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## SUBMICRON CARBON FILAMENT CEMENT-MATRIX COMPOSITES FOR ELECTROMAGNETIC INTERFERENCE SHIELDING

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### ABSTRACT

Carbon filaments of diameter 0.1 mm were found to be a much more effective additive than conventional carbon fibers of diameter 10 mm in providing cement pastes capable of electromagnetic interference shielding. With 0.54 vol.% filaments and a shield thickness of 4 mm, a shielding effectiveness of 30 dB was attained at 1-2 GHz. However, the filaments were less effective than the fibers for reinforcing and for providing strain sensing cement-matrix composites. *Copyright © 1996 Elsevier Science Ltd*

### Introduction

As the environment is increasingly sensitive to electronic pollution, the ability of a building to shield electromagnetic radiation is of increasing importance. This is particularly true for buildings housing electronics and for electric power plants. As cement itself lacks the ability to shield electromagnetic radiation, admixtures are needed in order to attain the ability to shield. Earlier work [1] has shown that the addition of short carbon fibers (10 mm diameter) is effective for enhancing shielding. Due to the skin effect, electromagnetic radiation at high frequencies interacts only with the region of a conductor (such as a carbon fiber) near its surface. As a result, the shielding effectiveness is expected to increase with decreasing fiber diameter, when the fiber volume fraction is fixed. Therefore, the use of fibers of diameter smaller than those previously used is of interest.

Conventional carbon fibers are typically of diameter around 10 mm and are made from either pitch or a polymer such as polyacrylonitrile [2]. In contrast, carbon filaments made catalytically from carbonaceous gases (such as methane) are typically 0.1 mm in diameter [2]. Though conventional carbon fibers can be continuous in length, carbon filaments are discontinuous. However, due to the tiny diameter, the aspect ratio is typically quite large ( $> 1000$ ). Though conventional carbon fibers are straight, carbon filaments are bent and intertwined, resembling cotton wool. In spite of considerable previous work on the use of conventional carbon fibers in concrete [3-17], no previous work has been reported on the use of carbon filaments in concrete. This paper shows that the use of the carbon filaments is much

more effective than the use of conventional carbon fibers in enhancing the electromagnetic shielding effectiveness, but is less effective for reinforcing and for providing strain sensing cement-matrix composites.

### Experimental Methods

Cement paste made from Portland cement (Type I) from Lafarge Corp. (Southfield, MI) was used for the cementitious material. The admixtures used include (i) latex, a styrene butadiene polymer (Dow Chemical Co., Midland, MI 460NA) with the polymer making up about 48% of the solution and with styrene and butadiene in the weight ratio 66 : 34, such that the latex (20% by weight of cement) was used along with an antifoam (Dow Corning Corp., Midland, MI, #2410, 0.5% by weight of latex), (ii) methylcellulose (Dow Chemical Corp., A15-LV, 0.4% by weight of cement), which was used along with a defoamer (Colloids Inc., Marietta, GA, Colloids 1010, 0.13 vol.%), (iii) silica fume (Elken Materials Inc., Pittsburgh, PA, 15% by weight of cement), (iv) carbon filaments, which were of diameter 0.1 mm and length > 100 mm, as obtained from Applied Sciences Inc. (Cedarville, Ohio), and (v) carbon fibers, which were of diameter 10 mm and length 5 mm, as obtained from Ashland Petroleum Co. (Ashland, Kentucky). The filaments or fibers were used in the amount of 0.5% by weight of cement; this amount corresponded to a filament/fiber volume fraction of 0.51%. The water reducing agent was a sodium salt of a condensed naphthalenesulfonic acid (TAMOL SN, Rohm and Haas Company, Philadelphia, PA) used in amounts as shown in Table 1 for various mixes. Table 1 also shows the water/cement ratio for each mix. The amounts in Table 1 were chosen in order to maintain the slump at around 170 mm. No aggregate (whether fine or coarse) was used.

A Hobart mixer with a flat beater was used. For cement pastes containing latex, the latex, antifoam and filaments/fibers were first mixed by hand for about 1 min. Then this mixture, cement and water were mixed in the Hobart mixer for 5 min. For pastes containing methylcellulose, methylcellulose was dissolved in water and then filaments/fibers and the defoamer were added and stirred by hand for about 2 min. Then this mixture, cement, water and silica fume (if applicable) were mixed in the mixer for 5 min. After pouring the mix into oiled molds, an external vibrator was used to decrease the amount of air bubbles. The specimens

TABLE 1  
Amounts of Water and Water Reducing Agent (WR) for Each Mix

	<u>Water/cement ratio</u>	<u>WR/cement ratio</u>
Plain	0.45	0
+ F	0.40	0
+ M + F	0.32	1%
+ M + SF + F	0.35	1%
+ L + F	0.23	0

Note: L = latex, M = methylcellulose, SF = silica fume, F = fibers/filaments.

were demolded after 1 day and then allowed to cure at room temperature in air (relative humidity = 30%) for 28 days. All testing was performed at 28 days.

The shielding effectiveness was measured in terms of the attenuation using the coaxial cable method. The set-up consisted of an Elgal SET 19A shielding effectiveness tester with its input and output connected to a Hewlett-Packard (HP) 8510A network analyzer. An HP APC-7 calibration kit was used to calibrate the system. The frequency was scanned from 1 to 2 GHz such that 21 data points were taken within this frequency range. The sample placed in the center plane of the tester (with the input and output of the tester on the two sides of the sample) was in the form of an annular ring of outer diameter 97 mm and inner diameter 32 mm. The sample thickness ranged from 3.6 to 4.1 mm. Six specimens of each type were tested.

The DC volume electrical resistivity was measured by the four-probe method (outer two probes for passing current and inner two probes for voltage measurement), using silver paint for the electrical contacts, which were applied around the perimeter of the specimen ( $160 \times 40 \times 40$  mm) in four parallel planes perpendicular to the current direction (along the longest dimension of the specimen). Six specimens of each type were tested.

Tensile testing was performed on dogbone shaped specimens. The specimen cross section was  $30 \times 20$  mm in the narrow part of the dogbone shape. Six specimens of each type were tested. The Sintech 2/D screw action mechanical testing system was used at a cross head speed of 1.27 mm/min. The strain was measured by using a strain gage attached to the narrow part of the dogbone shaped specimen. The strain allowed determination of the tensile modulus and ductility.

For compressive testing according to ASTM C109-80, specimens were prepared by using a  $2 \times 2 \times 2$  in ( $5.1 \times 5.1 \times 5.1$  cm) mold. Compression testing was performed using a hydraulic material testing system (MTS). The cross head speed was 1.27 mm/min.

Testing of the strain sensing ability was conducted under cyclic tension and cyclic compression, as described in Ref. 15. In this test, the electrical resistance was measured in the stress direction using the four-probe method while cyclic tension/compression was applied at a stress amplitude of 0.70 of the corresponding tensile/compressive fracture stress. Each

TABLE 2  
Shielding Effectiveness and Electrical Resistivity of Cement Pastes Without and With Carbon Filaments

Cement paste	Shielding effectiveness				
	Attenuation (dB)			Thickness (mm) ( $\pm 0.2$ mm)	Resistivity ( $\Omega \cdot \text{cm}$ )
	1.0 GHz	1.5 GHz	2.0 GHz		
Plain	0.4( $\pm 3.1\%$ )	0.5( $\pm 2.5\%$ )	1.5( $\pm 2.1\%$ )	3.6	$1.62 \times 10^5$
+ F	26.4( $\pm 1.8\%$ )	25.8( $\pm 2.2\%$ )	26.1( $\pm 3.2\%$ )	4.0	$1.93 \times 10^4$
+ M + F	27.5( $\pm 3.3\%$ )	27.1( $\pm 2.7\%$ )	26.8( $\pm 2.5\%$ )	3.8	$2.75 \times 10^4$
+ M + SF + F	29.7( $\pm 3.2\%$ )	28.8( $\pm 2.8\%$ )	28.2( $\pm 2.9\%$ )	3.9	$1.34 \times 10^4$
+ L + F	30.2( $\pm 1.9\%$ )	28.7( $\pm 3.1\%$ )	29.3( $\pm 2.2\%$ )	4.1	$8.14 \times 10^4$

Note: F = filaments, M = methylcellulose, SF = silica fume, L = latex.

tension cycle took 42.2 s; each compression cycle took 32.6 s. The strain sensing ability was tested only for the cement paste with methylcellulose, silica fume and filaments.

## Results

Table 2 shows the shielding effectiveness at 1.0, 1.5 and 2.0 GHz for plain cement pastes and five types of pastes with carbon filaments. The filaments greatly increased the shielding effectiveness, whatever other ingredients were present. Comparison with corresponding data for cement pastes with carbon fibers [1] shows that the filaments are much more effective than the fibers for shielding. For example, at similar volume fractions, fibers gave 10 dB at 1.5 GHz, whereas filaments gave 26 dB at 1.5 GHz.

Table 2 also shows the electrical volume resistivity for each type of paste. The filaments decreased the resistivity, whatever other ingredients were present. Among the four types of

TABLE 3  
Comparison of the Mechanical Properties of Cement Pastes (28 Days) Reinforced with Carbon Fibers and Carbon Filaments, both at 0.51 vol.%

	P	+F	+F+M	+F+M+SF	+F+L
<b>Tensile strength (MPa)</b>					
Fibers	0.91 ( $\pm 2.7\%$ )	1.72 ( $\pm 1.3\%$ )	2.01 ( $\pm 3.2\%$ )	1.97 ( $\pm 5.1\%$ )	3.18 ( $\pm 3.1\%$ )
Filaments	0.91 ( $\pm 2.7\%$ )	1.23 ( $\pm 1.9\%$ )	1.52 ( $\pm 2.5\%$ )	1.67 ( $\pm 3.1\%$ )	2.86 ( $\pm 3.2\%$ )
<b>Tensile modulus (GPa)</b>					
Fibers	11.2 ( $\pm 2.1\%$ )	12.9 ( $\pm 1.8\%$ )	10.3 ( $\pm 2.1\%$ )	13.8 ( $\pm 2.6\%$ )	7.6 ( $\pm 2.1\%$ )
Filaments	11.2 ( $\pm 2.1\%$ )	12.4 ( $\pm 1.9\%$ )	8.7 ( $\pm 2.3\%$ )	12.8 ( $\pm 1.2\%$ )	6.8 ( $\pm 1.2\%$ )
<b>Tensile ductility (%)</b>					
Fibers	0.0041 ( $\pm 1.9\%$ )	0.0125 ( $\pm 2.3\%$ )	0.0198 ( $\pm 1.1\%$ )	0.0167 ( $\pm 2.6\%$ )	0.0462 ( $\pm 1.9\%$ )
Filaments	0.0041 ( $\pm 1.9\%$ )	0.0090 ( $\pm 2.5\%$ )	0.0160 ( $\pm 2.1\%$ )	0.0140 ( $\pm 1.8\%$ )	0.0360 ( $\pm 2.2\%$ )
<b>Compressive strength (MPa)</b>					
Fibers	57.9 ( $\pm 3.2\%$ )	42.8 ( $\pm 1.3\%$ )	44.2 ( $\pm 1.8\%$ )	49.3 ( $\pm 2.7\%$ )	46.1 ( $\pm 3.2\%$ )
Filaments	57.9 ( $\pm 3.2\%$ )	40.9 ( $\pm 2.1\%$ )	41.6 ( $\pm 2.8\%$ )	47.2 ( $\pm 2.8\%$ )	43.3 ( $\pm 2.0\%$ )
<b>Compressive modulus (GPa)</b>					
Fibers	2.92 ( $\pm 2.3\%$ )	6.27 ( $\pm 1.2\%$ )	4.09 ( $\pm 2.6\%$ )	4.12 ( $\pm 2.7\%$ )	4.31 ( $\pm 1.5\%$ )
Filaments	2.92 ( $\pm 2.3\%$ )	5.75 ( $\pm 1.2\%$ )	3.52 ( $\pm 1.8\%$ )	3.55 ( $\pm 1.4\%$ )	3.72 ( $\pm 2.3\%$ )
<b>Compressive ductility (%)</b>					
Fibers	1.72 ( $\pm 2.2\%$ )	1.21 ( $\pm 1.2\%$ )	1.28 ( $\pm 2.3\%$ )	1.34 ( $\pm 1.6\%$ )	1.42 ( $\pm 3.1\%$ )
Filaments	1.72 ( $\pm 2.2\%$ )	1.12 ( $\pm 2.1\%$ )	1.23 ( $\pm 1.7\%$ )	1.29 ( $\pm 2.2\%$ )	1.33 ( $\pm 1.5\%$ )

Note: P = plain, F = fibers/filaments, M = methylcellulose, SF = silica fume, L = latex.

pastes with filaments, the one containing latex gave the highest resistivity while the one containing methylcellulose + silica fume gave the lowest resistivity, because methylcellulose + silica fume is most effective for dispersing the filaments while latex is least effective, as previously shown for the case of carbon fibers [11].

Although latex and (methylcellulose + silica fume), when used with filaments, gave similar shielding effectiveness, latex gave a higher volume resistivity than (methylcellulose + silica fume). This is because of the high filament/cement contact electrical resistivity in the case of latex, as previously shown for the case of carbon fibers [18]. This contact resistivity affects the volume resistivity more than the shielding effectiveness.

Table 3 shows the tensile and compressive properties of cement pastes with fibers and those with filaments. The pastes with filaments exhibited lower strength, modulus and ductility (both tensile and compressive) than the counterparts with fibers. Nevertheless, the filaments were still an effective reinforcement, as shown by comparison with plain cement paste. Due to the impossibility of single filament tensile testing, the mechanical properties of the filaments are not known, though those of the fibers are known. The inferior mechanical properties of the cement pastes with filaments compared to those with fibers is probably due to the difference in the mechanical properties and morphology between filaments and fibers.

Fig. 1 gives the fractional resistance increase ( $DR/R_0$ ) during cyclic tensile loading for the cement paste with filaments, methylcellulose and silica fume. The  $DR/R_0$  appeared to increase upon loading and decrease upon unloading (as in the case of fibers in place of filaments [15]), but the noise in  $DR/R_0$  was severe. During cyclic compression,  $DR/R_0$  appeared to decrease upon loading and increase upon unloading (as in the case of fibers in place of

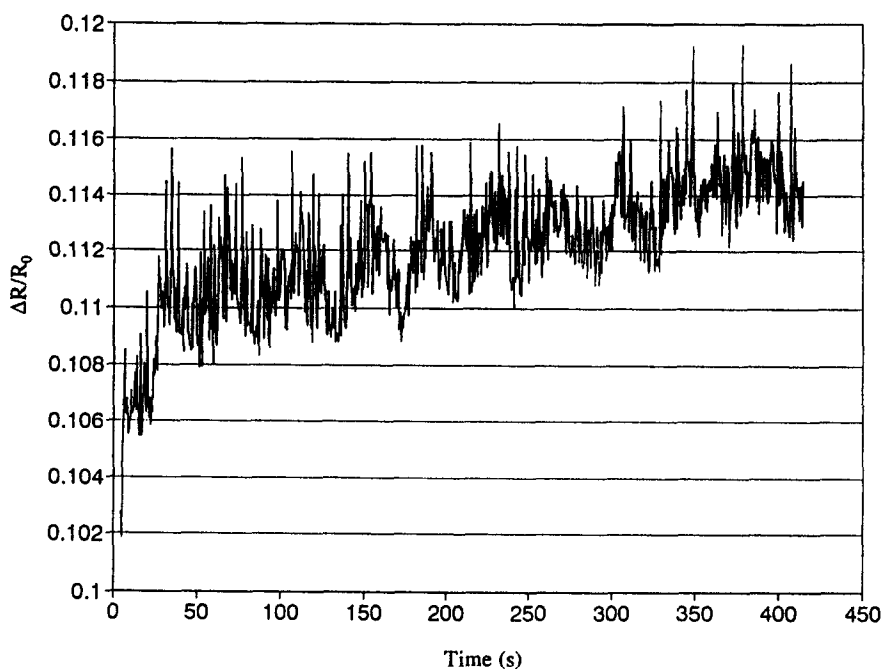


FIG. 1.

Fractional resistance increase ( $DR/R_0$ ) during the first 10 cycles of tensile loading for cement paste with filaments, methylcellulose and silica fume.

filaments [15]), but the noise in DR/R<sub>0</sub> was severe. Comparison with results for carbon fibers in place of carbon filaments [15,16] shows that the noise in DR/R<sub>0</sub> was much greater for filament composites than fiber composites. This difference is attributed to the difference in morphology between fibers and filaments. The bent and intertwined morphology probably made the filaments not able to cause the resistivity of the composite to change when the composite was loaded. Hence, the strain sensing ability was inferior for composites with filaments than those with fibers.

### Conclusion

Carbon filaments of diameter 0.1 mm were much more effective than carbon fibers of diameter 10 mm in electromagnetic interference shielding, but were less effective for reinforcing and for providing strain sensing cement-matrix composites.

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### References

1. Jeng-Maw Chiou, Qijun Zheng and D.D.L. Chung, *Composites* 20(4), 379 (1989).
2. D.D.L. Chung, *Carbon Fiber Composites*, Butterworth-Heinemann, 1994.
3. P.-W. Chen and D.D.L. Chung, *Composites* 24(1), 33 (1993).
4. X. Yang and D.D.L. Chung, *Composites* 23(6), 453 (1992).
5. P. Soroushian, F. Aouadi and M. Nagi, *ACI Materials J.* 88(1), 11 (1991).
6. P.-W. Chen and D.D.L. Chung, *Smart Mater. Struct.* 2, 22 (1993).
7. P.-W. Chen, X. Fu and D.D.L. Chung, *Cem. Concr. Res.* 25(3), 491 (1995).
8. P.-W. Chen and D.D.L. Chung, *J. Am. Ceramic. Soc.* 78(3), 816 (1995).
9. D.D.L. Chung, *Smart Mater. Struct.* 4, 59 (1995).
10. P.-W. Chen and D.D.L. Chung, *ACI Materials J.* 93(2), 129 (1996).
11. P.-W. Chen, X. Fu and D.D.L. Chung, *ACI Materials J.*, in press.
12. P.-W. Chen and D.D.L. Chung, *ACI Materials J.*, in press.
13. X. Fu and D.D.L. Chung, *Cem. Concr. Res.* 25(4), 689 (1995).
14. X. Fu and D.D.L. Chung, *Cem. Concr. Res.* 25(7), 1391 (1995).
15. X. Fu and D.D.L. Chung, *Cem. Concr. Res.* 26(1), 15 (1996).
16. P.-W. Chen and D.D.L. Chung, *Composites: Part B* 27B, 11 (1996).
17. P.-W. Chen and D.D.L. Chung, *Composites: Part B* 27B, 269 (1996).
18. X. Fu, W. Lu and D.D.L. Chung, *Cem. Concr. Res.*, in press.