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Bond Strength and Contact Electrical Resistivity between Cement and Stainless Steel Fiber: Their Correlation and Dependence on Fiber Surface Treatment and Curing Age


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The contact electrical resistivity of the steel-cement interface was found to correlate strongly with the shear bond strength, so that it provides a non-destructive method of bond strength assessment. 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. For acid washed stainless steel fibers, the contact resistivity was lower, increased with decreasing bond strength, and increased during debonding, all due to the effect of interfacial voids; thus contact resistivity provided an indication of the progress of debonding. 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. The bond strength decreased and the contact resistivity increased with increasing curing age from 1 to 28 days for as-received and acetone washed stainless steel fibers, due to interfacial void content increase.

Keywords: bond strength; cement; concrete; contact electrical resistivity; curing age; stainless steel fiber; surface treatment.

INTRODUCTION

The bond strength between steel and cement is critical to the performance of steel bar reinforced concrete and steel fiber reinforced concrete. Without good bonding, load transfer from the cementitious material to the steel is ineffective, thus degrading the reinforcing action of the steel. Bond strength is conventionally measured under shear by steel bar/fiber pull-out testing,[1] which is a destructive method. Non-destructive methods such as microscopy and interface chemical analysis can provide information on the structure of the steel-matrix interface, but they do not provide measurement of the bond strength and are tedious. A non-destructive method that provides an indirect measurement of the bond strength is provided in this paper. This method involves measurement of the contact electrical resistivity between steel and the cementitious material. Good correlation was found in this work between bond strength and contact electrical resistivity for a given curing age and a given surface treatment of the steel fiber. Through this correlation, information on the structure of the steel-cement interface was also obtained.

Due to the variation of the bond strength obtained by pull-out testing from one interface sample to another prepared in the same way, the dependence of the bond strength on curing age and steel surface treatment had not been previously investigated. Through correlation between bond strength and contact electrical resistivity, this problem was eliminated, so that the dependence of the bond strength on the curing age and steel surface treatment was determined in this work.

Curing age is an important variable that affects the mechanical properties of concrete, which increases in strength as the curing age increases. The effect of curing age on the steel-cement bonding, though also important, has not been previously investigated. In this work, it was found that the steel-cement bond strength decreases with curing age.

Surface treatment is commonly applied to reinforcements in composites for the purpose of enhancing the bonding between the reinforcement and the matrix. Surface treatment is to be distinguished from coating application. For example, epoxy coating on steel serves to enhance the corrosion resistance of the steel reinforcement. Surface treatments can involve solvent cleansing, acid treatment, plasma treatment, laser treatment, or other methods. For reinforcements in concrete, only low-cost surface treatments are practical. Of the various surface treatments, solvent cleansing is the least expensive from equipment and safety points of view. In this work, we found that solvent cleansing of steel using acetone is effective for increasing the steel-cement bond strength.

This work is an extension of the authors' earlier work on steel rebars[2] and carbon fibers.[3] All measurements in this work were made on steel fibers, in particular stainless steel fibers.

RESEARCH SIGNIFICANCE

This paper provides a new non-destructive method of steel-cement bond strength assessment and steel-cement debonding monitoring. It also provides a steel surface treat-

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ment for increasing the steel-cement bond strength and improving the steel-cement bond strength reproducibility. Moreover, this paper provides the finding that the steel-cement bond strength decreases with curing age.

EXPERIMENTAL METHODS

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 percent hydrychloric 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.

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 percent by weight of cement was TAMOL SN (Rohm and Haas Co., Philadelphia, PA), which contained 93-96 percent 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.

The contact electrical resistivity between the fiber and the cement paste was measured at 1, 7, 14, and 28 days of

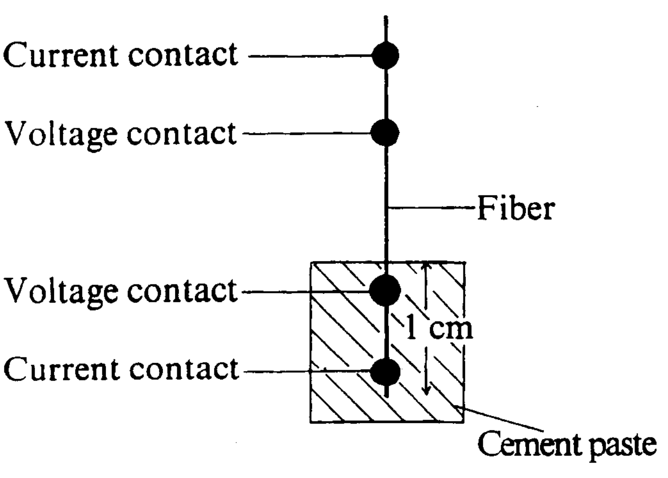

Fig. 1—Sample configuration for measuring the contact electrical resistivity of the interface between a fiber and cement paste

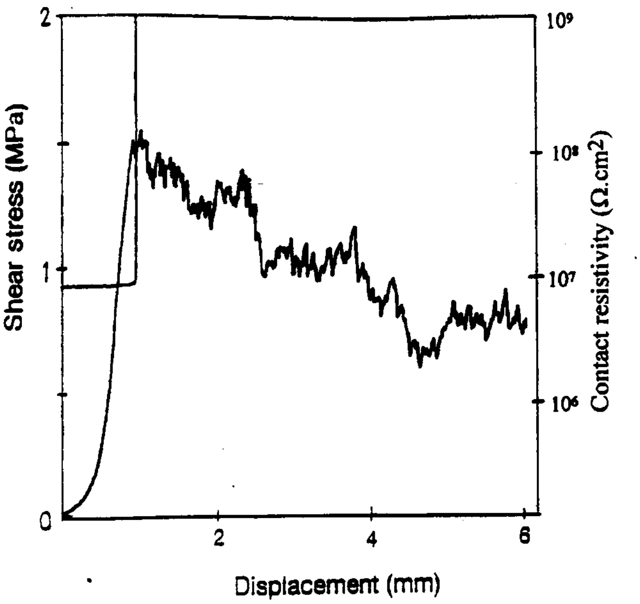

Fig. 2—Plots of shear stress vs. displacement and of contact electrical resistivity vs. displacement simultaneously obtained during pull-out testing of as-received stainless steel fiber from cement paste at 28 days of curing

curing using the four-probe method and silver paint as electrical contacts, as illustrated in. 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. The resistance between the two voltage probes was measured; 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 $\Omega.cm^2$) 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 contact resistivity was measured. For pull-out testing, one end of the fiber was embedded in cement paste, as in Fig. 1. 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

Table 1—Properties of steel fibers

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

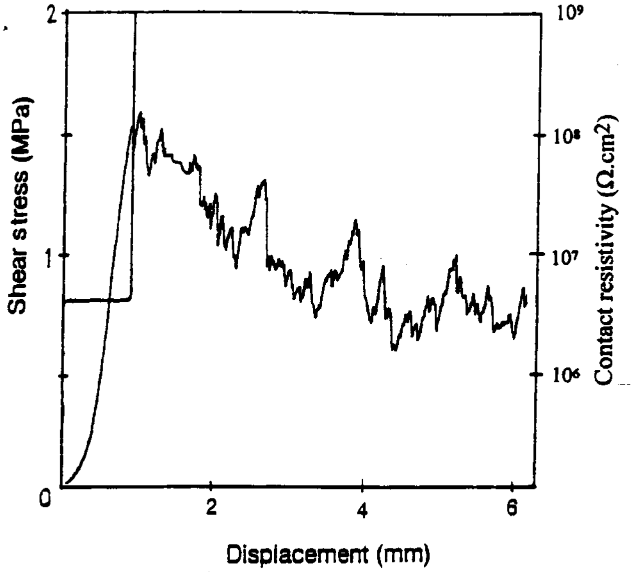

Fig. 3—Plots of shear stress vs. displacement and of contact electrical resistivity vs. displacement simultaneously obtained during pull-out testing of acetone washed stainless steel fiber from cement paste at 28 days of curing

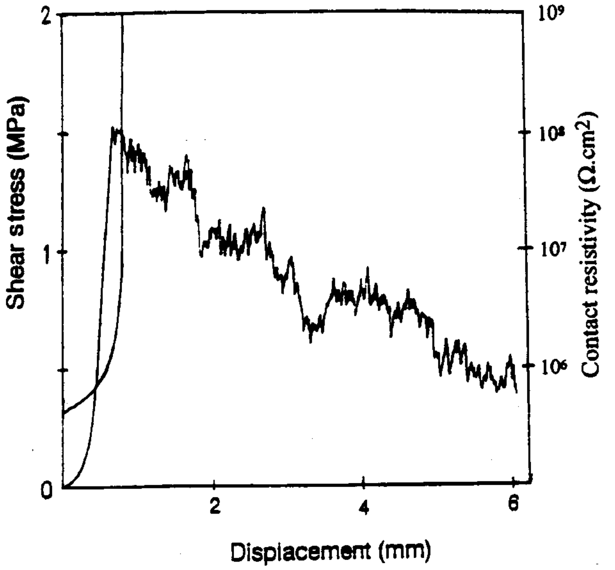

Fig. 4—Plots of shear stress vs. displacement and of contact electrical resistivity vs. displacement simultaneously obtained during pull-out testing of acid washed stainless steel fiber from cement paste at 28 days of curing

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.

RESULTS

Fig. 2-4 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 (Fig. 2 and 3), the contact resistivity did not change before the abrupt increase when the shear stress had reached its maximum. For the acid washed fibers (Fig. 4), the contact resistivity gradually increased prior to the abrupt increase when the shear stress had reached its maximum.

Fig. 5 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. For the as-received and acetone washed fibers, the contact resistivity increased with increasing bond strength. 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.

Fig. 6-8 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 (Fig. 6 and 7); 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. 8), 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.

Fig. 9 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 ± 0.5) percent and that due to acid washing was (20.4 ± 1.2) percent.

DISCUSSION

Fig. 5 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

(Fig. 5). 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. 9) and the 20 percent 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 from 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 (Fig. 2 and 3). 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. 4). 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. 4).

Acetone washing increases the bond strength and decreases the contact resistivity (Fig. 5). This is partly because of the removal of organic material by the acetone washing (consistent with the 3 percent weight loss after washing), the electrically insulating character of the organic material, and the ... of the organic interfacial layer on the bond strength. As shown in Fig. 9, 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. As shown by comparing Fig. 6 and 7, acetone washing strengthens the linear correlation between the contact resistivity and bond strength. This effect is consistent with the cleansing action of acetone. That acetone washing increases the bond strength is technologically significant, even though the fractional increase in bond strength is small, since acetone washing is a relatively inexpensive method of surface treatment.

Fig. 6 and 7 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 cases. The effects are significant from 1 to 14 days and much less significant from 14 to 28 days. These effects suggest that, as curing occurs, the interfacial void content increases, probably due to drying shrinkage of the cement paste. 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 (Fig. 6 and 7) is attributed to the need to have a low interfacial void content in order to attain a high bond

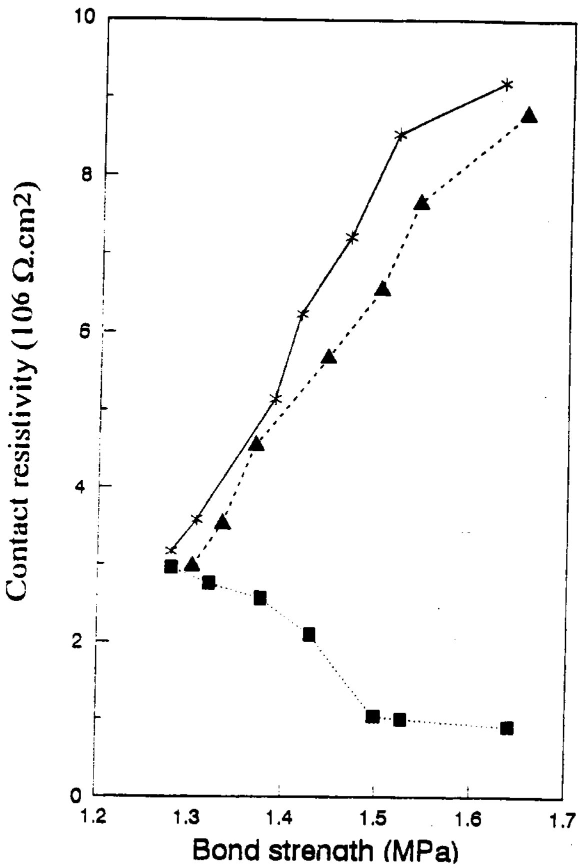

Fig. 5—Variation of contact electrical resistivity with bond strength at 28 days of curing for as-received (stars), acetone washed (triangles), and acid washed (squares) stainless steel fibers

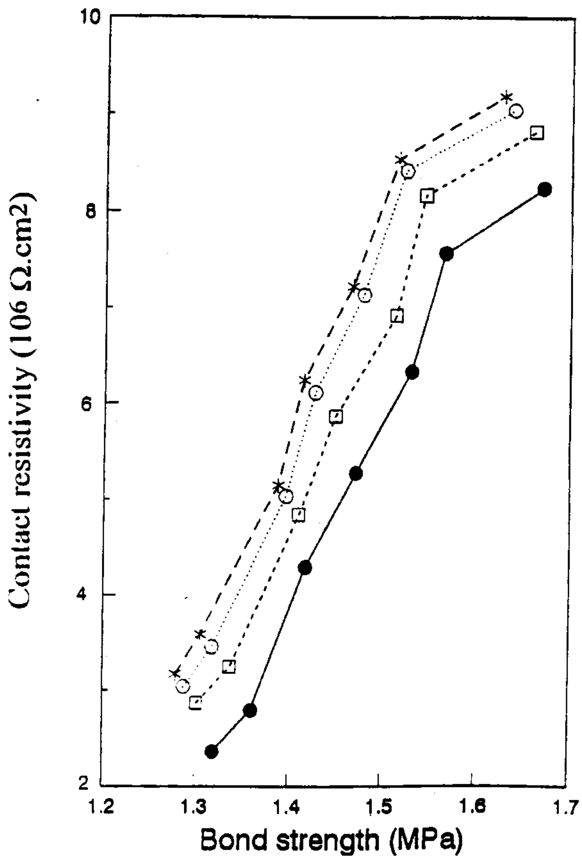

Fig. 6—Variation of contact electrical resistivity with bond strength for as-received stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing

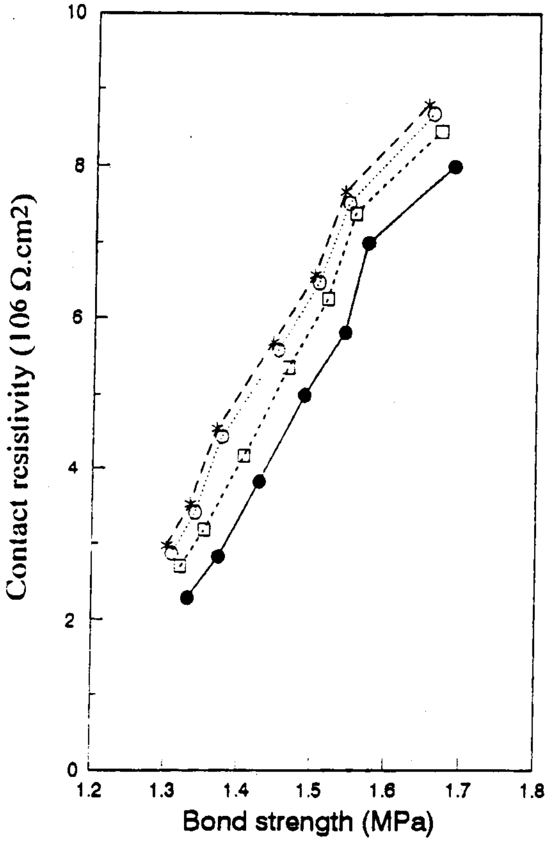

Fig. 7—Variation of contact electrical resistivity with bond strength for acetone washed stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing

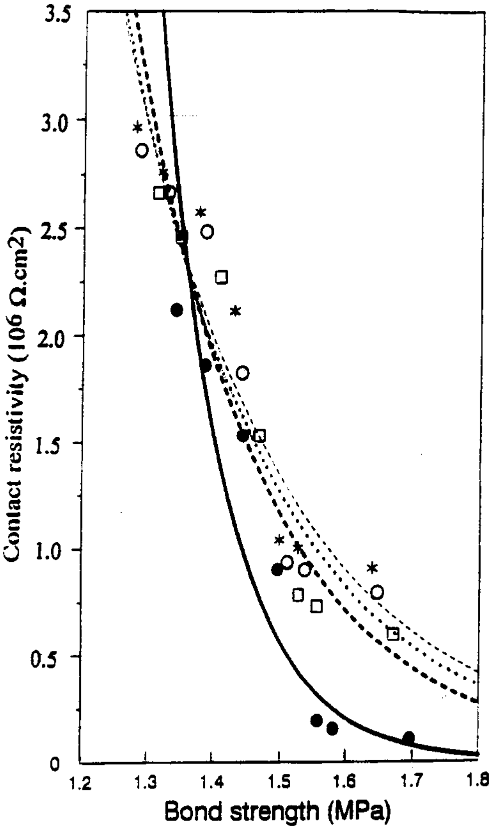

Fig. 8—Variation of contact electrical resistivity with bond strength for acid washed stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing

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.

The strong correlation between bond strength and contact resistivity for a particular surface treatment and a particular curing age means that contact resistivity measurement can be used to provide an indirect measure of the bond strength. For the as-received and acetone washed cases, this correlation is roughly linear, so that Fig. 6 and 7 can be used as calibration curves for conversion of contact resistivity to bond strength.

Comparison of the results of Ref. 2 on steel rebar pull-out from concrete with those of this work on steel fiber pull-out from cement paste shows that both bond strength and contact resistivity are higher for the former than the latter. The difference in bond strength is attributed to the surface deformations on the steel rebar and the smoothness of the steel fiber surface; the surface deformations lead to mechanical interlocking between rebar and concrete, thereby increasing the bond strength. The difference in contact resistivity is attributed to the larger interfacial void content in the case of steel rebar pull-out from concrete; the larger void content is partly due to the surface deformations on the rebar and partly due to the presence of large aggregate in the concrete. Comparison of the results in Ref. 2 and those of this work also shows that the relationship between contact resistivity and bond strength is more linear in the former. In particular, the data in Ref. 2 do not show the negative deviation from linearity observed in this work. This difference is attributed to the important contribution of mechanical interlocking and the relatively little contribution of the interfacial void content in affecting the bond strength between rebar and concrete. In the case of steel fiber pull-out from cement paste, the essential absence of mechanical interlocking makes the interfacial void content important and the negative deviation from linearity is due to the void content contribution.

Comparison of the results in Ref. 3 on carbon fiber pull-out from cement paste with those of this work on steel fiber pull-out from cement paste shows that the carbon fibers behave like acid-washed steel fibers in that the contact resistivity increases during debonding and decreases with increasing bond strength. This behavior is in contrast to that of as-received or acetone-washed steel fibers, for which the contact resistivity is quite constant during debonding and increases with increasing bond strength. The similarity between carbon fibers and acid-washed steel fibers is due to the absence in both cases of the interfacial phase of high volume resistivity that helps the bonding. In contrast, this interfacial phase is present in the cases of as-received and acetone-washed steel fibers.

Practical findings of this work include the following:

1. Acetone washing of stainless steel fibers serves to increase the fiber-matrix bond strength and improve fiber-matrix bond strength reproducibility.

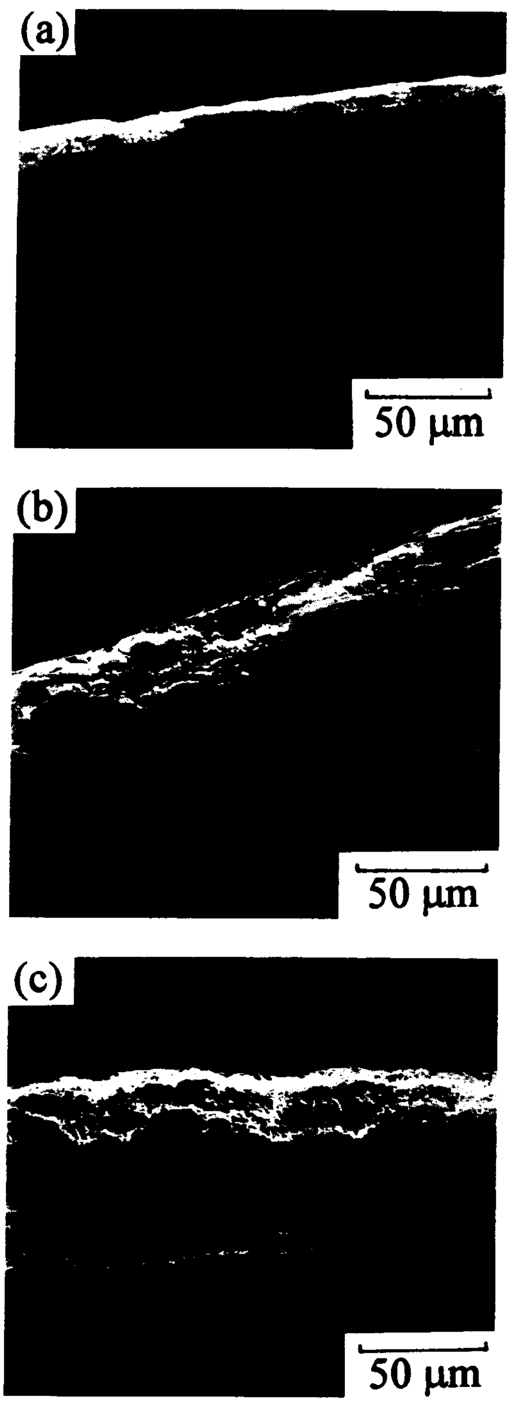

Fig. 9—SEM photographs of (a) as-received; (b) acetone washed; and (c) acid washed stainless steel fibers

2. The fiber-matrix bond strength decreases with curing age, at least for stainless steel fibers.

3. The contact resistivity between stainless steel fiber and the cement matrix correlates strongly with the fiber-matrix bond strength for a given surface treatment of the fiber and for a given curing age, thus allowing non-destructive bond strength measurement.

4. In the case of acid washed stainless steel fibers, the contact resistivity increases during fiber-matrix debonding, thus providing a new method of monitoring the progress of debonding.

CONCLUSIONS

1. The contact electrical resistivity of the steel-cement interface was found to correlate strongly with the shear bond strength, so that it provides a new non-destructive method for bond strength measurement and fiber-matrix interface study.

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 percent weight loss and slightly increased the roughness of the stainless steel fibers; the acid washing caused a 20 percent 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.

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