% by weight of cement [14]. The silica fume is typically used along with a small amount (0.4% by weight of cement) of methylcellulose for helping the dispersion of the fibers and the workability of the mix [14]. Latex (typically 15–20% by weight of cement) is much less effective than silica fume for helping the fiber dispersion, but it enhances the workability, flexural strength, flexural toughness, impact resistance, frost resistance and acid resistance [14,25,26]. The ease of dispersion increases with decreasing fiber length [24].

The improved structural properties rendered by carbon fiber addition pertain to the increased tensile and flexible strengths, the increased tensile ductility and flexural tough-

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ness, the enhanced impact resistance, the reduced drying shrinkage and the improved freeze–thaw durability [13–15,17–25,27–38].

This paper addresses silane-treated carbon fiber for reinforcing cement by investigating the surface chemistry of the fiber as well as the properties of cement–matrix composites containing the fibers. Furthermore, this paper includes a comparison of this fiber with as-received, ozone-treated, and dichromate-treated carbon fibers.

2. Experimental methods

The carbon fibers were isotropic pitch-based, unsized, and of length ~5 mm, as obtained from Ashland Petroleum Co. (Ashland, KY, USA). The fiber properties are shown in Table 1. As-received and three types of surface-treated fibers were used. The fiber content was 0.5% by weight of cement. The surface-treatments involved (i) ozone (O_3), (ii) an aqueous solution of potassium dichromate ($K_2Cr_2O_7$, 30 wt.%) and sulfuric acid (H_2SO_4, 40 wt.%, which enhances the oxidation ability), and (iii) silane. The ozone treatment for surface oxidation involved exposure of the fibers to O_3 gas (0.6 vol.%, in O_2) at 160°C for 5 min. Prior to O_3 exposure, the fibers had been dried at 160°C in air for 30 min. The potassium dichromate treatment for surface oxidation involved immersing in the dichromate solution and heating to 60°C while stirring for 2 h, followed by filtration and washing with water and then drying at 110°C for 6 h. For the silane-treatment, the silane-coupling agent was a 1:1 (by weight) mixture of Z-6020 [$H_2NCH_2CH_2NHCH_2CH_2CH_2Si(OCH)_3)_3$] and Z-6040 [$OCH_2$ $CHCH_2OCH_2CH_2CH_2Si(OCH)_3)_3$] from Dow Corning Corp. (Midland, MI, USA). The amine group in Z-6020 serves as the catalyst for the curing of epoxy and consequently allows the Z-6020 molecule to attach to the epoxy end of the Z-6040 molecule. The trimethylsiloxy ends of the Z-6020 and Z-6040 molecules then connect to the –OH functional group on the surface of silica fume or carbon fibers. The silane was dissolved in ethylacetate. Surface treatment was performed by immersing in the silane solution, heating to 75°C while stirring, and holding at 75°C for 1 h, followed by filtration, washing with ethylacetate, and drying. After this, heating was conducted in a furnace at 110°C for 12 h.

Table 1
Properties of carbon fibers

Filament diameter	15±3 μm
Tensile strength	690 MPa
Tensile modulus	48 GPa
Elongation at break	1.4%
Electrical resistivity	3.0×10^{-3} Ω cm
Specific gravity	1.6 g cm^{-3}
Carbon content	98 wt.%

No aggregate (fine or coarse) was used. The water/cement ratio was 0.35. A water-reducing agent (TAMOL SN, Rohm and Haas Co., Philadelphia, PA, USA; sodium salt of a condensed naphthalenesulphonic acid) was used in the amount of 2% by weight of cement.

The cement used was portland cement (Type I) from Lafarge Corp. (Southfield, MI, USA). The silica fume (Elkem Materials, Inc., Pittsburgh, PA, USA; EMS 965) was used in the amount of 15% by weight of cement. The methylcellulose, used in the amount of 0.4% by weight of cement, was obtained from Dow Chemical (Midland, MI, USA) Methocel A15-LV. The defoamer (Colloids Inc., Marietta, GA, USA; 1010) used whenever methylcellulose was used in the amount of 0.13 vol.%.

A rotary mixer with a flat beater was used for mixing. Methylcellulose (if applicable) was dissolved in water and then the defoamer was added and stirred by hand for about 2 min. Then this mixture (if applicable), cement, water, water-reducing agent, silica fume, and fibers (if applicable) were mixed in the mixer for 10 min. After pouring into molds, an external vibrator was used to facilitate compaction and decrease the amount of air bubbles. The samples were demolded after 24 h and then cured in air at room temperature and a relative humidity of 100% for 28 days.

Dog-bone shaped specimens of the dimensions shown in Fig. 1 from Ref. [39] were used for tensile testing. They were prepared by using molds of the same shape and size. Tensile testing was performed using a screw-type mechanical testing system (Sintech 2/D). The displacement rate was 1.27 mm/min. The strain was measured by using a resistive strain gage that was mounted on each specimen. Four specimens of each composition were tested. Twelve compositions, as listed in Table 2, were studied. Six of the compositions had as-received silica fume; the other six had silane-treated silica fume.

The air void content was measured by using ASTM method C185-95. Three specimens of each composition were tested.

The drying shrinkage measurement was conducted as a function of curing age on samples of size 280×25.4×25.4

Table 2
Tensile strength (MPa) of cement pastes with and without fibers

Formulation[a]	Silica fume	
	As-received	Silane-treated
A	1.53±0.06	2.04±0.06
A^+	1.66±0.07	2.25±0.09
A^+F	2.00±0.09	2.50±0.11
A^+O	2.25±0.07	2.67±0.09
A^+K	2.32±0.08	2.85±0.11
A^+S	2.47±0.11	3.12±0.12

[a] A=Cement+water+water reducing agent+silica fume, A^+=A+methylcellulose+defoamer, $A^+F=A^+$+as-received fibers, $A^+O=A^+ + O_3$-treated fibers, $A^+K=A^+$+dichromate-treated fibers, $A^+S=A^+$+silane-treated fibers.

mm. A stainless steel contact point was positioned at each end along the length of each sample, such that the exterior tips of the two contact points were outside the sample and the interior tips of the two contact points were inside the sample and were separated by a distance of 250 mm, which was the gage length for the drying shrinkage strain measurement. The measurement was conducted in accordance with ASTM Method C490-93a.

The surface composition of carbon fibers, with and without treatment, was measured by electron spectroscopy for chemical analysis (ESCA).

3. Results

Tables 2 and 3 show the tensile strength and modulus respectively of twelve types of cement pastes. The strength is slightly increased by the addition of methylcellulose and defoamer, but the modulus is slightly decreased by the addition of methylcellulose and defoamer. However, both strength and modulus are increased by the addition of fibers. The effectiveness of the fibers in increasing strength and modulus increases in the order: as-received fibers>O_3-treated fibers>dichromate-treated fibers>silane-treated fibers. This trend applies whether the silica fume is as-received or silane-treated. For any of the formulations, silane-treated silica fume gives substantially higher strength and modulus than as-received silica fume. The highest tensile strength and modulus are exhibited by cement paste with silane-treated silica fume and silane-treated fibers. The strength is 56% higher and the modulus is 39% higher than those of the cement paste with as-received silica fume and as-received fibers. The strength is 26% higher and the modulus is 14% higher than those of the cement paste with as-received silica fume and silane-treated fibers. Hence, silane-treatments of silica fume and of fibers are about equally valuable in providing strengthening.

Table 4 shows the tensile ductility. It is slightly increased by the addition of methylcellulose and defoamer, and is further increased by the further addition of fibers. The effectiveness of the fibers in increasing the ductility increases in the order: as-received fibers>O_3-treated fibers>dichromate-treated fibers>silane-treated fibers. This trend applies whether the silica fume is as-received or silane-treated. For any of the formulations involving surface treated fibers, silane-treated silica fume gives higher ductility than as-received silica fume. The highest ductility is exhibited by cement paste with silane-treated silica fume and silane-treated fibers. The ductility is 39% higher than that of the cement paste with as-received silica fume and as-received fibers. It is 14% higher than that of the cement paste with as-received silica fume and silane-treated fibers.

Table 5 shows the air void content. It is decreased by the addition of methylcellulose and defoamer, but is increased by the further addition of fibers, whether the fibers have been surface treated or not. Among the formulations with fibers, the air void content decreases in the order: as-received fibers>O_3-treated fibers>dichromate-treated fibers>silane-treated fibers. This trend applies whether the silica fume is as-received or silane-treated. For any of the formulations (including those without fibers), silane-treated

Table 3
Tensile modulus (GPa) of cement pastes with and without fibers

Formulation[a]	Silica fume	
	As-received	Silane-treated
A	10.2±0.7	11.5±0.6
A^+	9.3±0.5	10.7±0.4
A^+F	10.9±0.3	12.9±0.7
A^+O	11.9±0.3	13.1±0.6
A^+K	12.7±0.4	14.3±0.4
A^+S	13.3±0.5	15.2±0.8

[a] A=Cement+water+water reducing agent+silica fume, A^+=A+methylcellulose+defoamer, $A^+F=A^+$+as-received fibers, $A^+O=A^+ + O_3$-treated fibers, $A^+K=A^+$+dichromate-treated fibers, $A^+S=A^+$+silane-treated fibers.

Table 4
Tensile ductility (%) of cement pastes with and without fibers

Formulation[a]	Silica fume	
	As-received	Silane-treated
A	0.020±0.0004	0.020±0.0004
A^+	0.023±0.0004	0.021±0.0004
A^+F	0.025±0.0003	0.024±0.0004
A^+O	0.026±0.0003	0.027±0.0004
A^+K	0.028±0.0003	0.030±0.0004
A^+S	0.031±0.0004	0.034±0.0004

[a] A=Cement+water+water reducing agent+silica fume, A^+=A+methylcellulose+defoamer, $A^+F=A^+$+as-received fibers, $A^+O=A^+ + O_3$-treated fibers, $A^+K=A^+$+dichromate-treated fibers, $A^+S=A^+$+silane-treated fibers.

Table 5
Air void content (±0.12%) of cement pastes with and without fibers

Formulation[a]	Silica fume	
	As-received	Silane-treated
A	3.73	3.26
A^+	3.42	3.01
A^+F	5.32	4.89
A^+O	5.07	4.65
A^+K	5.01	4.49
A^+S	4.85	4.16

[a] A=Cement+water+water reducing agent+silica fume, A^+=A+methylcellulose+defoamer, $A^+F=A^+$+as-received fibers, $A^+O=A^+ + O_3$-treated fibers, $A^+K=A^+$+dichromate-treated fibers, $A^+S=A^+$+silane-treated fibers.

Table 6
Drying shrinkage strain (10^{-4},±0.015) at different curing ages

Formulation[a]	With as-received silica fume				With silane-treated silica fume			
	1 day	4 days	8 days	28 days	1 day	4 days	8 days	28 days
B	1.13	3.02	3.72	4.41	1.01	2.88	3.62	4.18
BF	0.83	2.42	3.05	3.51	0.78	2.25	2.81	3.19
BO	0.83	2.36	3.02	3.48	0.76	2.24	2.79	3.09
BK	0.82	2.32	3.02	3.48	0.76	2.23	2.79	3.09
BS	0.81	2.32	2.98	3.31	0.75	2.12	2.72	2.99

[a] B=Cement+water+water reducing agent+silica fume+methylcellulose+defoamer, BF=B+as-received fibers, BO=B+O_3-treated fibers, BK=B+dichromate-treated fibers, BS=B+silane-treated fibers.

silica fume gives lower air void content than as-received silica fume.

Table 6 gives the drying shrinkage strains of ten types of cement paste as a function of curing age. The drying shrinkage is decreased by the addition of carbon fibers; it decreases in the order: as-received fibers>ozone-treated fibers>dichromate-treated fibers>silane-treated fibers (Table 6 and Fig. 1). This trend applies for any curing age, whether the silica fume is as-received or silane-treated. The drying shrinkage is decreased by the use of silane-treated silica fume in place of as-received silica fume, whether fibers are present or not (Table 6, Figs. 2 and 3). The drying shrinkage strain at 28 days is decreased by 5% when fibers are absent and silane-treated silica fume is used in place of as-received silica fume. When silane-treated fibers are present, it is decreased by 10% when silane-treated silica fume is used in place of as-received silica fume. The drying shrinkage strain at 28 days is decreased by 15% when both silane-treated silica fume and silane-treated carbon fibers are used in place of as-received silica fume and as-received carbon fibers. Compared to the cement paste without fibers but with as-received silica fume, the drying shrinkage strain is decreased by 25% when silane-treated fibers are added. Compared to the cement paste without fibers but with silane-treated silica fume, the drying shrinkage strain is decreased by 28% when silane-treated fibers are added. Compared to the cement paste without fibers but with as-received silica

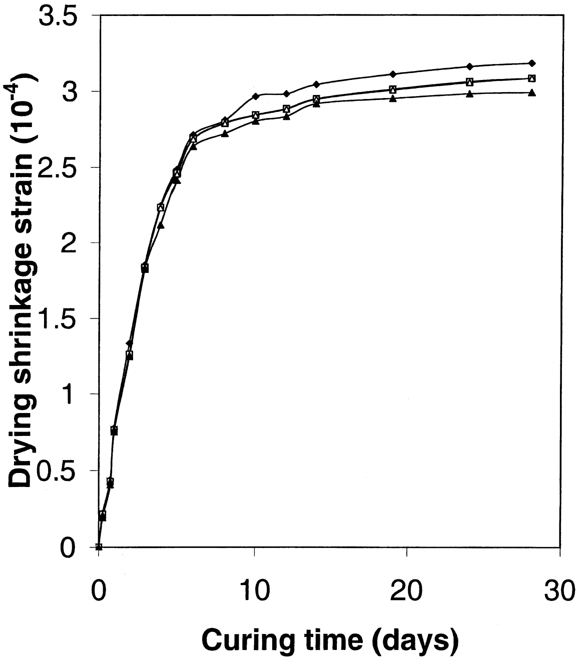

Fig. 1. Drying shrinkage strain vs. curing time for cement paste with as-received silica fume and as-received fibers (♦), ozone-treated fibers (□), dichromate-treated fibers (△), and silane-treated fibers (▲).

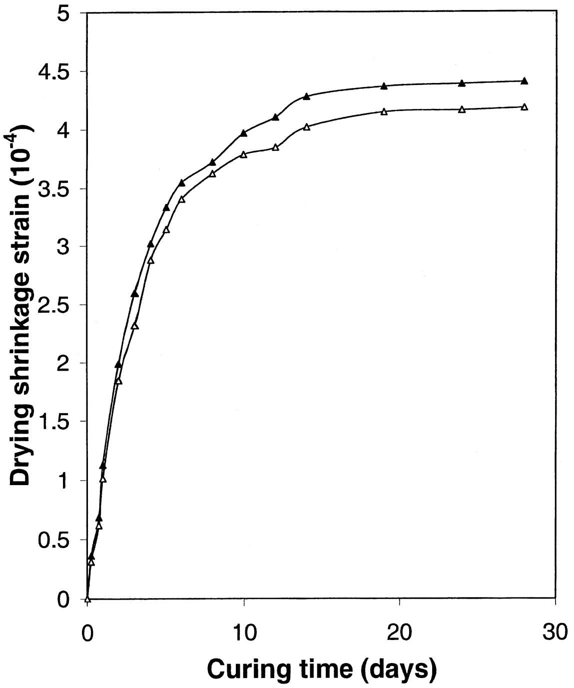

Fig. 2. Drying shrinkage strain vs. curing time for cement paste with as-received silica fume (no fiber) (▲) and for cement paste with silane-treated silica fume (no fiber) (△).

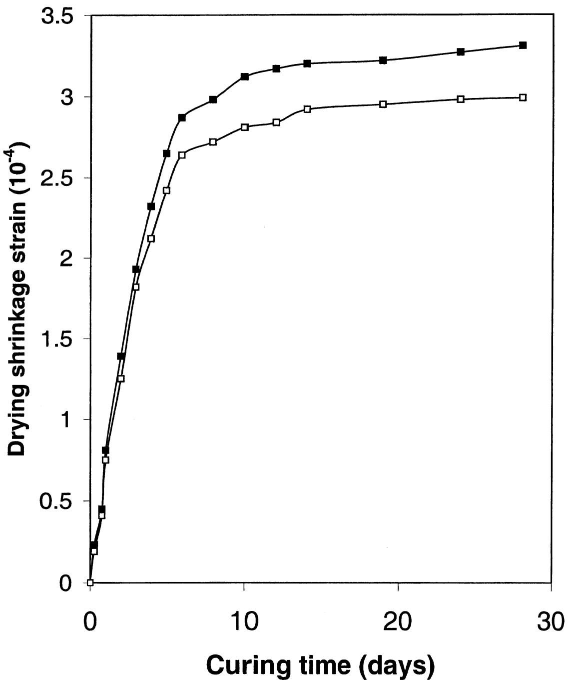

Fig. 3. Drying shrinkage strain vs. curing time for cement paste with as-received silica fume and silane-treated fibers (■) and for cement paste with silane-treated silica fume and silane-treated fibers (□).

fume, the drying shrinkage strain is decreased by 32% when silane-treated fibers are added and silane-treated silica fume is used in place of as-received silica fume.

ESCA results are shown in Tables 7–10 for carbon fibers. The surface carbon concentration is decreased and the surface oxygen concentration is increased by any of the three surface treatments (Table 7). In the case of the O_3 and dichromate treatments, this is due to the oxidation of the fiber surface and the introduction of hydrophilic functional groups such as –OH and –COOH to the surface. The charge corrected binding energies (C_{1s} and O_{1s}) of both O_3 and dichromate treated carbon fiber surfaces (Tables 8 and 9) confirm the existence of these functional groups. In the case of the silane treatment, the oxygen, nitrogen, and silicon concentrations on the surface increase and the carbon concentration on the surface decreases after the treatment, due to the composition of the silane coating of the surface (Table 7). That the surface chloride concentration is not zero after silane treatment is a result that is not understood. The charge corrected binding energies of the silane-treated fiber surface (Table 10) confirm the presence of silane.

Table 7
Surface elemental composition (at.%) of carbon fibers

Element	As-received	O_3 treated	Dichromate-treated	Silane-treated
O	13.0	20.2	20.6	21.8
N	–	–	–	9.3
C	85.4	79.8	79.4	54.3
Cl	–	–	–	4.0
Si	1.7	–	–	10.6

Table 8
Charge corrected binding energies (eV) and possible assignment for O_3-treated carbon fibers

Energy level	Binding energy (eV)	Assignment
C_{1s}	285.0	$(CH_2)_n$
	286.7	C–O
	288.6	C=O
O_{1s}	531.8	C=O
	533.6	C–O

Table 9
Charge corrected binding energies (eV) and possible assignment for dichromate-treated carbon fibers

Energy level	Binding energy (eV)	Assignment
C_{1s}	285.0	$(CH_2)_n$
	286.7	C–O
	288.6	C=O
O_{1s}	531.8	C=O
	533.6	C–O

4. Discussion

The hydrophilic nature of the silane molecule is believed to improve the bond between fibers and cement paste or that between silica fume and cement paste, thereby increasing the tensile strength, modulus and ductility and decreasing the air void content of the cement–matrix composite. Both ozone treatment and dichromate treatment involve surface oxidation; the oxidation results in oxygen-containing functional groups which help improve the hydrophilicity. The dichromate treatment is more effective than

Table 10
Charge corrected binding energies (eV) and possible assignment for silane-treated carbon fibers

Energy level	Binding energy (eV)	Assignment
C_{1s}	285.0	$(CH_2)_n$
O_{1s}	531.9	Si–O
Si_{2p}	102.2	Organic silicone
N_{1s}	399.0	C–N
	401.1	N–O

the ozone treatment (probably due to the greater uniformity of the resulting surface treatment), but both ozone and dichromate treatments are inferior to the silane-treatment. The superiority of the silane-treatment over the other two treatments of the fibers applies whether the silica fume is as-received or silane-treated.

Carbon fiber addition is effective for decreasing the drying shrinkage, as previously reported [13,15,40–42]. The effectiveness is further enhanced by surface treatment of the fibers, although the effect of the fiber surface treatment is small compared to the effect of the fiber addition. The drying shrinkage strain at 28 days is decreased by 6% when silane-treated fibers are used in place of as-received fibers, in the presence of as-received silica fume. The drying shrinkage strain at 28 days is decreased by 10% when silane-treated silica fume is used in place of as-received silica fume, in the presence of silane-treated carbon fibers. The drying shrinkage strain at 28 days is decreased by 15% when both silane-treated silica fume and silane-treated carbon fibers are used in place of as-received silica fume and as-received carbon fibers. The greater effectiveness of silane-treatment compared to the other fiber surface treatments for drying shrinkage reduction is consistent with its greater effectiveness for improving the mechanical properties.

The ESCA results show that the carbon fiber surface is partly coated by or bonded to silane molecules. Due to the hydrophilic nature of silane, the treated fibers are expected to be more uniformly distributed in the cement. More importantly, the formation of chemical bonds at which silane serves as bridges between the surface of fiber and the cement matrix is expected to make the composite denser and stronger [12]. Therefore, the drying shrinkage strain is decreased by silane-treatment of fibers.

5. Conclusion

The tensile strength, modulus and ductility were increased and the air void content was decreased, when the fibers in carbon fiber reinforced cement paste had been surface treated. The effectiveness of treatment decreased in the order: silane>dichromate>ozone. Additional strengthening and air void content reduction were observed when the silica fume in the carbon fiber reinforced cement paste had been surface treated with silane. These effects of silane-treatment are attributed to the hydrophilic nature of silane.

Silane-treatment of silica fume and/or carbon fiber is effective for decreasing the drying shrinkage of cement paste. The increase of the hydrophilic character of fibers and particles after the treatment and the formation of chemical bonds between fibers/particles and cement are believed to be the main reasons for the observed decrease of the drying shrinkage. By adding silane-treated carbon fibers and replacing as-received silica fume by silane-treated silica fume, the shrinkage at 28 days is decreased by 32%.

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