Disc. 103-M38/From the Sept.-Oct. 2006 *ACI Materials Journal*, p. 340

Monitoring Fiber Dispersion in Fiber-Reinforced Cementitious Materials: Comparison of AC-Impedance Spectroscopy and Image Analysis. Paper by Nilufer Ozyurt, Leta Y. Woo, Thomas O. Mason, and Surendra P. Shah

Discussion by D. D. L. Chung

Composite Materials Research Laboratory, University at Buffalo, State University of New York, Buffalo, N.Y.

APPROACH OF AUTHORS

The authors' notion that steel fibers are insulating under DC and conductive under AC is flawed. The use of NaCl solution (an ionic conductor) for passing current, so electron injection is impossible and the electrons' role is limited. Because steel is an electronic conductor, the sensitivity for the fiber is low. Even at high frequencies that hinder the ionic response, electronic conduction is limited. At low frequencies, ions contribute, making the relative role of electrons even less. The oxide on the steel and the polarization layer at the fiber-matrix interface are ineffective ionic conductors. If an electronic conductor is used for passing current, electronic conduction will be more significant and the method will be more sensitive. Silver paint functions after drying. Aqueous solutions are wet. Thus, conductive pastes are more practical than solutions.

The fiber dispersion is particularly poor for 1 vol.% fiber (with regions without fibers, Fig. 3(a)). The authors cannot distinguish dispersion degrees corresponding to different fiber contents, as shown by dispersion factor (DF) data and their scatter (Table 2 and Fig. 7).

The oxide on the fiber may interact with ions. The interaction depends on the frequency and may help ionic conduction.[33] Ozone treatment of carbon fiber provides functional groups,[34] thereby helping DC ionic conduction.[33] Moreover, the dielectric constant is increased by carbon fiber.[35] A small dielectric effect is indicated by the effect of the steel fiber on the imaginary part of the impedance (Fig. 1). The higher the frequency is, the smaller the skin depth is. Thus, the fiber's electronic conduction ability diminishes with increasing frequency.

Mechanisms may include: 1) the frequency-dependent interaction of the fiber surface with the ions; 2) the decreasing role of ionic conduction and the consequent increasing role of electronic conduction (hence increasing the contribution of the fiber to conduction) as the frequency increases; and 3) the decreasing conduction ability of the fiber as the frequency increases.

DC ELECTRICAL RESISTIVITY

The DC contact resistivity (silver paint) of the fiber-cement interface is 10^6 Ω.cm^2 (10^5 Ω.in.^{2}) for steel fiber (diameter 60 μm [2.4×10^{-3} in.])[36-38] and 10^5 Ω.cm^2 (10^4 Ω.in.^{2}) for carbon fiber (diameter 15 μm [5.9×10^{-4} in.]).[34,39,40] For the interface between steel reinforcing bar and concrete, it is 10^7 Ω.cm^2 (10^6 Ω.in.^{2}).[41,42] These values are above 10^{-2} Ω.cm^2 (10^{-3} Ω.in.^{2}) for the interface between adhesively bonded steel surfaces[43] and 10^{-4} Ω.cm^2 (10^{-5} Ω.in.^{2}) for the interface between bonded copper surfaces.[44,45] Nevertheless, the interface is not insulating. This is consistent with the DC volume resistivity decreasing with increasing fiber content.[46-48]

The authors imply that the DC method is insensitive to the fiber dispersion. The DC volume resistivity (silver paint) is sensitive, as shown for steel and carbon fibers.[33,49-52] For steel fiber (diameter 60 μm [2.4×10^{-3} in.]) below the percolation threshold, the resistivity is decreased by silica fume.[50] For steel fiber (diameter 60 μm [2.4×10^{-3} in.]) below the percolation threshold, the resistivity is decreased by silane.[49] Thus, DC resistivity reflects the effect of admixtures. DC is also attractive in its simpler instrumentation compared with AC.

Below the percolation threshold, the lower the resistivity is, the better the fiber dispersion. Above the threshold, the resistivity is not reliable for indicating fiber dispersion because percolation may be enhanced or degraded by fiber segregation. Below the threshold, differences in the fiber dispersion due to admixtures, fiber surface treatments, and curing ages have been shown by DC resistivity (silver paint).[33,49-52] The authors' percolation threshold is not reported. Equation (1), however, suggests the assumption that the threshold is not exceeded.

DISPERSION FACTOR CALCULATION LIMITATION

The authors describe DF as an upper limit for the fraction of dispersed fibers and calculate it based on AC results and Eq. (1), (2), and (4). The measured AC conductivity relative to that of the matrix is not reported, though this ratio is in the calculation. The DC resistances of cements with and without fiber are close (Fig. 1), suggesting that the fiber contributes little to conduction.

The ratio of the composite (high-frequency cusp) AC conductivity to the matrix DC conductivity is 1.4 (Fig. 1), which is low compared with the ratio of the DC composite conductivity (silver paint) to the DC matrix conductivity given in the following for other steel fibers.[50,53]

For silica fume cement paste containing 0.03 vol.% (below the percolation threshold of 0.3 vol.%[53]) steel fiber of diameter 8 μm (3.2×10^{-4} in.), DC conductivity ratio equals 7.6[53] and DF equals 0.56. At 0.06 vol.% fiber, the ratio equals 14[53] and DF equals 0.52. At 0.09 vol.% fiber, the ratio equals 136[53] and DF equals 6.8, which exceeds 1. Thus, the DF calculation is invalid when the conductivity ratio exceeds a value between 14 and 136 and is not useful for applications that require high conductivity.

For silica fume cement paste containing 0.1 vol.% (below the percolation threshold[47]) steel fiber with a diameter of 60 μm (2.4×10^{-3} in.), the conductivity ratio equals 10.9[50,54] and DF equals 22.2. In the absence of silica fume, 0.1 vol.% fiber gives a ratio of 6.3[50,54] and a DF of 11.9. For 0.2 vol.% fiber (below the percolation threshold[47]) in the presence of silica fume, the ratio equals 19.1[50] and DF equals 20.3. Hence, DF exceeds 1 when the conductivity ratio is 6.3 or above, even though the percolation threshold has not been exceeded.

The low conductivity ratio of 1.4 for cement paste containing the authors' 160 μm (6.3×10^{-3} in.) diameter steel fiber means that this fiber is ineffective for raising the conductivity (NaCl solution). The steel fibers of 60 μm

(2.4×10^{-3} in.)[50] and 8 μm (3.2×10^{-4} in.) diameter,[53] however, are effective in raising the conductivity (silver paint).

The aforementioned conductivity ratio of 1.4 (Fig. 1) corresponds to a DF of 0.30 (for 1 vol.% fiber), which is below 0.81 in Table 2, suggesting inconsistency within the paper.

FIBER CLUMPING AND DEPLETION

Fiber clumping is more significant when the fiber is concentrated. Local fiber depletion is more significant when the fiber is sparse (economical) (Fig. 3(a)). The attainment of fiber dispersion below the percolation threshold is more challenging than that above the threshold. The large data scatter (for example, 20%) among DF values measured in three directions (Table 2) suggests poor fiber dispersion. At the same fiber content, fiber clumping is more likely for a smaller diameter fiber and fiber depletion is more likely for a larger diameter fiber. The authors' fiber is larger than the 60 μm (2.4×10^{-3} in.) diameter steel fiber,[49] the 8 μm (3.2×10^{-4} in.) diameter steel fiber[53] and the 15 μm (5.9×10^{-4} in.) diameter carbon fiber of prior DC work.[48] Yet the authors address fiber clumping, but not fiber depletion.

Discerning subtle differences in the fiber dispersion, such as those resulting from admixtures and fiber surface treatments, is more practically useful than discerning gross differences. Small differences cannot be observed by microscopy, as shown by the large scatter in CF data (Table 2 and Fig. 7). Image analysis is more difficult as the fiber diameter decreases.

Table 2 shows that DF and 1 – CF are close, both at 0.7 to 0.9, though they may not be close outside this range. The authors conclude that AC and image analysis results agree within experimental uncertainty, which is large (for example, ±34%).

EFFECT OF WATER

The DC conductivity in carbon fiber cement (below the percolation threshold; not wet) is dominated by electronic conduction, as shown by DC resistivity (silver paint and salt solution contacts).[33] Little ionic conduction contribution means that water affects the DC resistivity negligibly. This is supported by the slight increase of the resistivity (silver paint) of carbon fiber cement with increasing curing age[55] and heating time.[56] This adds to the attraction of DC resistance (with an electronic conductor as contacts) for indicating fiber dispersion.

TWO-PROBE AND FOUR-PROBE METHODS

The two-probe method suffers from the inclusion of the contact impedance, whereas the four-probe method essentially excludes this impedance. Contacts that are similarly made can be different in quality. Therefore, two-probe data are more scattered. Contact degradation causes the two-probe resistance to increase.[57] That the DC four-probe resistance is close to the two-probe AC low-frequency cusp resistance (Fig. 1) supports the two-probe AC resistance. The two-probe and four-probe values may differ, however, when the composite is more conductive.

SUMMARY

Weaknesses relate to AC methodology and explanation, low sensitivity for fiber dispersion, limited applicability of the DF calculation, and ignoring prior DC work.

REFERENCES

33. Wen, S., and Chung, D. D. L., "The Role of Electronic and Ionic Conduction in the Electrical Conductivity of Carbon Fiber-Reinforced Cement," *Carbon*, V. 44, No. 11, 2006, pp. 2130-2138.

34. Fu, X.; Lu, W.; and Chung, D. D. L., "Ozone Treatment of Carbon Fiber for Reinforcing Cement," *Carbon*, V. 36, No. 9, 1998, pp. 1337-1345.

35. Wen, S., and Chung, D. D. L., "Effect of Admixtures on the Dielectric Constant of Cement Paste," *Cement and Concrete Research*, V. 31, No. 4, 2001, pp. 673-677.

36. Fu, X., and Chung, D. D. L., "Bond Strength and Contact Electrical Resistivity between Cement and Stainless Steel Fiber: Their Correlation and Dependence on Fiber Surface Treatment and Curing Age," *ACI Materials Journal*, V. 94, No. 3, May-June 1997, pp. 203-208.

37. Fu, X., and Chung, D. D. L., "Nondestructive Bond Strength Testing by Contact Electrical Resistivity Measurement," *Innovations in Nondestructive Testing of Concrete*, SP-168, S. Pessiki and L. Olsen, eds., American Concrete Institute, Farmington Hills, Mich., 1997, pp. 295-317.

38. Fu, X., and Chung, D. D. L., "Effect of Polymer Admixtures to Cement on the Bond Strength and Electrical Contact Resistivity between Steel Fiber and Cement," *Cement and Concrete Research*, V. 26, No. 2, 1996, pp. 189-194.

39. Fu, X.; Lu, W.; and Chung, D. D. L., "Improving the Bond Strength between Carbon Fiber and Cement by Fiber Surface Treatment and Polymer Addition to Cement Mix," *Cement and Concrete Research*, V. 26, No. 7, 1996, pp. 1007-1012.

40. Fu, X., and Chung, D. D. L., "Contact Electrical Resistivity between Cement and Carbon Fiber: Its Decrease with Increasing Bond Strength and Its Increase During Fiber Pull-Out," *Cement and Concrete Research*, V. 25, No. 7, 1995, pp. 1391-1396.

41. Fu, X., and Chung, D. D. L., "Effects of Water-Cement Ratio, Curing Age, Silica Fume, Polymer Admixtures, Steel Surface Treatments, and Corrosion on Bond between Concrete and Steel Reinforcing Bars," *ACI Materials Journal*, V. 95, No. 6, Nov.-Dec. 1998, pp. 725-734.

42. Fu, X., and Chung, D. D. L., "Linear Correlation of Bond Strength and Contact Electrical Resistivity between Steel Rebar and Concrete," *Cement and Concrete Research*, V. 25, No. 7, 1995, pp. 1397-1402.

43. Poeller, M., and Chung, D. D. L., "Effect of Heating on the Structure of an Adhesive Joint, as Indicated by Electrical Resistance Measurement," *Journal of Adhesion*, V. 79, No. 6, 2003, pp. 549-558.

44. Kim, K. D., and Chung, D. D. L., "Electrically Conductive Adhesive and Soldered Joints under Compression," *Journal of Adhesion Science and Technology*, V. 19, No. 11, 2005, pp. 1003-1023.

45. Kim, K. D., and Chung, D. D. L., "Effect of Heating on the Electrical Resistivity of Conductive Adhesive and Soldered Joints," *Journal of Electronic Materials*, V. 31, No. 9, 2002, pp. 933-939.

46. Wen, S., and Chung, D. D. L., "Electromagnetic Interference Shielding Reaching 70 dB in Steel Fiber Cement," *Cement and Concrete Research*, V. 34, No. 2, 2004, pp. 329-332.

47. Wen, S., and Chung, D. D. L., "Effect of Fiber Content on the Thermoelectric Behavior of Cement," *Journal of Materials Science*, V. 39, No. 13, 2004, pp. 4103-4106.

48. Chen, P.; Fu, X.; and Chung, D. D. L., "Microstructural and Mechanical Effects of Latex, Methylcellulose, and Silica Fume on Carbon Fiber Reinforced Cement," *ACI Materials Journal*, V. 94, No. 2, Mar.-Apr. 1997, pp. 147-155.

49. Cao, J., and Chung, D. D. L., "Improving the Dispersion of Steel Fibers in Cement Mortar by the Addition of Silane," *Cement and Concrete Research*, V. 31, No. 2, 2001, pp. 309-311.

50. Wen, S., and Chung, D. D. L., "Seebeck Effect in Steel Fiber Reinforced Cement," *Cement and Concrete Research*, V. 30, No. 4, 2000, pp. 661-664.

51. Cao, J., and Chung, D. D. L., "Carbon Fiber Reinforced Cement Mortar Improved by Using Acrylic Dispersion as an Admixture," *Cement and Concrete Research*, V. 31, No. 11, 2001, pp. 1633-1637.

52. Chung, D. D. L., "Dispersion of Short Fibers in Cement," *Journal of Materials in Civil Engineering*, V. 17, No. 4, 2005, pp. 379-383.

53. Wen, S., and Chung, D. D. L., "Electromagnetic Interference Shielding Reaching 70 dB in Steel Fiber Cement," *Cement and Concrete Research*, V. 34, 2004, pp. 329-332.

54. Wen, S., and Chung, D. D. L., "Carbon Fiber-Reinforced Cement as a Thermistor," *Cement and Concrete Research*, V. 29, No. 6, 1999, pp. 961-965.

55. Fu, X., and Chung, D. D. L, "Carbon Fiber Reinforced Mortar as an Electrical Contact Material for Cathodic Protection," *Cement and Concrete Research*, V. 25, No. 4, 1995, pp. 689-694.

56. Cao, J., and Chung, D. D. L., "Role of Moisture in the Seebeck Effect in Cement-Based Materials," *Cement and Concrete Research*, V. 35, No. 4, 2005, pp. 810-812.

57. Wen, S., and Chung, D. D. L., "Piezoresistivity-Based Strain Sensing in Carbon Fiber Reinforced Cement," *ACI Materials Journal*, V. 104, No. 2, Mar.-Apr. 2007, pp. 171-179.

AUTHORS' CLOSURE

Points of agreement with discussion

1) The authors agree that there are other techniques, including electrical resistivity[48,49,51,52] that are sensitive to fiber dispersion in fiber-reinforced concrete.

2) Neither AC-IS nor image analysis see substantive differences in dispersion between the three experimental fiber loadings in the original paper, within experimental error. This was not the thrust of the paper, however, but rather that the two techniques are in mutual agreement. The authors have since evaluated composites with much larger, detectable differences in fiber clumping by microstructural evaluation and in dispersion factor (DF) by AC-IS, again with good agreement between the two methods.[58]

3) Four-point measurements are preferred for DC measurements to obviate electrode polarization contributions. Four-point AC-IS measurements would also be desirable; however, large electrode impedances (at inner electrodes) and associated experimental artifacts preclude meaningful four-point AC-IS measurements in cement-based systems.[59]

4) Figure 1 of the original paper does not agree with the DFs plotted in Fig. 7. A citation[21] was inadvertently omitted from the caption of Fig. 1, which shows sample Nyquist plots from a previous work instead of the work in question, where no measurements of plain cement matrix were made. To correct the omission (of supporting data), Fig. A shows a typical *y*-direction result (1 vol% steel fiber specimen, *w/c* = 0.3, age = 7 days), from which a DF of 0.88 can be calculated. Averaging the DFs in all three directions yields 0.81 ± 0.17 (as in Fig. 7(a) of the original paper).

5) Fiber contact resistivity is important and can be readily measured. The value reported in the Discussion (106 Ω.cm^2) agrees well with values obtained by electrochemical IS (EIS), 10^5 to 10^6 Ω.cm^2.[60] A similar value was employed for the coating elements on conductive fibers in pixel-based finite difference computer modeling of a single steel fiber in cement.[61] The results show negligible current flow to/through the fiber at DC and low AC frequencies, but high current flow to/through the fiber at frequencies corresponding to the cusp frequency (~10^5 Hz), owing to displacement currents shorting out the resistive oxide layer. This is the basis of the so-called frequency-switchable coating impedance effect at the heart of the AC-IS technique.

6) Percolation is also very important. Based upon literature estimates of percolation threshold (PT) in FRCs,[62,63] at an aspect ratio of 37.5 (as in the original paper), the 1 vol% specimen is below PT, and the 2 vol% specimen is likely below the PT (lower bound estimated at 1.75 vol%, upper bound at 3.25 vol%). Although the 4 vol% FRC should be at or above the PT, no precipitous decrease in resistance was observed. The direction-averaged R_{DC} values for the three *w/c* = 0.35 composites were 227 Ω, 226 Ω, and 239 Ω, respectively. This is attributable to the fiber diameter (0.16 mm [0.0062 in.]) being much larger than the median cement particle size (~10 μm); cement particles coat the fibers and prohibit direct contact between the fibers, thereby forestalling the PT (as discussed elsewhere[64]).

Points of disagreement with discussion

1) The AC-IS DF approach has been carefully validated in several ways. First, as mentioned previously, pixel-based finite difference computer calculations successfully simulated the frequency-switchable coating behavior of a single steel fiber.[61] Second, steel and glass ball bearing (cement) composites were investigated by AC-IS.[64] DC conductivity decreased with increasing volume fraction of ball bearings, whether steel or glass (it made no difference), in a manner consistent with the Meredith-Tobias mixing law for insulating spheres, whereas the AC conductivity (high-frequency cusp) increased with the volume fraction of steel ball bearings in a manner consistent with the Meredith-Tobias mixing law for conducting spheres. Third, large diameter (0.5 mm [0.019 in.]) steel fibers were placed by hand in cement matrix composites to avoid percolation and achieve a random fiber loading.[64] The AC-IS results could be fully explained on the basis of the intrinsic conductivity approach, taking into account the aspect ratio of fibers and their volume fraction. Finally, in the original paper, both AC-IS and image analysis of FRCs were compared and found to be in good agreement regarding the degree of fiber clumping.

2) Dispersion factor cannot be calculated based upon DC resistance measurements, as attempted in the Discussion. The AC-IS method is unique in providing the necessary high-frequency cusp resistance from which the DF can be calculated.[64,65] This is only possible at the associated frequency (approximately 105 Hz), and is not available to DC resistance measurements.

3) The AC-IS DF approach is also unique in providing a normalization process (the ratio of two cusp resistances), which can account for differences in matrix conductivity from composite-to-composite.[64,65] In contrast, there is an unavoidable convolution of factors (fiber-induced changes, matrix conductivity changes) in DC measurements.

4) There is little difference electrochemically and functionally between ionic and electronic electrodes. In each instance there is a transition from an electronic conductor (steel in the aqueous electrode, silver in the silver electrode) to an ionic conductor (the pores of the cement matrix), with or without an intervening ionic solution of negligible resistance. Furthermore, the small amplitude alternating currents in two-point AC-IS obviate the problem of electrode polarization, and allow deconvolution of electrode polarization and bulk response (separate arcs).

To support this claim, a 2.5 x 2.5 x 10 cm (1 x 1 x 4 in.) specimen was made with 0.35 vol% steel fibers (2 mm [0.078 in.] long, 60 μm diameter) in cement matrix (*w/c* = 0.4) and maintained at 100% relative humidity until testing at 3

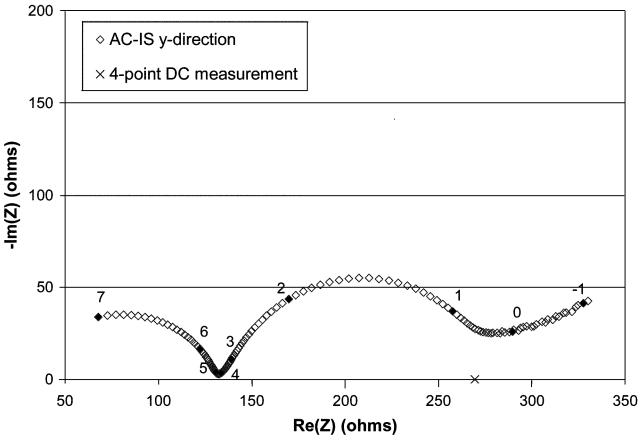

Fig. A—Typical y*-direction results for actual specimen in original paper of composite with 1 vol% steel fiber at* w/c *= 0.3 and 7 days of hydration. Four-point DC resistance is also shown on* Re(Z) *axis.*

days of hydration. Two-point AC-IS was performed with stainless steel electrodes (5 cm x 6 cm x 3 mm [2 x 2.36 x 0.118 in.]) lightly pressed against sponges (2.5 cm x 2.5 cm x 3 mm) saturated with 1 M NaCl solution, pressed against the two ends of the specimen. Four-point DC measurements were also made, with the inner electrodes at 3 cm (1.181 in.) spacing (3.5 cm [1.377 in.] from each end) with wrapped stainless steel wires secured by adhesive tape around the specimen. Subsequently, the sample ends were allowed to briefly dry (1 to 2 minutes) and silver paint was applied on the entire end surfaces to approximately 0.1 mm (0.003 in.) thickness, with a piece of stainless steel wire attached to each surface using a generous amount of silver paint. AC-IS and four-point DC measurements were made as soon as the silver paint was dry to the touch. The results are shown in Fig. B, where virtually identical bulk arcs were obtained for both types of electrodes, and in excellent agreement with the four-point DC resistances obtained with either type of electrode.

5) The Discussion statement that "…DC conductivity in carbon fiber cement (below the percolation threshold; not wet) is dominated by electronic conduction" is not correct. If truly below percolation (inter-fiber spacing greater than electron tunneling distances), conduction must proceed through the intervening cement, which is dominated by ionic conduction. Wilkosz and Young[66] documented resistivities in the 10^{11} to 10^{12} (Ω.cm) range for ordinary portland cement paste in the dried state. When resaturated (93% relative humidity), there was a six order of magnitude drop in resistivity,

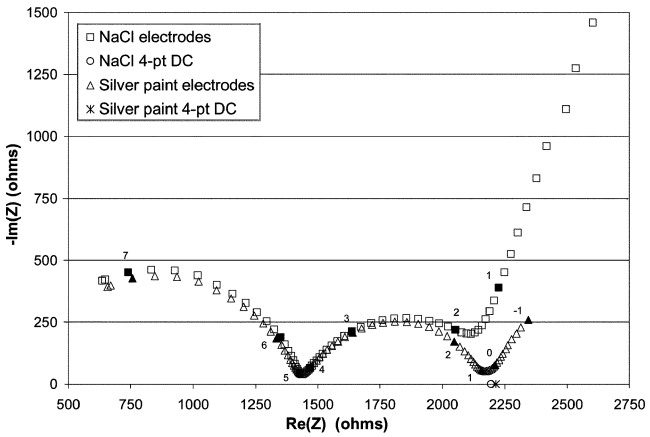

Fig B—Nyquist plots and four-point DC resistances of identical composite specimen tested using aqueous electrodes (NaCl solution/stainless steel) and silver paint electrodes.

confirming dominant ionic conduction via water-filled micro- and meso-pores.

6) Drying at electrode interfaces can be problematic for conductivity measurements, including silver paint electrodes. In work for this closure document, when silver end electrodes were allowed to dry, the agreement between AC and DC measurements became compromised. The authors previously documented drying-induced electrode artifacts in cement-based materials via AC-IS.[60] Such changes are obviated by the aqueous electroding scheme, which maintains pore-saturation beneath the electrodes throughout the experiments.

7) Electrical characterization of fiber dispersion in FRCs should be corroborated by careful microstructural examination/analysis of fiber dispersion in the identical specimens. The Discussion statement that "The fiber distribution is particularly poor [in Fig. 3(a)]…" is unsubstantiated; the higher fraction of fiber-free zones in the 1 vol% specimen is due to its relatively smaller fiber loading. In fact, the dispersion is no worse than in the other specimens. This was the thrust and central contribution of the authors' original work, that is, that AC-IS-based dispersion factors are in good agreement with fiber clumping factors derived by statistical image analysis. The authors are unaware of comparable electrical property-microstructure correlations involving DC resistivity measurements.

REFERENCES

58. Wansom, S., "Electrical Impedance Characterization of Ceramic-Matrix Composites with Various Inclusions," PhD thesis, Northwestern University, Evanston, Ill., 2006.

59. Mason, T. O.; Ford, S. J.; Shane, J. D.; Hwang, J.-H.; and Edwards, D. D., "Experimental Limitations in Impedance Spectroscopy of Cement-Based Materials," *Advances in Cement Research*, V. 10, No. 4, 1998, pp. 143-150.

60. Ford, S. J.; Shane, J. D.; and Mason, T. O., "Assignment of Features in Impedance Spectra of the Cement Paste/Steel System," *Cement and Concrete Research*, V. 28, No. 12, 1998, pp. 1737-1751.

61. Torrents, J. M.; Mason, T. O.; and Garboczi, E. J., "Impedance Spectra of Fiber-Reinforced Cement-Based Composites: A Modeling Approach," *Cement and Concrete Research*, V. 30, 2000, pp. 585-592.

62. Sundararajakumar, R. R.; and Koch, D. L., "Electrical Conductivity of Isotropic Fibre Suspensions," *Proceedings of the Royal Society of London*, V. 455, 1999, pp. 1923-1930.

63. Celzard, A.; McRae, E.; Deleuze, C.; Dufort, M.; Furdin, G.; and Mareche, J. F., "Critical Concentration in Percolating Systems Containing a High-Aspect-Ratio Filler," *Physical Review*, V. 53, No. 10, 1996, pp. 6209-6214.

64. Campo, M. A.; Woo, L. Y.; Mason, T. O.; and Garboczi E. J., "Frequency-Dependent Electrical Mixing Law Behavior in Spherical Particle Composites," *Journal of Electroceramics*, V. 9, 2002, pp. 49-56.

65. Hixson, A. D.; Woo, L. Y.; Campo, M. A.; Mason, T. O.; and Garboczi, E. J., "Intrinsic Conductivity of Short Conductive Fibers in Composites by Impedance Spectroscopy," *Journal of Electroceramics*, V. 7, 2001, pp. 189-195.

66. Wilkosz, D. E., and Young, J. F., "Effect of Moisture Adsorption on the Electrical Properties of Hardened Portland Cement Compacts," *Journal of the American Ceramic Society*, V. 78, No. 6, 1995, pp. 1673-1679.