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# Electric polarization in carbon fiber-reinforced cement

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**Abstract**

Electric polarization induced an increase of the measured electrical resistivity of carbon fiber-reinforced cement paste during resistivity measurement. The effect was diminished by increasing the conductivity of the cement paste through the use of carbon fibers that were more crystalline, the increase of the fiber content, or the use of silica fume instead of latex as an admixture. Intercalation of crystalline fibers further increased the conductivity of the composite, but it increased the extent of polarization. Voltage polarity switching effects were dominated by the polarization of the sample itself when the four-probe method was used, but were dominated by the polarization at the contact–sample interface when the two-probe method was used. Polarization reversal was faster and more complete for the latter. © 2001 Elsevier Science Ltd. All rights reserved.

*Keywords:* Fiber reinforcement; Cement paste; Electrical properties; Silica fume; Polarization

## 1. Introduction

The electrical behavior of carbon fiber-reinforced cement is relevant to the use of this material for strain sensing [1–7], which is important for smart structures, highway traffic monitoring, weighing of vehicles in motion, and structural vibration control. The addition of short carbon fibers to cement decreases the electrical resistivity, due to the high conductivity of the carbon fibers compared to cement. The decrease occurs even when the fibers are at a volume fraction below the percolation threshold [8,9], because the cement matrix is slightly conducting. Short fibers rather than continuous fibers are preferred for concretes because of the desire for low cost and feasibility of incorporation of the fibers in a concrete mix. A low volume fraction of fibers is preferred because of the importance of low cost, good workability, and high compressive strength (low air void content).

The strain-sensing ability of carbon fiber-reinforced cement [1–7] is associated with piezoresistivity, i.e., the change of the electrical resistivity with strain. The origin of the piezoresistivity relates to the effect of strain on the fiber–matrix contact resistivity. The effect is reversible. The fractional change in resistance per unit strain (i.e., the gage factor) is as high as 700.

In addition to providing the strain-sensing ability, carbon fiber addition to cement increases the tensile and flexural strengths, tensile ductility and flexural toughness, and decreases the drying shrinkage [10,11].

To help the dispersion of the short fibers in a concrete mix, silica fume (a particulate of particle size around 0.1 μm) is usually added to the mix [12]. The fine particulate nature of silica fume also causes the liquid permeability of the concrete to decrease, thereby improving the corrosion resistance of embedded steel reinforcing bars. Hence, in spite of the increased conductivity of the concrete due to the carbon fibers, the corrosion resistance is better than plain concrete [13].

Electric polarization refers to the phenomenon in which the centers of positive and negative charges do not coincide. It commonly occurs in a dielectric material when it is exposed to an electric field. As an electric field is present during electrical resistivity measurement, polarization can occur in a material during electrical resistivity measurement. As a consequence of the polarization-induced electric field in the material being opposite in direction from the applied electric field, polarization causes the measured resistivity to

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increase with time during resistivity measurement. This phenomenon complicates resistivity measurement and is undesirable for practical implementation of resistivity measurement in the field.

The more conductive a material is, the less is the tendency to polarize, since a conductor cannot support a significant electric field. Therefore, polarization is diminished when carbon fibers are added to cement. Nevertheless, polarization still occurs in carbon fiber-reinforced cement and needs attention in view of the application of this material in strain sensing. This article addresses this phenomenon and its dependence on the grade and volume fraction of the carbon fibers, and on the admixture (silica fume and latex). The fiber grade, fiber volume fraction, and admixture all affect the electrical conduction behavior of the composite, thereby affecting the tendency to polarize.

Resistivity measurement can be made by using the four- or two-probe method. The former involves using four electrical contacts — the outer two for passing current, the inner two for voltage measurement, so that the contact resistance is not included in the measured resistance. The latter involves using two electrical contacts — each for both current and voltage, so that the contact resistance is included in the measured resistance. Since polarization can occur within the sample, as well as at the contact–sample interface, the observed polarization effects are expected to differ between the four- and two-probe methods. Therefore, this article provides a comparison of the effects of the four- and two-probe methods.

## 2. Experimental methods

### 2.1. Materials

Three grades of carbon fibers were used. They were (i) amorphous pristine fibers, (ii) crystalline pristine fibers, and (iii) crystalline intercalated fibers.

The amorphous fibers were isotropic pitch-based, unsized, and of length ~5 mm and density 1.6 g/cm$^3$, as obtained from Ashland Petroleum (Ashland, KY). The fiber resistivity is $3.0 \times 10^{-3}$ $\Omega$ cm. The crystalline (actually partly crystalline) carbon fibers (Thornel P-100) were mesophase pitch-based, unsized, and of length ~5 mm and density 2.16 g/cm$^3$, as obtained from Amoco Performance Products, (Ridgefield, CT). The fiber resistivity is $2.2 \times 10^{-4}$ $\Omega$ cm. Intercalation of the crystalline carbon fibers was conducted by exposure of the fibers to bromine vapor in air at room temperature for 2 weeks to attain a Stage 2 (saturated, $C_{16}Br_2$, with 83% weight uptake) intercalation compound. After this, the fibers were removed from the bromine vapor and allowed to undergo bromine desorption in air at room temperature for 2–3 months in order to attain a stable compound, with about 20% weight uptake (relative to the pristine material) and a density of 2.5 g/cm$^3$ [14–16].

Comparison of grades (i) and (ii) gives the effect of fiber crystallinity, which governs the fiber resistivity. Comparison of grades (ii) and (iii) gives the effect of intercalation, which cannot be attained in amorphous fibers and which enhances the fiber resistivity through charge transfer between the intercalate and carbon. Bromine is an acceptor, thereby increasing the hole concentration in the carbon.

No aggregate (fine or coarse) was used. The cement used was Portland cement (Type I) from Lafarge (Southfield, MI). The fibers used were in the amount of either 0.5% or 1.0% by mass of cement.

Either silica fume or latex was used as an admixture. Silica fume (EMS 965, Elkem Materials, Pittsburgh, PA) was used in the amount of 15% by mass of cement. The methylcellulose, used along with silica fume in the amount of 0.4% by mass of cement, was; Methocel A15-LV, Dow Chemical, Midland, MI. The defoamer (1010, Colloids, Marietta, GA) used along with methylcellulose was in the amount of 0.13 vol.%.

The latex, used in the amount of 20% by mass of cement, was a styrene butadiene copolymer (Dow Chemical, 460NA) with the polymer making up about 48% for the dispersion and with the styrene and butadiene having a mass ratio of 66:34. The latex was used along with an antifoaming agent (No. 2410, 0.5% by mass of latex, Dow Corning, Midland, MI).

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. Latex (if applicable) was mixed with the antifoam by hand for about 1 min. Then the methylcellulose mixture (if applicable), the latex mixture (if applicable), cement, water, silica fume (if applicable), and fibers were mixed in the mixer for 5 min. After pouring into oiled molds, an external electrical vibrator was used to facilitate compaction and decrease the amount of air bubbles. The samples were demolded after 1 day and cured in air at room temperature (relative humidity = 100%) for 28 days.

Twelve types of carbon fiber cement paste were prepared, as listed in Table 1. The water/cement ratio was 0.35 for pastes with silica fume and 0.23 for pastes with latex.

### 2.2. Testing

Electrical resistivity measurements were conducted using either the four- or two-probe method, as noted, with silver paint in conjunction with copper wires for electrical contacts. The two-probe method gave essentially the same result as the four-probe method, due to the high sample resistance. A Keithley (Cleveland, OH) 2001 multimeter was used. Samples were in the form of rectangular bars of size 150 × 12 × 11 mm$^3$. Each electrical contact was applied around the entire 12 × 11 mm$^2$ perimeter of the bar. The voltage contacts were at two parallel cross-sectional planes that were 40 mm apart.

Table 1
Resistivity and absolute thermoelectric power of carbon fiber cement pastes

| Fiber grade | Fiber content | | Admixture | Resistivity (Ω cm)[a] | Absolute thermoelectric power[b] (μV/°C) |
|---|---|---|---|---|---|
| | % by mass of cement | vol.% | | | |
| Amorphous pristine | 0.5 | 0.48 | SF | $(1.5 \pm 0.1) \times 10^4$ | $0.89 \pm 0.09$ |
| Amorphous pristine | 1.0 | 0.95 | SF | $(8.3 \pm 0.5) \times 10^2$ | $-0.48 \pm 0.11$ |
| Amorphous pristine | 0.5 | 0.41 | L | $(9.7 \pm 0.6) \times 10^4$ | $1.14 \pm 0.05$ |
| Amorphous pristine | 1.0 | 0.82 | L | $(1.8 \pm 0.2) \times 10^3$ | $0.24 \pm 0.08$ |
| Crystalline, pristine | 0.5 | 0.36 | SF | $(1.3 \pm 0.1) \times 10^4$ | $0.47 \pm 0.11$ |
| Crystalline, pristine | 1.0 | 0.70 | SF | $(7.7 \pm 0.8) \times 10^2$ | $-0.79 \pm 0.16$ |
| Crystalline, pristine | 0.5 | 0.30 | L | $(8.8 \pm 1.0) \times 10^4$ | $0.78 \pm 0.08$ |
| Crystalline, pristine | 1.0 | 0.61 | L | $(1.6 \pm 0.2) \times 10^3$ | $-0.14 \pm 0.12$ |
| Crystalline, intercalated | 0.5 | 0.31 | SF | $(6.7 \pm 0.5) \times 10^3$ | $-11.5 \pm 1.13$ |
| Crystalline, intercalated | 1.0 | 0.61 | SF | $(7.1 \pm 0.6) \times 10^2$ | $-16.6 \pm 1.32$ |
| Crystalline, intercalated | 0.5 | 0.26 | L | $(5.3 \pm 0.4) \times 10^4$ | $-7.42 \pm 1.09$ |
| Crystalline, intercalated | 1.0 | 0.52 | L | $(1.6 \pm 0.1) \times 10^3$ | $-10.2 \pm 1.07$ |

SF: silica fume, L: latex.

[a] Measured within 1 s from the start of resistivity measurement in order to avoid polarization effect.

[b] $1.96 \pm 0.05$ μV/°C for plain cement paste (without fiber or admixture), $1.98 \pm 0.03$ μV/°C for silica fume cement paste without fiber, and $2.04 \pm 0.02$ μV/°C for latex cement paste without fiber [18].

The electrical resistivity was measured using the two-probe method as a function of time from the start of the resistivity measurement in order to observe the effect of polarization. For the purpose of confirming the occurrence of polarization, the polarity of the resistivity measurement was switched after a period of time of resistivity measurement. The resistivity measurement was conducted continuously before and after the switching, which was conducted by controlling the voltage. Both the two- and four-probe methods were used for the switching experiment.

The thermoelectric power gives information on the conduction behavior, so it was measured in this work. Thermopower measurement was performed on rectangular samples of size $75 \times 15 \times 15$ mm$^3$, such that heat (up to 65°C) was applied at one of the $15 \times 15$ mm$^2$ ends of a sample by contacting this end with a resistance-heated platen of size much larger than $15 \times 15$ mm$^2$. The other end of the sample was near room temperature. The thermal contact between the platen and the sample end was enhanced by using a copper foil covering the $15 \times 15$ mm$^2$ end surface. Silver paint was applied between the foil and the sample surface covered by the foil to further enhance the thermal contact. Underneath the copper foil was a copper wire that had been wrapped around the perimeter of the sample for the purpose of voltage measurement. Silver paint was present between the copper wire and the sample surface under the wire. The other end of the rectangular sample was similarly wrapped with copper wire and then covered with copper foil. The copper wires from the two ends were fed to a Keithley 2001 multimeter for voltage measurement. A T-type thermocouple was attached to the copper foil at each of the two ends of the sample for measuring the temperatures of the two ends. Voltage and temperature measurements were done simultaneously using the multimeter. The voltage difference divided by the temperature difference yielded the Seebeck coefficient with copper as the reference, since the copper wires at the two ends of a sample were at different temperatures. This Seebeck coefficient plus the absolute thermoelectric power of copper (+2.34 μV/°C) [17] is the absolute thermoelectric power of the sample. Six samples of each of the three types of cement paste were tested. Each sample was heated at one end at a rate of 0.009°C/s and then cooled with the power of the platen turned off. The heating rate was constant, but the cooling rate was not.

## 3. Results and discussion

Table 1 shows the resistivity (two-probe method) and absolute thermoelectric power (during cooling, due to possible moisture loss from the cement matrix during heating) for various cement pastes. The resistivity is decreased slightly by using crystalline fibers rather than amorphous fibers and is further decreased by intercalating the crystalline fibers, in spite of the lower fiber volume fractions for the crystalline fiber cement pastes. For the same fiber grade and the same fiber content (in % by mass of cement), the use of latex as an admixture gave a higher resistivity than the use of silica fume. This is partly due to the lower fiber volume fraction for the corresponding paste with latex, and is partly due to the lower effectiveness of latex compared to silica fume for dispersing the fibers [12].

The absolute thermoelectric power is positive, indicating electron conduction, for the pastes without fibers (footnote b of Table 1). It is decreased by the addition of amorphous pristine fibers, indicating contribution to hole conduction by the fibers, as previously reported [18–20]. The use of crystalline pristine fibers instead of amorphous pristine fibers further decreases the absolute thermoelectric power, due to the increased extent of hole conduction, as reflected by the decrease in resistivity. The use of crystalline intercalated fibers instead of crystalline pristine fibers further

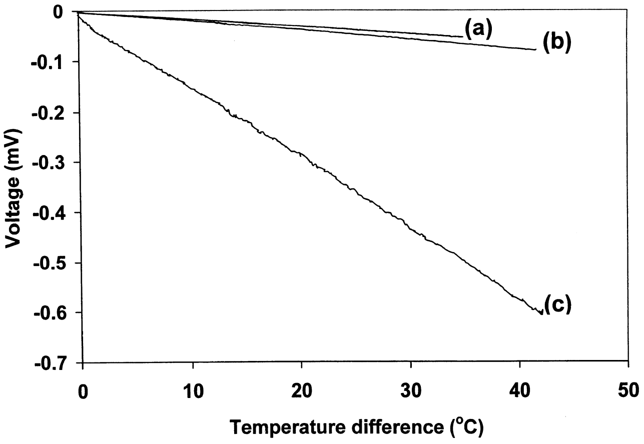

Fig. 1. Variation of the Seebeck voltage (with copper as the reference) vs. the temperature difference during heating and cooling for silica fume cement pastes with (a) amorphous pristine carbon fibers, (b) crystalline pristine fibers, and (c) crystalline intercalated carbon fibers.

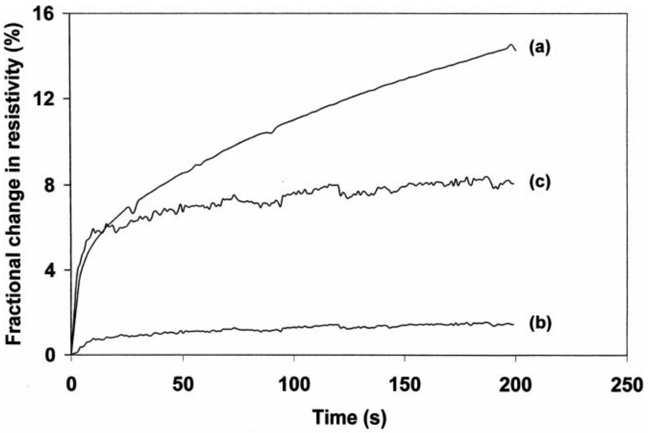

Fig. 3. Variation of the measured resistivity with time for cement pastes with fibers (0.5% by weight of cement) and latex. (a) Amorphous pristine carbon fibers, (b) crystalline pristine carbon fibers, and (c) crystalline intercalated carbon fibers.

decreases the absolute thermoelectric power so much that it becomes negative, indicating predominant hole conduction in the composite. Fig. 1 shows the curves of Seebeck voltage (with copper as the reference) vs. temperature difference for silica fume cement pastes with the three grades of carbon fibers. The slope is the Seebeck coefficient with copper as the reference. The curves during heating and cooling essentially overlap, although the curves shown are for cooling. Comparison of corresponding pastes with the two admixtures showed that the absolute thermoelectric power was more positive (or less negative) for the paste with latex than that with silica fume. This is consistent with the high resistivity for the latex case.

Figs. 2–5 show the variation of the measured resistivity (two-probe method) with time for cement pastes with fibers (0.5% by mass of cement) and silica fume, cement pastes with fibers (0.5% by mass of cement) and latex, cement pastes with fibers (1.0% by mass of cement) and silica fume, and cement pastes with fibers (1.0% by mass of cement) and latex, respectively. In all cases, the measured resistivity increased with time, such that the fractional increase was highest for the cement paste with amorphous pristine fibers, less for the cement paste with crystalline intercalated fibers, and lowest for the cement paste with crystalline pristine fibers. This means that the extent of polarization increased in the order: crystalline pristine fiber cement paste, crystalline intercalated fiber cement paste, and amorphous pristine fiber cement paste.

Comparison of Figs. 2 and 3 and of Figs. 4 and 5 shows that latex resulted in more polarization than silica fume. Comparison of Figs. 2 and 4 and of Figs. 3 and 5 shows that a higher fiber content resulted in much less polarization.

The observation that the cement paste with amorphous pristine fibers exhibited the most polarization (Figs. 2–5) is

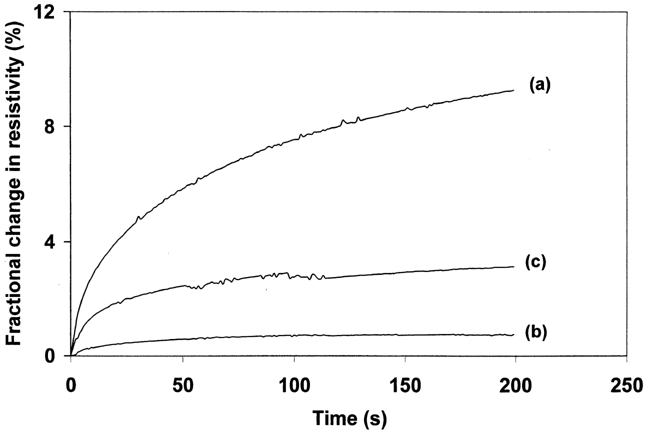

Fig. 2. Variation of the measured resistivity with time for cement pastes with fibers (0.5% by weight of cement) and silica fume. (a) Amorphous pristine carbon fibers, (b) crystalline pristine carbon fibers, and (c) crystalline intercalated carbon fibers.

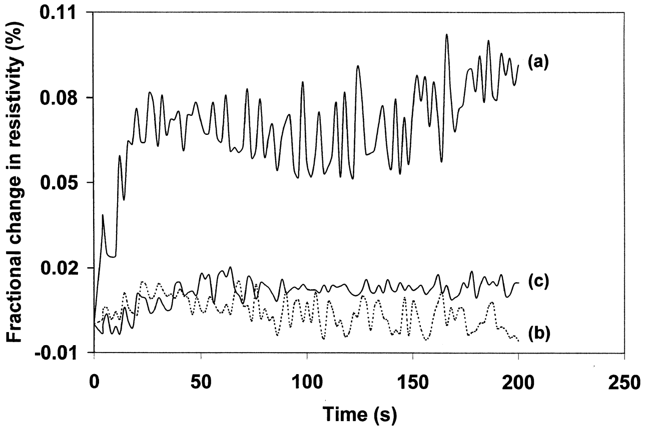

Fig. 4. Variation of the measured resistivity with time for cement pastes with fibers (1.0% by weight of cement) and silica fume. (a) Amorphous pristine carbon fibers, (b) crystalline pristine carbon fibers, and (c) crystalline intercalated carbon fibers.

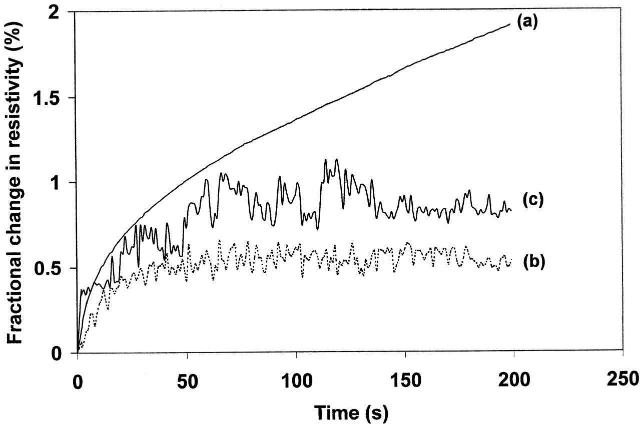

Fig. 5. Variation of the measured resistivity with time for cement pastes with fibers (1.0% by weight of cement) and latex. (a) Amorphous pristine carbon fibers, (b) crystalline pristine carbon fibers, and (c) crystalline intercalated carbon fibers.

consistent with its highest resistivity and most positive (least negative) value of the absolute thermoelectric power (Table 1). Although the cement paste with crystalline intercalated fibers exhibited lower resistivity than the corresponding paste with crystalline pristine fibers (Table 1), it exhibited more polarization (Figs. 2–5). This is attributed to the poor fiber–matrix interface, which probably resulted from the interaction between water in the matrix and bromine in the intercalated fiber. The poor interface is supported by the high activation energy of carrier hopping across the interface, as shown by data on the temperature dependence of the electrical resistivity [21].

Fig. 6 shows the effect of polarity switching on the measured resistivity (four-probe method) for the silica fume cement pastes with amorphous pristine fibers, crystalline pristine fibers, and crystalline intercalated fibers at 0.5% by mass of cement. The measured resistivity abruptly dropped at the time of the switching to a value below the initial value. After the switching, the measured resistivity gradually increased, but failed to reach the value immediately before the switching. The abrupt drop in measured resistivity during switching confirmed the occurrence of polarization.

The observation that the resistivity did not return to the value before the switching indicates some degree of irreversibility in the polarization. The degree of irreversibility increased in the order: amorphous pristine fiber cement paste, crystalline intercalated fiber cement paste, and crystalline pristine fiber cement paste; just like the trend for decrease in the extent of polarization. A large extent of polarization tended to overshadow the irreversibility phenomenon.

Fig. 7 shows results similar to Fig. 6, except that latex was used in place of silica fume. The trend for the increase in the degree of irreversibility in the polarization is the same as in Fig. 6.

Fig. 8 shows results similar to Fig. 6 for the silica fume case, except that the two-probe method was used. The two-probe method gave a slightly larger fractional change in resistivity than the four-probe method, indicating slightly more polarization. The resistivity did not drop as much at the time of switching and the resistivity rise after the

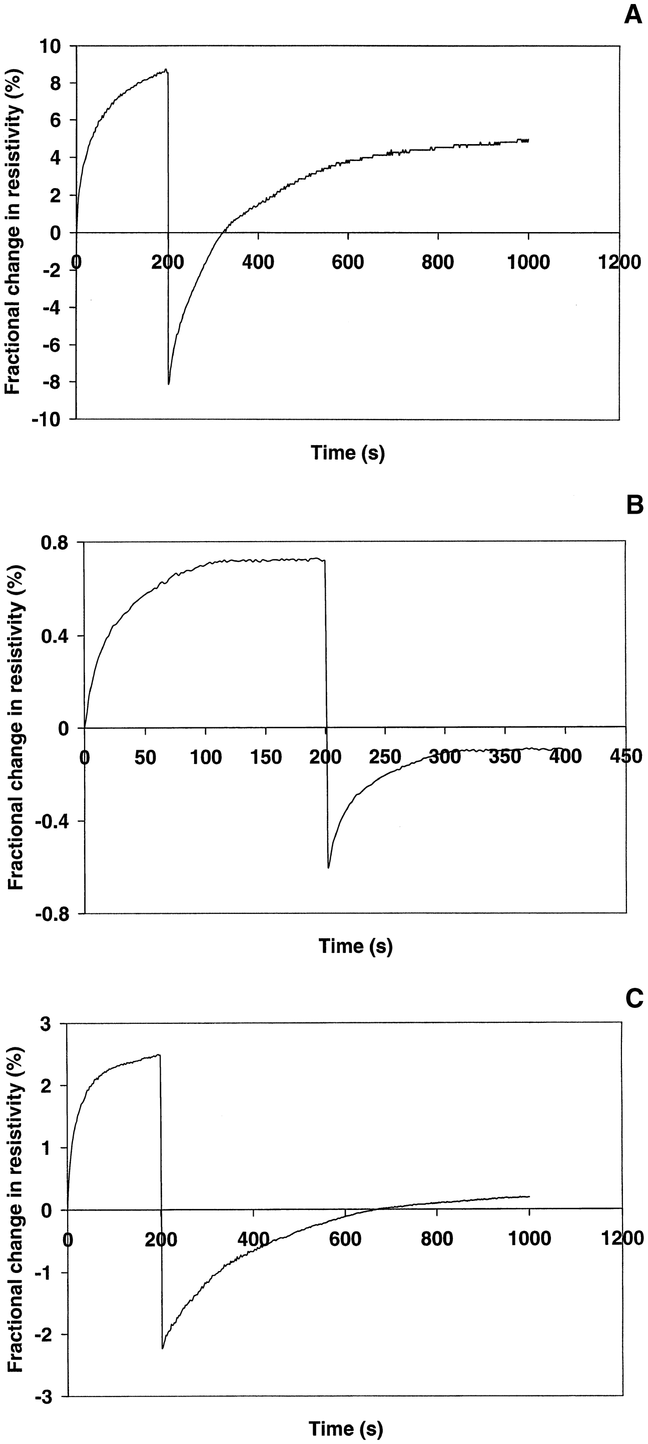

Fig. 6. Variation of the measured resistivity (four-probe method) with time before and after voltage polarity switching for cement pastes with fibers (0.5% by weight of cement) and silica fume. (A) Amorphous pristine carbon fibers, (B) crystalline pristine carbon fibers, and (C) crystalline intercalated carbon fibers.

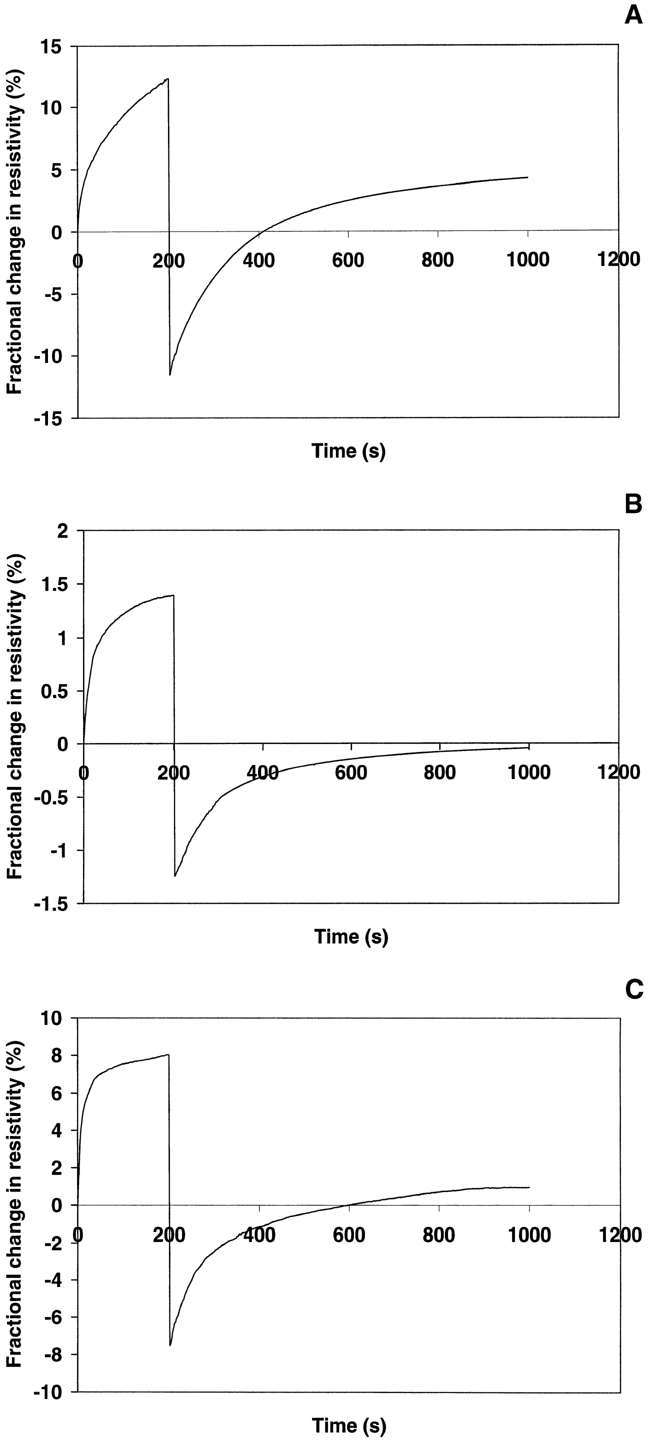

Fig. 7. Variation of the measured resistivity (four-probe method) with time before and after voltage polarity switching for cement pastes with fibers (0.5% by weight of cement) and latex. (A) Amorphous pristine carbon fibers, (B) crystalline pristine carbon fibers, and (C) crystalline intercalated carbon fibers.

switching was faster and more complete (reaching the value just before the switching). As the two-probe method included the contact resistance in the measured resistance, it included the polarization at the contact–sample interface in addition to the polarization within the sample in the overall polarization. The polarization reversal associated with the contact–sample interface was faster than that associated with the sample itself, thus causing the shorter rise time for the case of the two-probe method, as shown by

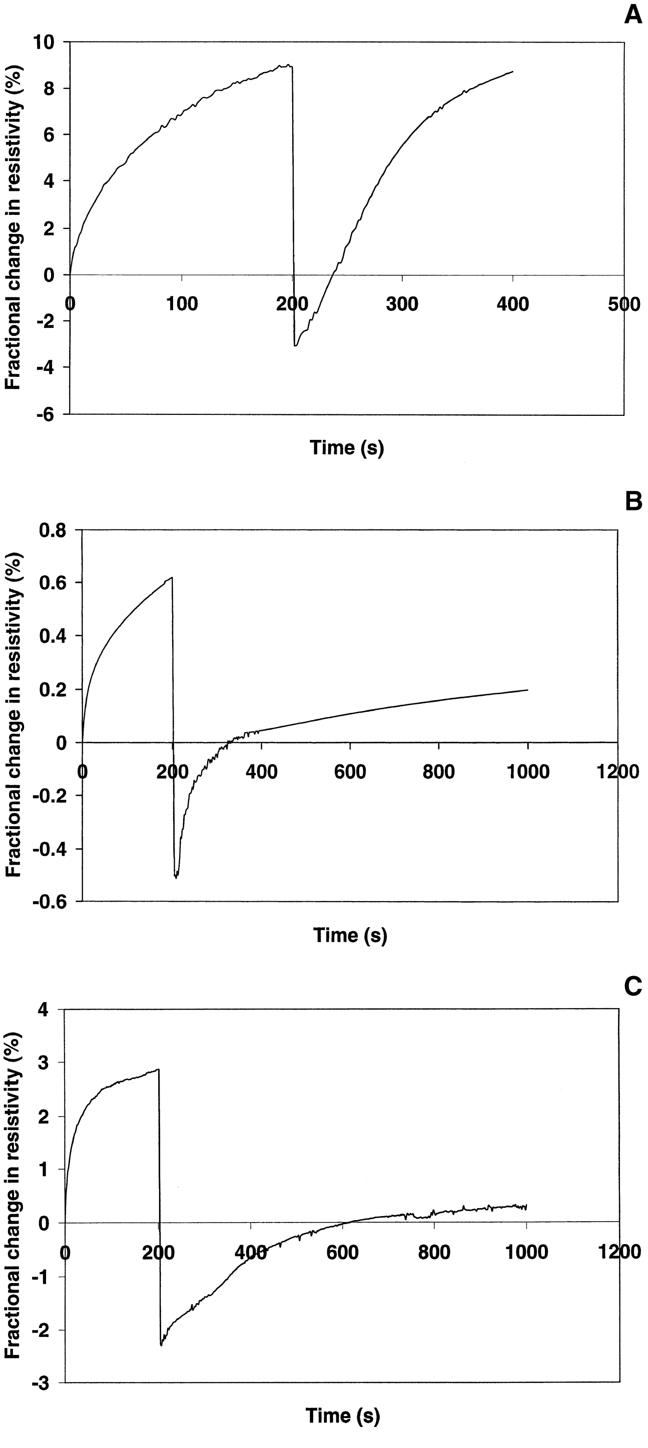

Fig. 8. Variation of the measured resistivity (two-probe method) with time before and after voltage polarity switching for cement pastes with fibers (0.5% by weight of cement) and silica fume. (A) Amorphous pristine carbon fibers, (B) Crystalline pristine carbon fibers, and (C) crystalline intercalated carbon fibers.

comparing Fig. 6(A) with Fig. 8(A), and comparing Fig. 6(B) with Fig. 8(B). The observation that the resistivity drop at the time of switching was less for the two-probe method was also due to the faster polarization reversal for the contact–sample interface. The polarization reversal associated with the contact–sample interface was also more complete than that associated with the sample itself. Due to the fast response of the contact–sample interface, it dominated the observed effect of polarity switching in Fig. 8. In contrast, the polarization of the sample itself dominated the observed effect of polarity switching in Fig. 6.

In measuring the electrical resistivity during stress application, as performed in the study of the strain-sensing ability of carbon fiber-reinforced cement, the polarization-induced resistivity increase over time is negligible [1]. This is because of the effect of stress, as described in a separate article.

## 4. Conclusion

Electric polarization during resistivity measurement caused the measured resistivity of carbon fiber cement pastes to increase with time. The extent of polarization was diminished by increasing the conductivity through the use of carbon fibers that were more crystalline, the increase of the fiber content, or the use of silica fume in place of latex as an admixture. Increasing the fiber crystallinity increased the conductivity of the composite and decreased the extent of polarization. Intercalation of the crystalline fibers further increased the conductivity of the composite, but it increased the extent of polarization.

Polarization reversal upon polarity switching was faster and more complete when the two-probe method was used in place of the four-probe method, due to the contribution of polarization at the contact–sample interface.

The polarization within the sample dominated the polarity switching effects obtained using the four-probe method. The less the polarization was, the less reversible was the polarization upon polarity switching.

## Acknowledgment

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