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# IMPROVING THE TENSILE PROPERTIES OF CARBON FIBER REINFORCED CEMENT BY OZONE TREATMENT OF THE FIBER

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#### ABSTRACT

The tensile strength, modulus and ductility of carbon fiber reinforced cement paste were increased by ozone treatment of the fibers prior to using the fibers. Increases were observed whether or not the paste contained methylcellulose/silica fume/latex. The ozone treatment involved exposure to O<sub>3</sub> gas (0.3 vol.%, in air) for 10 min at 160°C. Copyright © 1996 Elsevier Science Ltd

## Introduction

Cementitious materials are much weaker under tension than compression. The tensile strength and ductility of these materials can be greatly increased by the addition of short fibers, such as carbon, steel and polymer fibers [1-9]. Among various types of fibers, carbon fibers are particularly effective for increasing the tensile strength [10]. The tensile strength is even larger when latex is present [10-14]. It was reported that ozone (O<sub>3</sub>) treatment of carbon fibers enhances the bond strength between fiber and cement paste, as indicated by single fiber pull-out testing [15], but the effect of the ozone treatment on the mechanical properties of carbon fiber cement-matrix composites was not reported. This effect is the subject of this paper. It was addressed in the context of different formulations of cement pastes, including pastes with methylcellulose [16], silica fume and latex.

## **Experimental Methods**

The carbon fibers were isotropic pitch based and unsized, as obtained from Ashland Petroleum Co. (Ashland, Kentucky). The fiber properties are shown in Table 1. As-received and surface treated fibers were used in the amount of 0.5% by weight of cement, i.e., 0.51 vol.% of cement paste. The surface treatment involved exposure of the fibers to O<sub>3</sub> gas (0.3 vol.%, in air) for 10 min at 160°C. Prior to O<sub>3</sub> exposure, the fibers had been dried at 110°C in air for 1 h.

Cement paste made from Portland cement (Type 1) from Lafarge Corp. (Southfield, MI) was used for the cementitious material. Five types of pastes were used, namely (i) plain cement paste (with only cement and water, such that the water/cement ratio is 0.45, (ii) cement paste with fibers (together with water reducing agent in the amount of 1% by weight of cement and with water-cement ratio = 0.32), (iii) cement paste with fibers and methylcel-

TABLE 1
Properties of Carbon Fibers

Filament diameter Tensile strength Tensile modulus Elongation at break	10 μm 690 MPa 48 GPa 1.4%
Electrical resistivity	3.0x10 <sup>-3</sup> Ω cm
Specific gravity	1.6 g cm <sup>-3</sup>
Carbon content	98 wt.%

lulose in the amount of 0.4% by weight of cement (together with water reducing agent in the amount of 1% by weight of cement, and with water-cement ratio = 0.32), (iv) cement paste with fibers, methylcellulose in the amount of 0.4% by weight of cement and silica fume in the amount of 15% by weight of cement (together with water reducing agent in the amount of 3% by weight of cement, and with water-cement ratio = 0.35), and (v) cement paste with latex in the amount of 20% by weight of cement (water-cement ratio = 0.23, without water reducing agent). The water reducing agent used was TAMOL SN (Rohm and Haas Co., Philadelphia, PA), which contained 93-96% sodium salt of a condensed naphthalenesulfonic acid. The methylcellulose used was Dow Chemical, Midland, MI, Methocel A15-LV. The defoamer (Colloids Inc., Marietta, GA, 1010) used whenever methylcellulose was used was in the amount of 0.13 vol.%. The latex (Dow Chemical, Midland, MI, 460NA) used was a styrene-butadiene polymer. The antifoam (Dow Corning, Midland, MI, 2410) used whenever latex was used was in the amount of 0.5% of the weight of the latex.

A Hobart mixer with a flat beater was used for mixing. For the case of cement paste containing latex, the latex and antifoam first were mixed by hand for about 1 min. Then this mixture, cement, water and the water reducing agent were mixed in the mixer for 5 min. For the case of cement paste containing methylcellulose, methylcellulose was dissolved in water and then the defoamer was added and stirred by hand for about 2 min. Then this mixture, cement, water, water reducing agent and silica fume (if applicable) were mixed in the mixer for 5 min. After pouring the mix into oiled molds, a vibrator was used to decrease the amount of air bubbles. The specimens were demolded after 1 day and then allowed to cure at room temperature in air for 28 days.

Dog-bone shaped specimens of the dimensions shown in Fig. 1 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. During compressive or tensile loading up to fracture, the strain was measured by the cross-head displacement in compressive testing or by a strain gage in tensile testing. Six specimens of each composition were tested.

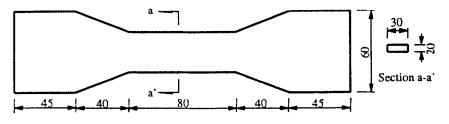


FIG. 1. Shape and dimensions (in mm) of the specimens tested under tension.

TABLE 2

Effect of Ozone Treatment of Carbon Fiber on Tensile Properties of Carbon Fiber (0.51 vol.%) Reinforced Cement Paste at 28 Days

	P	+F	+F+M	+F+M+SF	+F+L
Strength (MPa)					
Without treatment	0.91	1.36	2.01	1.97	3.18
	(±2.7%)	(±1.2%)	$(\pm 3.2\%)$	(±5.1%)	(±3.1%)
With treatment	0.91	1.58	2.78	2.23	3.42
	(±2.7%)	(±3.8%)	(±3.2%)	(±2.7%)	(±3.1%)
Modulus (GPa)					
Without treatment	11.2	9.4	10.3	13.8	7.6
	(±2.1%)	(±1.2%)	(±2.1%)	(±2.6%)	(±2.1%)
With treatment	11.2	10.3	12.7	18.2	13.8
	(±2.1%)	(±3.4%)	(±2.0%)	(±2.1%)	(±2.4%)
Ductility (%)					
Without treatment	0.0041	0.0121	0.0198	0.0167	0.0462
	(±1.9%)	(±3.1%)	(±1.1%)	(±2.6%)	(±1.9%)
With treatment	0.0041	0.0140	0.0231	0.0211	0.0413
	(±1.9%)	(±2.2%)	(±1.8%)	(±2.1%)	(±1.7%)

Note: P = plain (no fiber), F = fiber, M = methylcellulose, SF = silica fume, L = latex.

#### Results

Table 2 shows the effect of ozone treatment of the carbon fiber on the tensile properties of cement pastes. Ozone treatment increased the tensile strength, modulus and ductility for all four formulations of carbon fiber reinforced cement paste, except that the treatment decreased the ductility for the formulation with latex (due to the high fiber-matrix bond strength for the latex case, even without the fiber treatment [15]). The fractional increase in tensile strength due to the fiber treatment was particularly large (38%) for the formulation with methylcellulose (but no silica fume). The fractional increase in tensile modulus due to the fiber treatment was particularly large (82%) for the formulation with latex. The fractional increase in tensile ductility due to the fiber treatment was particularly large (26%) for the formulation with methylcellulose and silica fume. With the fiber treatment, the highest tensile strength reached was 3.42 MPa (with latex), the highest tensile modulus reached was 18.2 GPa (with methylcellulose and silica fume), and the highest tensile ductility reached was 0.0413% (with latex).

## Conclusion

The tensile strength, modulus and ductility of carbon fiber reinforced cement paste were increased by ozone treatment of the carbon fibers prior to using the fibers. Increases in all three parameters were observed whether or not the paste contained methylcellulose/silica fume/latex. The only exception is that the ductility was decreased by the ozone treatment for the case of cement paste with latex.

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