

Single-fiber electromechanical pull-out testing and its application to studying the interface between steel fiber and cement

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Abstract—The experimental technique of single-fiber electromechanical pull-out testing was introduced and used to study the interface between steel fiber and cement. The technique involves measuring both the contact electrical resistivity between fiber and matrix and the shear bond strength in a fiber–matrix interface sample. Samples that are identically prepared differ in contact resistivity and bond strength, which correlate. The correlation allows determination of even small differences in bond strength due to differences in sample preparation conditions, such as fiber surface treatment, cement curing age and admixtures to the cement paste. It also gives information on the structure of the interface and allows the bond strength to be non-destructively determined by measuring the contact resistivity.

Keywords: Fiber; pull-out; electrical resistivity; bond strength; composite; contact resistivity.

1. INTRODUCTION

The bond between fibers and matrix is critical to the quality of a fibrous composite material. The shear bond strength is commonly determined by single-fiber pull-out testing, which involves embedding one end of a single fiber in the matrix and pulling the fiber out of the matrix during the test [1–7]. A problem of this method is the large amount of scatter in the shear bond strength data obtained on different samples that are identically prepared, whether the embedment length is fixed or not. Due to the scatter, the standard deviation is large and the shear bond strength determination is limited in accuracy [2, 7]. As a result, small but real differences in bond strength between samples that are not identically prepared (say, with different surface treatments of the fiber) are hard to measure. The origin of the data scatter had long been assumed to be experimental error associated with pull-out testing, though fracture mechanics analysis suggested that the data scatter is inherent in the specimens themselves [7]. By measuring the contact electrical resistivity between fiber and matrix on every sample that was subjected to pull-out testing, we experimentally confirmed

that the data scatter is not due to experimental error in pull-out testing, but rather due to real differences in the structure of the fiber–matrix interface among samples that are identically prepared. A strong correlation between the bond strength and the contact resistivity was observed. The curve describing this correlation changed (say, shifted) when either the fiber or the matrix was modified, so that even small differences in bond strength due to fiber or matrix modification were determined. This new technique is hereby called ‘single-fiber electromechanical pull-out testing’. This technique is valuable for measuring even small differences in bond strength and provides information on the origin of the bonding and on the structure of the interface. Moreover, contact resistivity measurement (without fiber pull-out), is a nondestructive method for measuring the bond strength, provided that the curve correlating the two quantities is given. The experimental method and its demonstration are provided in this paper.

2. METHOD OF SINGLE-FIBER ELECTROMECHANICAL PULL-OUT TESTING

The method of single-fiber electromechanical pull-out testing involves embedding one end of a single fiber in a matrix, as in conventional single-fiber pull-out testing. In addition, four electrical contacts are applied—two on the fiber and two on the matrix surrounding the fiber, as illustrated in Fig. 1. The outer two contacts (the upper one on the fiber; the lower one on the matrix and not touching the fiber) serve as the current contacts; the inner two contacts (the upper one on the fiber; the lower one on the matrix and not touching the fiber) serve as the voltage contacts. An electric current (DC) is passed through the two current contacts, while the two voltage contacts are connected to a voltmeter. The use of four contacts is in accordance with the four-probe method of electrical measurement. The voltage measured between the voltage contacts is the sum of the voltage drop along the fiber due to the volume resistivity of the fiber, the voltage drop across of the fiber–matrix interface due to the contact resistivity of the interface, and the voltage drop across the matrix in the direction radially outward in the plane perpendicular to the fiber due to the volume resistivity of the matrix. By using a fiber that is much more conducting than the matrix (which is not totally insulating) and a matrix which is small in dimension in the plane perpendicular to the

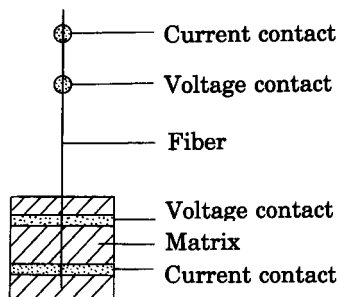


Figure 1. Sample configuration for measuring the contact electrical resistivity of the interface between a fiber and matrix.

fiber, the measured voltage drop is essentially all due to the contact resistivity of the interface. In case the matrix dimension is not small, the contribution of the matrix to the measured voltage drop can be calculated from the volume resistivity of the matrix (separately measured) and the dimensions of the matrix. The voltage drop divided by the current gives the resistance R .

The contact resistance R_c is related to the contact resistivity ρ_c by

$$R_c = \frac{\rho_c}{A_c}, \quad (1)$$

where A_c is the contact area. The volume resistance R_v is related to the volume resistivity ρ_v by

$$R_v = \rho_v \frac{l}{A_v}, \quad (2)$$

where l is the length of the conductor in the current direction and A_v is the cross-sectional area of the conductor. The total measured resistance R_T between the voltage probes is

$$R_T = R_v^{\text{fiber}} + R_c + R_v^{\text{matrix}}. \quad (3)$$

By choosing a highly conducting fiber (such as carbon or steel), R_v^{fiber} is negligible, so that equation (3) becomes

$$R_T = R_c + R_v^{\text{matrix}}. \quad (4)$$

By further choosing a matrix that is small in dimension in the plane perpendicular to the fiber and choosing a matrix that is not too high in volume resistivity, R_v^{matrix} is negligible, so that equation (4) becomes

$$R_T = R_c. \quad (5)$$

Once R_c is determined, ρ_c can be obtained from equation (1). The quantity ρ_c does not depend on geometry and depends only on the structure of the interface.

The measurement of ρ_c does not require any pull-out of the fiber, as it is non-destructive. However, measurement of ρ_c during fiber pull-out (optional) can give further information on the interface. As in conventional single-fiber pull-out testing, the shear bond strength is determined using the sample configuration of Fig. 1. Thus, the configuration of Fig. 1 allows measurement of bond strength and contact resistivity on the same sample. This combined measurement constitutes the heart of single-fiber electromechanical pull-out testing.

3. DEMONSTRATION OF SINGLE-FIBER ELECTROMECHANICAL PULL-OUT TESTING

The technique of single-fiber electromechanical pull-out testing was demonstrated by using cement paste (slightly conducting) as the matrix and stainless steel as the fiber. The effects of matrix modification, cement curing age and fiber modification on the bond strength and contact resistivity were studied.

3.1. Effect of fiber modification

Cement paste made from Portland cement (Type I) from Lafarge Corp. (Southfield, MI) was used for the cementitious material. The water/cement ratio was 0.35. The water reducing agent used in the amount of 0.5% by weight of cement was TAMOL SN (Rohm and Haas Co., Philadelphia, PA), which contained 93–96% sodium salt of a condensed naphthalenesulfonic acid. The volume electrical resistivity of the cement paste was 1.40×10^5 , 1.53×10^5 , 1.58×10^5 and 1.62×10^5 Ω cm at 1, 7, 14 and 28 days of curing respectively, as measured by the four-probe method using silver paint for electrical contacts.

Stainless steel (Fe–Cr–Al) fibers described in Table 1 were used for the steel material. The as-received fibers were subjected to two different surface treatments, namely, acetone (reagent grade) washing and acid (37.71% hydrochloric acid, reagent grade) washing. The washing was conducted by stirring the fibers in a beaker containing either acetone or acid for 5–10 min. For the case of acetone washing, washing was followed by air drying at room temperature for 10–15 min. For the case of acid washing, washing was followed by rinsing in water and then air oven drying at 200°C for 5–10 min.

Immediately after preparing the cement paste by mixing using a Hobart mixer with a flat beater, a small amount of the cement paste was used to embed one end of a single fiber and to affix it to a rectangular piece of paper with a rectangular hole cut in its middle, as illustrated in Fig. 2. The other end of the fiber was affixed to the paper using an adhesive, such that the end in the adhesive was above the center hole and the end in the cement paste was below the center hole. The cement paste was then allowed to cure in air. The curing time was 28 days unless noted otherwise. At the end of the curing period, the paper with the fiber affixed to it was mounted vertically on a tensile tester and then cut with scissors along the horizontal dashed line shown in Fig. 2 so as to separate the paper into two halves.

The contact electrical resistivity between the fiber and the cement paste was measured at 1, 7, 14 and 28 days of curing using the four-probe method and silver paint as electrical contacts, as illustrated in Fig. 1. One current contact and one voltage contact were on the fiber, while the other voltage and current contacts were on the cement paste embedding the fiber to a distance of 1 cm. The cement paste thickness was 1.5 mm on each side sandwiching the fiber. The fiber length was 5 cm.

Table 1.
Properties of steel fibers

Type of steel	Stainless 434
Manufacturer	International Steel Wool Corp. (Springfield, OH)
Length	5 mm
Diameter	60 μ m
Density	7.7 g cm ³
Modulus	200 GPa (2.9×10^7 psi)
Elongation at break	3.2%
Tensile strength	970 MPa (1.4×10^5 psi)
Volume electrical resistivity	6×10^{-5} Ω cm

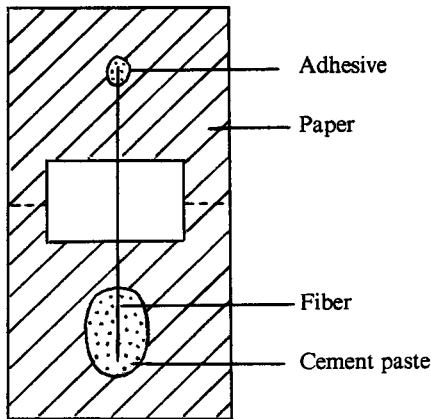


Figure 2. Sample configuration for single-fiber pull-out testing.

The resistance between the two voltage probes was measured using a Keithley 2001 multimeter; it corresponds to the sum of the fiber volume resistance, the interface contact resistance and the cement paste volume resistance. The measured resistance turned out to be dominated by the contact resistance, to the extent that the two volume resistance terms can be neglected. The contact resistivity (in $\Omega \text{ cm}^2$) is given by the product of the contact resistance (in Ω) and the contact (interface) area (in cm^2).

Single-fiber pull-out testing was conducted on the same interface samples and at the same time as the cout testing, one end of the fiber was embedded in cement paste, as in Figs 1 and 2. A Sintech 2/D screw-action mechanical testing system was used. The contact resistivity was taken as the value prior to pull-out testing. The bond strength was taken as the maximum shear stress during pull-out testing. Seven interface samples were tested for each combination of fiber surface treatment (as-received, acetone washed or acid washed) and curing time (1, 7, 14 or 28 days).

Scanning electron microscopy was performed on the as-received, acetone-washed and acid-washed fibers in order to investigate the effect of surface treatment on the surface morphology.

Weight measurements were conducted on the fibers both before and after acetone washing and before and after acid washing in order to investigate the effect of surface treatment on the weight. The washing process included the drying step. Six specimens were used for each type of washing.

Figures 3–5 give typical plots of shear stress *vs.* displacement and simultaneously obtained plots of contact electrical resistivity *vs.* displacement for as-received, acetone-washed and acid-washed fibers respectively at 28 days of curing. In all three cases, the contact resistivity abruptly increased when the shear stress reached its maximum, i.e. when fiber–matrix debonding was completed. For the as-received and acetone-washed fibers (Figs 3 and 4), the contact resistivity did not change before the abrupt increase when the shear stress had reached its maximum. For the acid-washed fibers (Fig. 5), the contact resistivity gradually increased prior to the abrupt increase when the shear stress had reached its maximum.

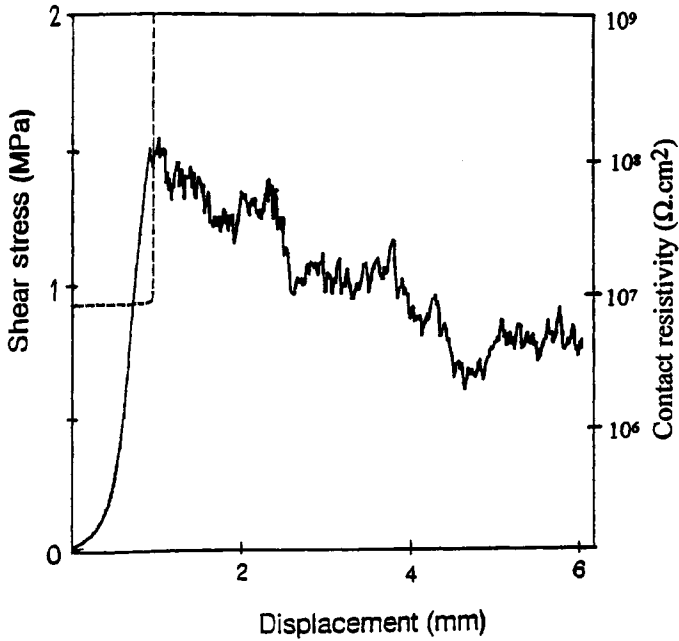


Figure 3. Plots of shear stress vs. displacement (solid curve) and of contact electrical resistivity vs. displacement (dashed curve) simultaneously obtained during pull-out testing of as-received stainless steel fiber from cement paste at 28 days of curing.

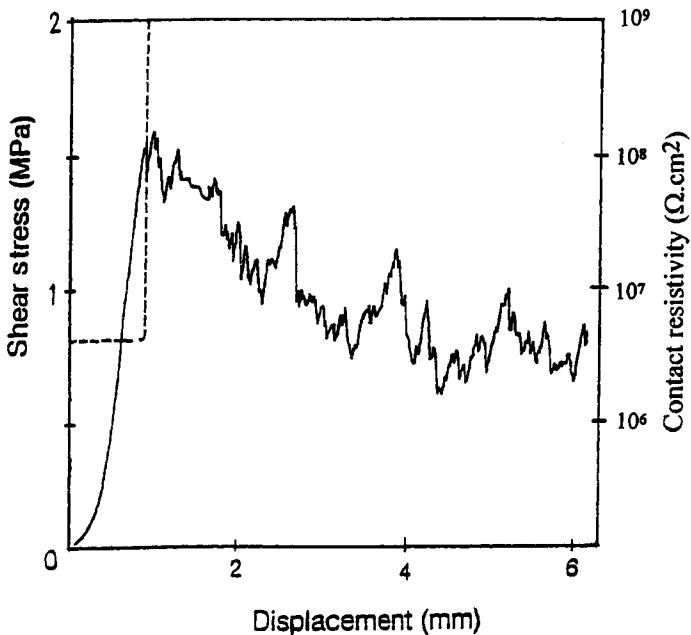


Figure 4. Plots of shear stress vs. displacement (solid curve) and of contact electrical resistivity vs. displacement (dashed curve) simultaneously obtained during pull-out testing of acetone-washed stainless steel fiber from cement paste at 28 days of curing.

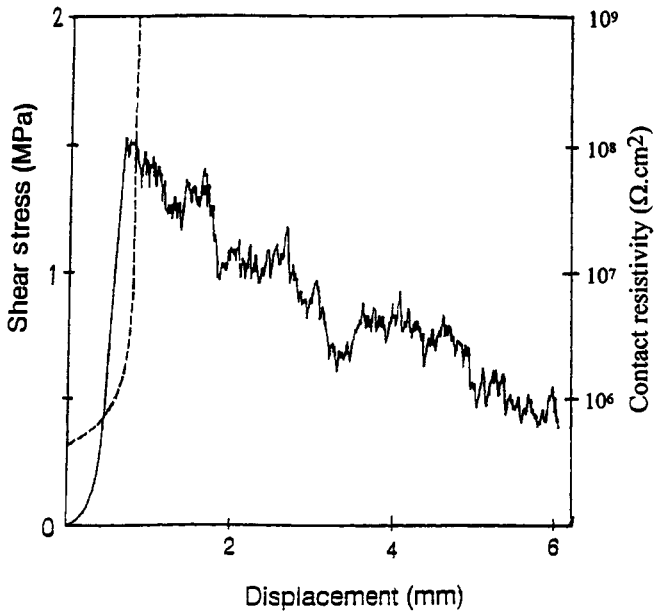


Figure 5. Plots of shear stress vs. displacement (solid curve) and of contact electrical resistivity vs. displacement (dashed curve) simultaneously obtained during pull-out testing of acid-washed stainless steel fiber from cement paste at 28 days of curing.

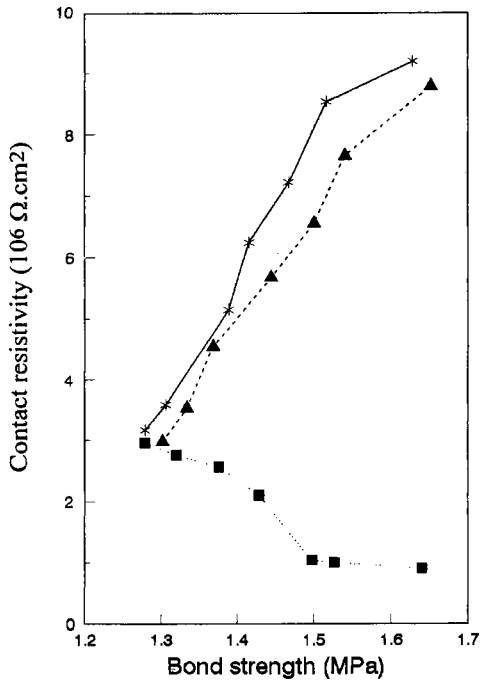


Figure 6. Variation of contact electrical resistivity with bond strength at 28 days of curing for as-received (stars), acetone-washed (triangles) and acid washed (squares) stainless steel fibers.

Figure 6 shows the correlation of the contact resistivity with the bond strength at 28 days for the as-received, acetone-washed and acid-washed fibers. For each type of surface treatment, the bond strength as well as contact resistivity varied among the seven samples (identically prepared) tested. Nevertheless, the contact resistivity correlated strongly with the bond strength among the data for each type of surface treatment. The increase was roughly linear, except for negative deviation from linearity in the high bond strength regime. For the as-received and acetone-washed fibers, the contact resistivity increased with increasing bond strength. The increase was roughly linear, except for negative deviation from linearity in the high bond strength regime. For the case of acid-washed fibers, the contact resistivity decreased with increasing bond strength. The range of bond strength was similar for the three types of surface treatment, but the range of contact resistivity was lower for the acid-washed case than the as-received and acetone-washed cases.

Figure 7 shows scanning electron microscope (SEM) photographs of the as-received, acetone-washed and acid-washed fibers. Acetone washing slightly roughened the surface, whereas acid washing significantly roughened the surface. The fractional weight loss due to acetone washing was $(2.9 \pm 0.5)\%$ and that due to acid washing was $(20.4 \pm 1.2)\%$.

Figure 6 shows that the contact resistivity increases with increasing bond strength for the as-received and acetone-washed fibers but decreases with increasing bond strength for the acid-washed fibers. This means that high resistivity phase(s) at the steel-cement interface (higher in resistivity than the cement paste) dominates the mechanism for enhancing the bonding for the as-received and acetone-washed fibers, whereas decrease in the amount of interfacial voids (which are high in resistivity) dominates the mechanism for enhancing the bonding for the acid-washed fibers. This interpretation is consistent with the fact that the range of contact resistivity exhibited by the acid-washed fibers is lower than that exhibited by the as-received or acetone-washed fibers (Fig. 6). The acid washing removed some phase(s) (probably metal oxides and other compounds) from the surface of the fibers, as suggested by the roughening of the surface (Fig. 7) and the 20% weight loss. The removal of the phase(s) by acid washing apparently makes it impossible for the high resistivity phase(s) that enhance the bonding to form when the fiber subsequently encounters the cement paste. Due to the presence of the high resistivity phase(s) that enhance bonding in the as-received and acetone-washed cases, the interfacial voids (which are also high in resistivity) cannot be distinguished electrically, leading to no change in contact resistivity during debonding (Figs 3 and 4). On the other hand, for the acid-washed case, the interfacial voids govern the bond strength, so the contact resistivity increases as the interfacial void content increases during debonding (Fig. 5). In all three cases, the contact resistivity shoots up by orders of magnitude at the completion of debonding and the start of fiber pull-out. Thus, the contact resistivity measurement provides information on the structure of the fiber-cement interface. In the case of the acid-washed fibers, contact resistivity measurement also provides a means of monitoring the progress of debonding (Fig. 5).

Acetone washing increases the bond strength and decreases the contact resistivity (Fig. 6). This is partly because of the removal of organic material by the acetone

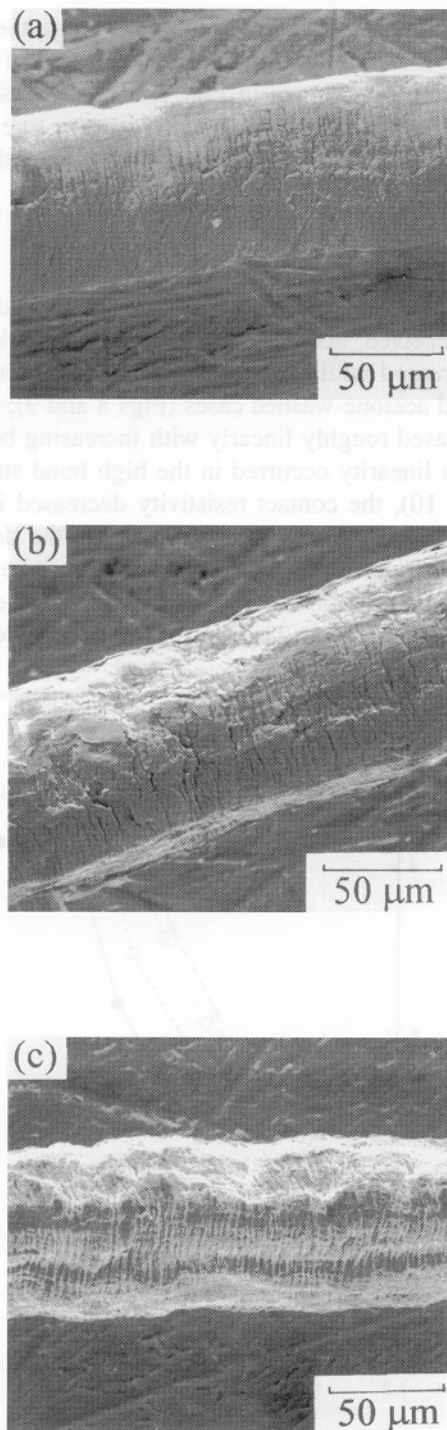


Figure 7. SEM photographs of (a) as-received, (b) acetone-washed, and (c) acid-washed stainless steel fibers.

washing (consistent with the 3% weight loss after washing), the electrically insulating character of the organic material and the detrimental effect of the organic interfacial layer on the bond strength. As shown in Fig. 7, acetone washing slightly roughens the fiber surface. The surface roughening increases the true interfacial area, so it also plays a role in increasing the apparent bond strength and decreasing the apparent contact resistivity.

3.2. Effect of cement curing age

Figures 8–10 show the dependence of the bond strength and contact resistivity on the curing age for as-received, acetone-washed and acid-washed fibers respectively. The bond strength decreased while the contact resistivity increased with curing age for both as-received and acetone-washed cases (Figs 8 and 9); at each curing age, the contact resistivity increased roughly linearly with increasing bond strength, such that negative deviation from linearity occurred in the high bond strength regime. For the acid-washed case (Fig. 10), the contact resistivity decreased in a non-linear fashion with increasing bond strength; due to the non-linearity, the dependence of the bond strength and contact resistivity on the curing age could not be determined.

Figures 8 and 9 show that the bond strength decreases while the contact resistivity increases with curing age from 1 to 28 days for the as-received and acetone-washed

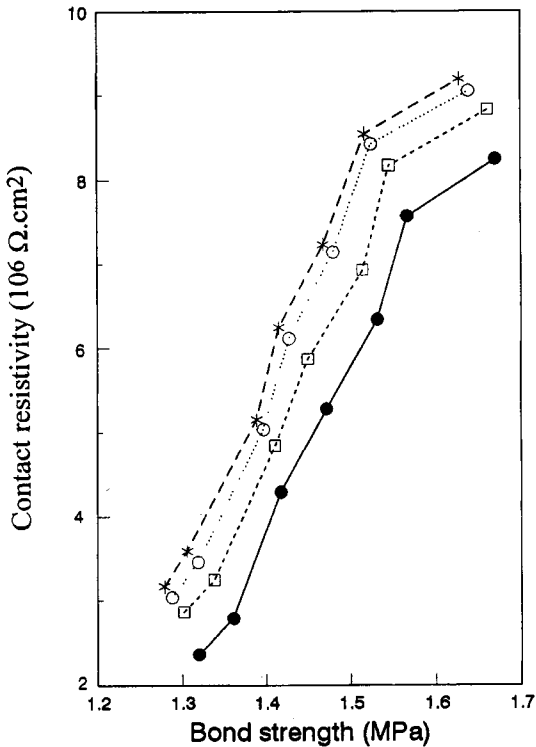


Figure 8. Variation of contact electrical resistivity with bond strength for as-received stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing.

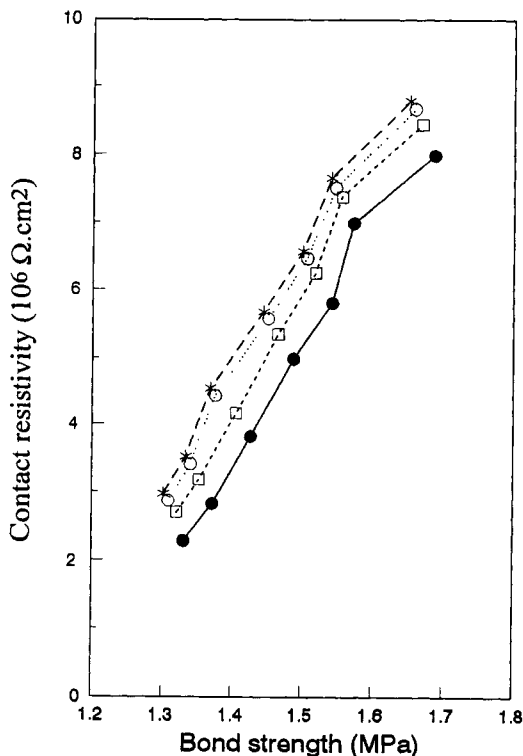


Figure 9. Variation of contact electrical resistivity with bond strength for acetone-washed stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing.

cases. These effects suggest that, as curing occurs, the interfacial void content increases. Thus, as steel reinforced concrete cures, the cementitious material strengthens while the bond strength between steel and the cementitious material weakens. This finding is useful for consideration of early strength development in steel reinforced concrete.

The negative deviation from linearity in the high bond strength regime (Figs 6, 8 and 9) is attributed to the need to have a low interfacial void content in order to attain a high bond strength and the decrease of the contact resistivity when the interfacial void content is decreased. In other words, both the high resistivity interfacial phase that helps bonding and a low interfacial void content are needed in order to attain a high bond strength.

3.3. Effect of matrix modification

Stainless steel (Fe–Cr–Al) fibers described in Table 1 were used. The as-received fibers were washed in acetone (reagent grade) by stirring the fibers in a beaker containing acetone for 5–10 min. Washing was followed by air drying at room temperature for 10–15 min.

Cement paste made from Portland cement (Type I) from Lafarge Corp. (Southfield, MI) was used for the cementitious material. Three types of cement pastes were used,

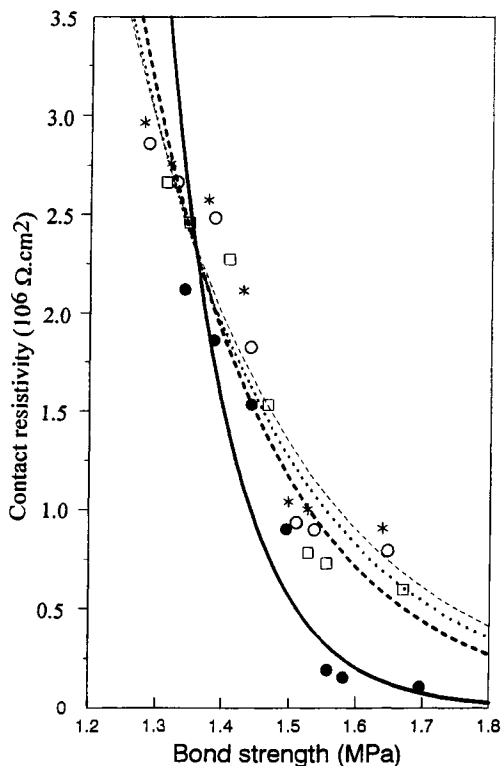


Figure 10. Variation of contact electrical resistivity with bond strength for acid-washed stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing.

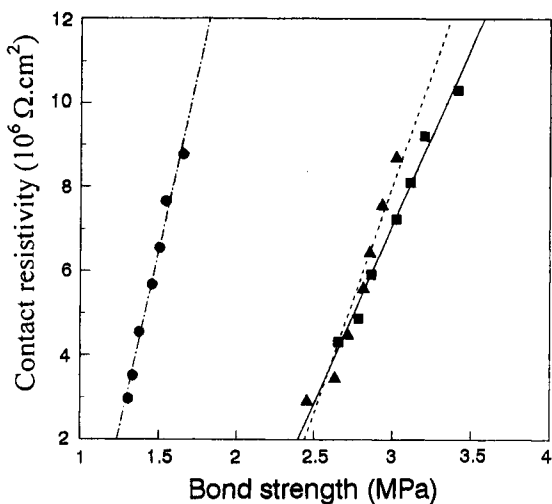


Figure 11. Correlation of the contact electrical resistivity with the bond strength for the interface between stainless steel fiber and cement paste at 28 days of curing; ● – plain cement paste; ▲ – cement paste with methylcellulose; ■ – cement paste with latex.

namely (i) plain cement paste (with only cement and water, such that the water/cement ratio was 0.45), (ii) cement paste with methylcellulose 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), and (iii) 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 in cement paste (ii) was TAMOL SN (Rohm and Haas Co., Philadelphia, PA), which contained 93–96% sodium salt of a condensed naphthalenesulfonic acid. Methylcellulose (Dow Chemical, Midland, MI, Methocel A15-LV) in the amount of 0.4% of the cement weight was used in cement paste (ii). The defoamer (Colloids Inc., Marietta, GA, 1010) used along with it was in the amount of 0.13 vol.%; it was used whenever methylcellulose was used. The latex (Dow Chemical, Midland, MI, 460NA) used in cement paste (iii) was a styrene-butadiene polymer emulsion; it was used in the amount of 0.20 of the weight of the cement. The antifoam (Dow Corning, Midland, MI, 2410, an emulsion) used was in the amount of 0.5% of the weight of the latex; it was used whenever latex was used.

For the case of cement paste containing latex, the latex and antifoam first were mixed by hand for about 1 min. Then cement, water and the water reducing agent were mixed in the Hobart 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 cement, water and water reducing agent were mixed in the Hobart mixer for 5 min. Specimens in the form of Fig. 2 were allowed to cure in air for 28 days.

Plots of shear stress *vs.* displacement similar to Fig. 4 were obtained. The contact resistivity abruptly increased when the shear stress reached its maximum, i.e. when fiber-matrix debonding was completed. It did not change before the abrupt increase when the shear stress had reached its maximum.

Figure 11 shows the correlation of the contact resistivity with the bond strength. The contact resistivity increased linearly with the bond strength among the data for each type of cement paste. The bond strength was lower for the plain cement paste than the cement paste with methylcellulose and that with latex. On the average, the paste with latex gave slightly higher bond strength than that with methylcellulose, but the difference was small. On the average, the contact resistivity was higher for the cement paste with latex than the other two pastes, which were similar in contact resistivity.

Whether the polymer is latex (20% by weight of cement) or methylcellulose (0.4% by weight of cement), polymer admixtures to the cementitious matrix help the bond between fiber and matrix. In spite of the large difference in concentration between latex and methylcellulose, the effect on the bond strength is similar. On the other hand, methylcellulose addition does not alter the contact electrical resistivity between fiber and matrix, whereas latex addition increases this resistivity. This suggests that the contact resistivity is less sensitive to a small amount of polymer addition than is the bond strength. For the purpose of cost saving and a low contact resistivity, methylcellulose is preferred to latex as an admixture to the cementitious matrix.

4. CONCLUSION

Single-fiber electromechanical testing is a new technique for fiber–matrix bond strength measurement and interface study. The technique is limited to fibers (e.g. steel, carbon, etc.) that are much more conducting than the matrix and to matrices (e.g. cement) that are not totally insulating. By measuring both bond strength and contact resistivity for each interface sample, a curve of contact resistivity *vs.* bond strength is obtained for each set of identically prepared samples. By determining the effect of sample preparation conditions on this curve, even slight differences in bond strength between samples that are prepared differently can be determined. Moreover, the shape of this curve gives information on the origin of bonding and the structure of the interface. By using such curves as calibration curves, the measurement of the contact resistivity provides a nondestructive method for determining the bond strength.

Through single-fiber electromechanical pull-out testing, the following new information was obtained.

1. The scatter in shear bond strength data is not due to the experimental error associated with pull-out testing, but is due to real differences in interface structure (e.g. interfacial phase content and interfacial void content) among interface samples that are identically prepared.
2. For as-received and acetone-washed stainless steel fibers, the contact resistivity increased linearly with increasing bond strength, because interfacial phase(s) of volume resistivity higher than that of cement paste enhanced the bonding.
3. For acid-washed stainless steel fibers, the contact resistivity decreased with increasing bond strength, because the interfacial voids caused the resistivity to increase and the bond strength to decrease; the contact resistivity was lower than for as-received or acetone-washed stainless steel fibers.
4. The bond strength decreased and the contact resistivity increased with increasing curing age for as-received and acetone-washed stainless steel fibers, due to interfacial void content increase.
5. Acetone washing increased the bond strength, decreased the contact resistivity, and caused the contact resistivity to correlate linearly with the bond strength more strongly, due to its cleansing action.
6. The contact resistivity increased gradually during fiber–matrix debonding for the case of acid-washed stainless steel fibers, because interfacial voids increased in abundance during debonding. The contact resistivity did not change during fiber–matrix debonding, except for an abrupt increase at the completion of debonding, for the case of as-received and acetone-washed stainless steel fibers, because interfacial voids and the high resistivity interfacial phase(s) that enhanced bonding could not be distinguished electrically. Thus, for the case of acid-washed stainless steel fibers, the progress of fiber–matrix debonding can be monitored by contact resistivity measurement.
7. The acetone washing caused a 3% weight loss and slightly increased the roughness of the stainless steel fibers; the acid washing caused a 20% weight loss and greatly increased the roughness of the stainless steel fibers. The surface roughening can

at least partly explain the slight decrease in apparent contact resistivity and slight increase in apparent bond strength after acetone washing. However, surface roughening cannot explain the large decrease in contact resistivity and the reversal of the trend of the variation between contact resistivity and bond strength after acid washing.

8. The acid washing removed certain phase(s) from the surface of the stainless steel fiber. These phase(s) were needed for the formation of high resistivity fiber–matrix interfacial phase(s) that would have enhanced bonding. Nevertheless, the roughening effect of acid washing helped maintain the apparent bond strength to the level of the as-received fiber.
9. The addition of methylcellulose (0.4% by weight of cement) or latex (20% by weight of cement) to cement paste gave similar increases in the bond strength between stainless steel fiber and cement paste. The methylcellulose addition did not change the contact electrical resistivity between fiber and paste, whereas the latex addition increased this resistivity. For low cost and low contact resistivity, methylcellulose is preferred to latex.

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