Dispersion of Short Fibers in Cement

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Abstract: The degree of dispersion of short microfibers in cement, as assessed by electrical resistivity measurement for the case of electrically conductive fibers at a volume fraction below the percolation threshold, is improved by the use of admixtures (namely, silica fume, acrylic particle dispersion, methylcellulose solution, and silane) and fiber surface treatment (such as ozone treatment). Acrylic particle dispersion is more effective than latex particle dispersion.

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Introduction

Short fibers are used as admixtures in cement-based materials for the purpose of decreasing the drying shrinkage, increasing the flexural toughness, and, in some cases, increasing the flexural strength as well (Park and Lee 1993; Chen et al. 1997; Chen and Chung 1993; Toutanji et al. 1993; Banthia and Sheng 1996). In the case that the fibers are electrically conductive, the fibers may also provide nonstructural functions, such as self-sensing (for sensing the strain, damage, or temperature), self-heating (for deicing), and electromagnetic reflection (for electromagnetic interference shielding, i.e., EMI shielding) (Chung 2003).

Although continuous fibers are more effective than short fibers as a reinforcement (Wen and Chung 1999), they are not amenable to incorporation in a concrete mix and they are relatively expensive. Low cost is critical to the practical viability of cement-based materials. Thus, this paper is focused on short fibers in cement-based materials.

Although macroscopic steel fibers of around 1 mm diameter are used, the most effective fibers are usually microfibers of diameters ranging from 5 to 100 μm. For example, carbon fibers are typically around 10 μm in diameter (Chung 1994). Nanofibers of diameters typically around 0.1 μm are less effective than microfibers as a reinforcement, although they are more effective than microfibers for providing EMI shielding (due to the small diameter and the skin effect) (Chung 2001). In general, the smaller the fiber diameter is (which relates to a higher aspect ratio), the more difficult the fiber dispersion. Similarly, the smaller the fiber length is (which relates to a lower aspect ratio), the easier the fiber dispersion. This is due to the tendency for fibers of a small diameter or a long length to cling to one another.

The effectiveness of a fiber admixture for improving the structural or functional properties of cement-based materials is greatly affected by the degree of fiber dispersion. The attainment of a high degree of fiber dispersion is particularly critical when the fiber volume fraction is low. A low fiber volume fraction is usually preferred, because the material cost increases, the workability decreases, the air void content increases, and the compressive strength decreases as the fiber content increases (Chen and Chung 1996).

The fiber dispersion is enhanced by improving the hydrophylicity of the fibers, as the cement mix is water-based. The hydrophylicity can be controlled by surface treatment of the fibers prior to incorporation of the fibers in the cement mix (Fu et al. 1998a; Xu and Chung 2000). Furthermore, the fiber dispersion is affected by the admixtures that may be used along with the fibers. These admixtures may be fine particles [such as silica fume (Chung 2002), which has a typical particle size around 0.1 μm], the presence of which helps the fibers break loose from one another as mixing occurs (Chen et al. 1997; Chen and Chung 1995). Other admixtures may be polymers such as latex particle dispersions, which help the fiber-cement bond as well as the fiber dispersion (Chen et al. 1997; Fu and Chung 1997a,b; Chung 2004).

This paper provides a review of the dispersion of short fibers in cement-based materials. The methods for enhancing fiber dispersion, the factors that affect fiber dispersion, and the relationship of fiber dispersion with the properties of the cement-based materials are all considered. The use of special mixers to improve fiber dispersion is not considered in this review, because it increases the processing cost and limits the field applicability (due to the limited availability of special mixers in the field).

The use of microscopy to assess the degree of fiber dispersion is ineffective, as it can tell if fiber clumps are present, but it is insensitive to small differences in the degree of fiber dispersion (Akkaya et al. 2003).

Although the mechanical properties are more important to concrete applications than the electrical conductivity, the electrical conductivity can reflect the degree of fiber dispersion better than the mechanical properties. In cases where the fiber volume fraction is below the percolation threshold (the fiber volume fraction above which the fibers touch to form a continuous electrical conduction path), the greater the degree of fiber dispersion is, the higher the conductivity of the composite. This is because of the relatively long length of the conduction path within the matrix in the case of poor fiber dispersion, as illustrated in Fig. 1 (Cao and Chung 2001a). In the case of carbon fiber cement paste, the per-

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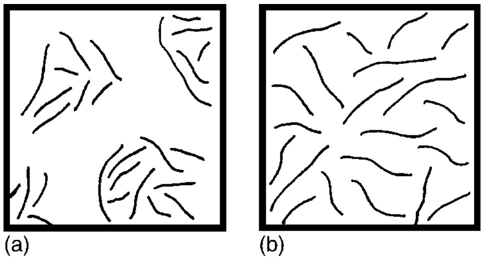

Fig. 1. Fiber dispersion below the percolation threshold: (a) poor dispersion; (b) good dispersion

colation threshold is between 0.5 and 1.0 volume % (Chen and Chung 1995). The mechanical properties depend on the fiber-matrix bonding and the amount of air voids, in addition to the degree of fiber dispersion. However, for fibers that are much more conductive than the matrix and are at volume fractions below the percolation threshold, the electrical conductivity of the cement-based material depends mostly on the degree of fiber dispersion. The greater the degree of fiber dispersion is, the higher the electrical conductivity of the cement-matrix composite. In contrast, above the percolation threshold, the electrical conductivity is higher when the fibers are segregated along the continuous conduction paths. Therefore, this paper uses volume electrical conductivity measurement below the percolation threshold to assess the degree of fiber dispersion on a relative scale. For this reason, the paper is focused on carbon fibers and steel fibers, which are electrically conductive.

A related technique is alternating current (AC) impedance measurement (Shui and Stroeven 1999), which is more costly and complicated than direct current (DC) resistance measurement. This paper addresses the DC method.

Effect of Admixtures Used along with Fibers

Admixtures used along with the short fibers in the cement mixture can cause an increase or decrease in the degree of fiber dispersion. Admixtures include silica fume, polymer particles, water-based dispersions, polymer water-based solutions, and silane.

Effect of Silica Fume

Silica fume is very fine noncrystalline silica produced by electric arc furnaces as a byproduct of the production of metallic silicon or ferrosilicon alloys. It is a powder with particles having diameters 100 times smaller than portland cement, i.e., mean particle size between 0.1 and 0.2 μm. The SiO_2 content ranges from 85 to 98%. Silica fume is pozzolanic.

Silica fume used as an admixture in a concrete mix has significant effects on the properties of the resulting material (Chung 2002). These effects pertain to the strength, modulus, ductility, vibration damping capacity, sound absorption, abrasion resistance, air void content, shrinkage, bonding strength with reinforcing steel, permeability, chemical attack resistance, alkali-silica reactivity reduction, corrosion resistance of embedded steel reinforcement, freeze-thaw durability, creep rate, coefficient of thermal expansion (CTE), specific heat, and thermal conductivity. In addition, silica fume improves the degree of fiber dispersion in mixes containing short microfibers (Chen et al. 1997; Park et al. 1999; Pigeon and Cantin 1998).

At a carbon fiber (15 μm diameter; ~5 mm length) content of 0.35% [below the percolation threshold (Chen and Chung 1995)], cement mortar exhibits an electrical resistivity of $1.59 \times 10^6\ \Omega.\mathrm{cm}$ (±4.5%) in the absence of silica fume (Cao and Chung 2001b). In the presence of silica fume (15% by mass of cement), the resistivity is reduced to $1.13 \times 10^6\ \Omega.\mathrm{cm}$ (±3.9%) (Table 1) (Cao and Chung 2001b). Consistent with the reduction in electrical resistivity is the increase in tensile strength from 1.04±0.11 to 1.45±0.11 MPa (Table 1) (Cao and Chung 2001b). The reduction in resistivity is an indication of an improvement in the degree of fiber dispersion.

Effect of Polymer Solution

Due to the need for water in a cement mix, polymer solutions based on water are more suitable than those based on other solvents. Methylcellulose is a polymer that is soluble in water.

At a carbon fiber (15 μm diameter; ~5 mm length) content of 0.35% [below the percolation threshold (Chen and Chung 1995)], in the absence of silica fume, the electrical resistivity of cement mortar is reduced from $1.59 \times 10^6\ \Omega.\mathrm{cm}$ (±4.5%) to 0.68

Table 1. Volume Electrical Resistivity and Tensile Strength of Cement Mortars Containing 0.35 Volume % Short Carbon Fibers (15 μm Diameter; ~5 mm Length): Silica Fume (SF) (Cao and Chung 2001b)

Organic admixture	Organic admixture amount (% by mass of cement)	Apparent water/cement ratio	Resistivity ($10^6\ \Omega \cdot \mathrm{cm}$)		Tensile strength (MPa)	
			Without SF	With SF	Without SF	With SF
—	0	0.350[b]	1.59(±4.5%)	1.13(±3.9%)	1.45±0.11	1.04±0.11
Methylcellulose	0.4	0.350[b]	0.68(±2.8%)	0.31(±3.1%)	2.26±0.08	2.36±0.06
Acrylic	10[a]	0.297	0.67(±6.9%)	0.55(±5.3%)	2.15±0.08	2.26±0.07
Acrylic	15[a]	0.270	0.56(±4.3%)	0.49(±5.1%)	2.49±0.13	2.54±0.11
Acrylic	20[a]	0.244	0.58(±6.0%)	0.51(±3.1%)	2.56±0.20	2.46±0.09
Styrene acrylic	10[a]	0.295	1.01(±4.9%)	0.93(±3.4%)	1.98±0.14	2.04±0.10
Styrene acrylic	15[a]	0.267	0.96(±4.0%)	0.85(±5.2%)	2.07±0.20	2.28±0.15
Styrene acrylic	20[a]	0.240	0.83(±5.1%)	0.77(±4.6%)	2.23±0.12	2.41±0.09
Latex	10[a]	0.298	0.66(±5.8%)	0.56(±4.7%)	2.02±0.15	2.19±0.13
Latex	15[a]	0.272	0.70(±9.3%)	0.63(±3.7%)	2.25±0.17	2.49±0.11
Latex	20[a]	0.246	0.89(±7.7%)	0.70(±6.1%)	2.08±0.20	2.40±0.08

[a] Including mass of water in the dispersion.

[b] Same as the true water/cement ratio.

$\times 10^6\ \Omega$.cm (±2.8%) upon addition of methylcellulose (0.4% by mass of cement) (Table 1) (Cao and Chung 2001b). Consistent with the reduction in resistivity is the increase in tensile strength from 1.04±0.11 to 2.26±0.08 MPa (Table 1) (Cao and Chung 2001b).

The reduction in resistivity is an indication of an improvement in the degree of fiber dispersion. The presence of methylcellulose, which is not conductive, presumably at the fiber-matrix interface, is expected to increase the resistivity of the mortar. In spite of this, the resistivity of the fiber mortar is decreased by the methylcellulose addition. The increase in tensile strength upon methylcellulose addition is consistent with the increase in fiber-matrix shear bond strength upon methylcellulose addition (Fu and Chung 1998a).

Effect of Polymer Particle Dispersions

Due to the need for water in a cement mix, polymer particle dispersions based on water are more suitable than those based on other liquids. The polymers used in the dispersions are not soluble in the liquids used. As most polymers are not soluble in water, the choice of polymers for dispersions is wide compared to that of polymers for solutions.

At a carbon fiber (15 μm diameter; ~5 mm length) content of 0.35%, in the absence of silica fume, the electrical resistivity of the cement mortar is reduced from $1.59\times 10^6\ \Omega$.cm (±4.5%) to $0.58\times 10^6\ \Omega$.cm (±6.0%) upon addition of acrylic water-based dispersion (20% by mass of cement) (Table 1) (Cao and Chung 2001b). Latex and styrene acrylic water-based dispersions (also 20% by mass of cement) are less effective than acrylic dispersion, as they reduce the resistivity to $0.89\times 10^6\ \Omega$.cm (±7.7%) and $0.83\times 10^6\ \Omega$.cm (±5.1%), respectively (Table 1). As polymer particle dispersions are used in much larger amounts than methylcellulose, the cost of using the dispersions is relatively high.

The presence of polymer at the fiber-matrix interface is suggested by the increase in the fiber-matrix shear bond strength upon latex addition (Fu and Chung 1998a). In spite of this, the polymer dispersion addition causes the resistivity of the fiber mortar to be decreased by the addition of the polymer dispersion. This means that the addition of the polymer dispersion improves the degree of fiber dispersion.

In the absence of fiber, both flexural toughness and strength increase monotonically with increasing content of polymer particle dispersion (latex) (Chen et al. 1997). However, in the presence of fiber (e.g., carbon fiber), the flexural toughness decreases monotonically with increasing content of polymer particle dispersion, because the degree of fiber dispersion decreases with increasing content of polymer particle dispersion (Chen et al. 1997).

Fig. 2 (Chen et al. 1997) shows the flexural toughness of cement pastes containing various amounts of latex, with 0 and 0.53 volume % carbon fibers. The flexural toughness, as measured under three-point bending, increases monotonically with increasing latex/cement ratio when fibers are absent, but decreases monotonically with increasing latex/cement ratio when fibers are present. At any latex/cement ratio, fiber addition greatly increases the toughness.

Upon increase of the amount of acrylic dispersion from 10 to 20% by mass of cement, the electrical resistivity of carbon fiber mortar is decreased from $0.67\times 10^6\ \Omega\cdot$cm (±6.9%) to $0.58\times 10^6\ \Omega\cdot$cm (±6.0%) (Table 1) (Cao and Chung 2001b). However, for the case of latex in place of acrylic, the resistivity is increased from $0.66\times 10^6\ \Omega\cdot$cm (±5.8%) to $0.89\times 10^6\ \Omega\cdot$cm (±7.7%) (Table 1). In the absence of fiber, an increase in the polymer content causes the resistivity to increase, as shown at least for the case of the polymer being latex (Fu and Chung 1996). Therefore, the decrease in resistivity of carbon fiber mortar as the acrylic dispersion content is increased from 10 to 20% by mass of cement is attributed to an increase in the degree of fiber dispersion. However, the dependence of the resistivity of carbon fiber mortar with latex content is opposite in trend and the flexural toughness results in Fig. 2 point to a decrease in the degree of fiber dispersion with increasing latex content. Hence, the combination of electrical and mechanical results indicate that the degree of fiber dispersion increases with increasing acrylic dispersion content, but decreases with increasing latex dispersion content. Nevertheless, the use of a polymer particle dispersion (whether acrylic or latex) gives a higher degree of fiber dispersion than the use of no dispersion, at least for the range of dispersion content from 10 to 20% by mass of cement.

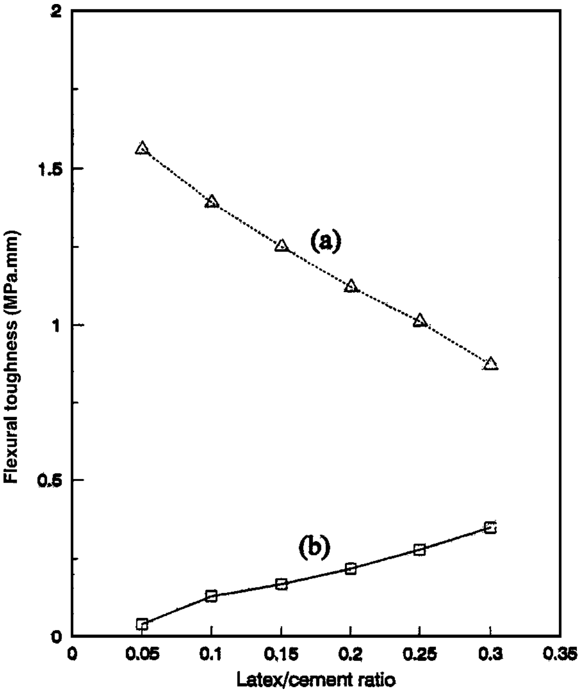

Fig. 2. Effect of latex/cement ratio on flexural toughness when the cement paste contains: (a) 0.53 volume % carbon fibers; (b) no fibers

Effect of Silica Fume in Combination with Polymer Solution

Both silica fume and methylcellulose solution, when used separately, help the fiber dispersion. The combined use of these two admixtures is even more helpful. The combined use of silica fume (15% by mass of cement) and methylcellulose (0.4% by mass of cement) causes the resistivity to decrease from $1.59\times 10^6\ \Omega$.cm (±4.5%) to $0.31\times 10^6\ \Omega$.cm (Table 1) (Cao and Chung 2001b). This decrease is more than that attained by using silica fume alone [decreased to $1.13\times 10^6\ \Omega$.cm (±3.9%)] or that attained by methylcellulose alone [decreased to $0.68\times 10^6\ \Omega$.cm (±2.8%)]. On the other hand, silica fume in combination with latex dispersion does not work, due to low workability (Fu and Chung 1998a).

Effect of Silane

Silane is molecular, but it is not a polymer. Due to its hydrophylic nature, silane is effective as an admixture for improving the degree of fiber dispersion, as is shown for steel fibers (60 μm diameter; 5 mm length). The electrical resistivity of steel fiber (0.05 volume %, much below the percolation threshold) mortar is decreased from $2.03\times10^6\ \Omega.\mathrm{cm}$ (±5.3%) to $1.65\times10^6\ \Omega.\mathrm{cm}$ (±3.9%) upon addition of silane (aqueous amino vinyl silane, chosen due to its stability in aqueous systems) (Cao and Chung 2001b). In the absence of fiber, silane addition has a negligible effect on the resistivity of mortar (Cao and Chung 2001b). Thus, the decrease in resistivity of the steel fiber mortar upon silane addition is attributed to increase in the degree of fiber dispersion.

Effect of Fiber Surface Treatments

Ozone Treatment

Fiber surface treatments that improve the wettability of the fiber by water are useful for improving the fiber dispersion in cement, in addition to improving the fiber-matrix bond (Fu et al. 1998a). At a carbon fiber content of 0.24 volume % (below the percolation threshold), the electrical resistivity of the cement mortar is decreased from 3.62×10^3 to $3.27\times10^3\ \Omega.\mathrm{cm}$ upon surface treatment of the carbon fibers by ozone prior to incorporation of the fibers in the cement mix (Fu et al. 1998a). Even though ozone treatment increases the contact resistivity between fiber and cement (Fu et al. 1998a), the volume resistivity of the fiber mortar is decreased by the ozone treatment. Thus, the decrease in volume resistivity indicates an increase in the degree of fiber dispersion.

Ozone treatment involves exposure to an O_2-O_3 mixture with 0.6 volume % O_3 at 160°C for 5 min (Fu et al. 1998a; Lu et al. 1998). It results in oxygen-containing surface functional groups and is effective for improving the wettability of carbon and steel fibers (Lu et al. 1998). A contact angle of 0° is attained by ozone treatment of carbon fibers (Lu et al. 1998).

Ozone treatment is effective for increasing the tensile strength, modulus, and ductility, as well as the compressive strength, modulus, and ductility, of both carbon fiber (15 μm diameter; 0.5 volume %) cement paste and carbon nanofiber (0.1 μm diameter; 0.5 volume %) cement paste (Chung 2001; Fu and Chung 1998b). The nanofiber paste is inferior to the fiber paste in terms of these properties. The strain-sensing ability of carbon fiber cement is also improved by ozone treatment of the fibers (Fu et al. 1998b).

Treatments with Acetone, Hydrochloric Acid, and Sodium Hydroxide

Acetone treatment involves immersion in acetone for 1 h. It is effective for improving the wettability of steel and polyethylene fibers (Lu et al. 1998). For polyethylene fibers, hydrochloric acid treatment (immersion in 1.0 N HCl solution for 24 h) and sodium hydroxide treatment (immersion in 1.0 N NaOH solution for 24 h) are more effective than acetone treatment (Lu et al. 1998).

Silane Treatment

Instead of using silane as an admixture, silane can be used to coat fibers prior to incorporation of the fibers in the cement mix. However, the use of silane to coat steel fibers (60 μm diameter; 5 mm length) is less effective than the use of silane as an admixture for improving the degree of fiber dispersion (Cao and Chung 2001b).

Silane coating has been applied to carbon fibers for the purpose of improving the tensile strength (Xu and Chung 2000). However, the effect of the silane coating on the degree of carbon fiber dispersion has not been assessed.

Conclusion

Short fibers are used as admixtures in cement-based materials for structural and functional reasons. Dispersion of the fibers is important and is particularly challenging for microfibers at a small volume fraction. Assessment of the degree of fiber dispersion by microscopy is ineffective. In this paper, the degree of dispersion of short microfibers (such as carbon and steel fibers) in cement mortar or cement paste is assessed by measurement of the volume electrical resistivity when the fibers are electrically conductive and are at a volume fraction below the percolation threshold. The degree of dispersion is improved by the use of admixtures such as silica fume, acrylic particle dispersion, methylcellulose solution, and silane. Acrylic particle dispersion is more effective than latex particle dispersion. The degree of fiber dispersion is also improved by the use of fiber surface treatment such as ozone treatment.

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