

Study of the Interface Strength in Steel Fiber-Reinforced Cement-based Composites



by Sun Wei, James A. Mandel, and Samir Said

The properties of fiber-reinforced cement-based composite materials are dependent on the characteristics of the fiber, the matrix, and the fiber-matrix interface. In general, the nature and behavior of the fiber and matrix are reasonably well understood, but those of the interface are known in considerably less detail.

Results of an experimental program demonstrate that the addition of an acrylic polymer to fiber-reinforced mortar and cement strengthens the matrix material and improves the structure of interface, thus increasing the interface bond strength.

Tensile tests, fiber pullout tests, microhardness studies, and electron microscopy studies were conducted on mortar matrix materials, with and without the addition of the acrylic polymer. The tensile strength of the matrix material, the interfacial bond strength between the matrix and steel fibers, and the energy required for fiber debonding and pullout were increased by a factor of almost four with the addition of 15 percent acrylic polymer by weight of cement.

To explain these increases, the microhardness and microstructure of an annular region of the composite surrounding a fiber (interface transition ring) were investigated. Addition of the acrylic polymer to a cement matrix resulted in increases in microhardness of the cement-matrix material of the same order of magnitude as increases obtained in tensile strength, bond strength, and energy required for fiber debonding and pullout. Observation with a scanning electron microscope indicated that cracking along the fiber-matrix interface (before loading) is substantially reduced by the addition of acrylic polymer. Possible explanations for this are a reduction in the film of water that surrounds the fiber and the filling of small cracks with the acrylic polymer material itself.

Keywords: acrylic resins; bonding; cements; metal fibers; microcracking; microhardness; mortars (material); pullout tests; strength.

The properties of fiber-reinforced cement-based composite materials are dependent on the characteristics of the fiber, the matrix, and the fiber-matrix interface. In general, the nature and behavior of the fiber and matrix are reasonably well understood, but those of the interface are known in considerably less detail. To increase the efficiency of the fiber reinforcement further, it is necessary to understand the mechanism of failure of the composite material. To accomplish this, the fiber-matrix interface must be thoroughly studied.

In fiber-reinforced cement-based composites, the principal beneficial effects of the fibers accrue after cracking of the matrix has occurred. For loads beyond

those causing the initial cracking, further cracking is resisted by the fibers near the crack front. Resistance to crack extension and propagation provided by the fibers will depend on the properties of the fiber (length, diameter, and modulus), the fiber orientation, and the fiber volume content. However, perhaps the most important parameter affecting the post-cracking behavior of the composite is the properties of the fiber-matrix interface.

Properties of the fiber-matrix interface influence both the tensile strength and toughness (capacity to absorb energy) of the composite. In fact, interface properties that result in a composite with a high toughness may not produce a composite with high tensile strength and vice versa. Since it is important to have composites with both high tensile strength and toughness, it is important to devise techniques to improve the properties of the composite at and near the fiber-matrix interface.

The behavior of the interfacial structure is revealed by studying an annular region of the composite surrounding a fiber (interface transition ring). The behavior of the interface transition ring is controlled to a great extent by the characteristics of the matrix, especially the value of water-cement ratio. Thus the properties of the matrix can directly affect the efficiency of the fiber reinforcement.

Results of an extensive experimental study of the properties of the interface transition ring are presented. Materials both with and without the addition of the acrylic polymer were studied. Tensile tests, fiber pullout tests, microhardness studies, and electron microscopy studies were conducted. Significant improvements in the tensile strength of the matrix material, the interfacial bond strength, and the microhardness of the ma-

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trix were obtained by the addition of the acrylic polymer. Possible mechanisms to explain this improvement will be discussed.

RESEARCH SIGNIFICANCE

Practical application of steel fiber-reinforced cementitious materials has been hampered by the absence of design specifications. Before adequate design specifications can be written, it is necessary to understand the physical behavior and the mechanisms of failure of fiber-reinforced cementitious materials. One method to accomplish this is the use of a micromechanical finite element model. Application of the model, along with experimental verification, can result in an understanding of the effect of the geometry of the composite material, and the mechanical properties of matrix material, fibers, and fiber-matrix interface on the internal stresses and failure mechanism of the composite.

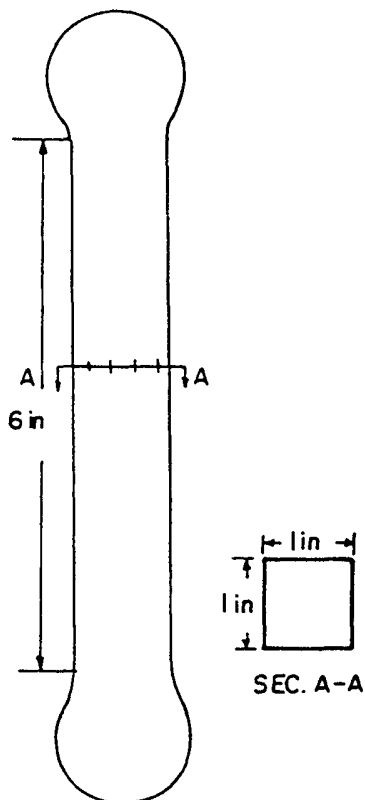


Fig. 1—Tensile test specimen

To develop such a model it is necessary to understand the nature and behavior of the fiber-matrix interface. Because of this, the studies reported in this paper were initiated. In addition, it has been shown that both the tensile strength and fracture toughness (resistance to crack growth) of fiber reinforced brittle material are significantly influenced by the properties of the fiber-matrix interface.

ACRYLIC POLYMER IN A MORTAR MATRIX

In fiber-reinforced cement-based composite materials, the strength and stiffness of the contact zone between the fiber and matrix often result from a combination of physical and chemical characteristics that develop during the formation of surface reaction products. The long-term properties of the composite are very much influenced by the nature of the interfacial bond, and often the contact zone (interface zone) is the weakest part of the system.

With steel fibers, the interfacial bond strength is a combination of adhesion, friction, and mechanical interlocking. Chemical interaction can also occur with time due to surface reactions. The mechanical properties for these kinds of composite materials are generally improved when the interfacial bond strength is increased. To fully utilize the fibers, premature failure at the fiber-matrix interface must not occur.

The effectiveness of the fiber can also be lost if failure occurs in the matrix material in the annular region surrounding the fiber (interface transition ring). Thus, to improve the effective bond strength of the fiber-matrix interface, the strength of both the matrix material and the fiber-matrix interface must be improved. Testing was conducted to measure both the bond and tensile strengths, and to relate the results of these studies to the internal structure of the material, the microhardness of the matrix material near the fiber-matrix interface was also measured. In addition, the surface features of the material was studied using a scanning electron microscope.

In the tests for tensile and bond strength, two matrix materials were used. The first matrix material was mortar, with a cement-to-sand ratio of 1 to 1.5 by weight and a water-cement ratio of 0.43 by weight. The second matrix material was also mortar with a cement-to-sand ratio of 1 to 1.5 by weight. However, a lower water-cement ratio (0.25 by weight) was achieved in the second matrix material by the addition of 15 percent by weight of cement of an acrylic polymer.* The resultant workability of the two matrix materials was 17 (determined using ASTM C 230-80).

Tensile strengths of the two matrix materials were measured using a specimen of the type shown in Fig. 1. Five specimens of each matrix material were tested.

The interfacial bond strength and the work done during fiber pullout were determined from fiber pullout tests with fiber groups (Fig. 2). A one-sixteenth in.

*Rhoplex E330, an acrylic polymer manufactured by the Rohm and Haas Company.

Table 1 — The effect of polymer on interfacial bond strength, tensile strength, and work done during fiber debonding and pullout

Mortar mix	Water-cement ratio	Cement-sand ratio	Acrylic polymer/cement (by weight), percent	Number of fibers	Fiber embedded length, mm	Fiber diameter, mm	Tensile strength of matrix, kg/cm ²	Average interface bond strength, kg/cm ²	Work done during fiber pullout from matrix (per fiber), kg-cm
1	0.25	1:1.5	15	16	20	0.5	87.6	85.1	20.5
2	0.43	1:1.5	0	16	20	0.5	23.6	22.7	5.43

The workability with and without acrylic polymer was the same; values shown are an average of the results from five specimens.

thick piece of Teflon was used to support the fibers while the mortar was being placed. The Teflon also prevented bonding between the two halves of the pullout test specimen. The fibers* used were 50 mm long by 0.5 mm in diameter. The hooks were cut off from one end of the fibers.

During the tests, the straight (unhooked) segments of the fibers (2 cm in length) were pulled out. In a previous unpublished study, pullout tests were conducted using fiber groups of 4, 9, 16, and 25 fibers. This resulted in fiber spacings of 8.3, 10, 16.6, and 25 fiber diameters, respectively, with an average bond strength per fiber for all four fiber groups being approximately the same. In this study the fiber spacing was kept constant and each pullout specimen had 16 fibers. Five specimens of each matrix type were tested. All tensile and pullout tests were performed at a specimen age of 28 days.

The specimens were tested in a closed-loop hydraulic testing machine using a constant displacement rate of one-half in. per minute. The results of the tensile and fiber pullout tests are summarized in Table 1. For the matrix with acrylic polymer, the tensile strength and the average interface bond strength (based on the peak load from the pullout tests) were 87.6 kg/cm² (1247 lb/in.²) and 85.1 kg/cm² (1210 lb/in.²) respectively. The corresponding values for the matrix without acrylic polymer were 23.6 kg/cm² (336 lb/in.²) and 22.7 kg/cm² (323 lb/in.²), respectively.

The addition of the acrylic polymer resulted in a 370 percent increase in both tensile and bond strengths. It is interesting to note that for both materials, there seemed to be a relationship between tensile strength and the average interfacial bond strength (i.e., they were approximately equal).

Typical load-displacement curves for the fiber pullout tests are in Fig. 3. The area under these curves is the energy required for fiber debonding and pullout. Area OAB (see insert in Fig. 3) is an upper limit to the energy due to debonding¹ and the remaining area is primarily energy due to friction during fiber pullout.

The addition of the acrylic polymer also increased the energy required for fiber debonding and pullout by a factor of 3.7. It is interesting to note that percentage

increases in tensile strength, interfacial bond strength, and energy required for fiber debonding and pullout resulting from acrylic polymer addition were essentially the same (370 percent).

One might try to attribute the increase in tensile strength resulting from acrylic polymer addition to the reduction in the water-cement ratio. However, this alone does not account for the same percentage increases in effective bond strength and fiber pullout energy, since most of the energy required for fiber debonding and pullout is due to friction during fiber pullout (Area OBC on Fig. 3).

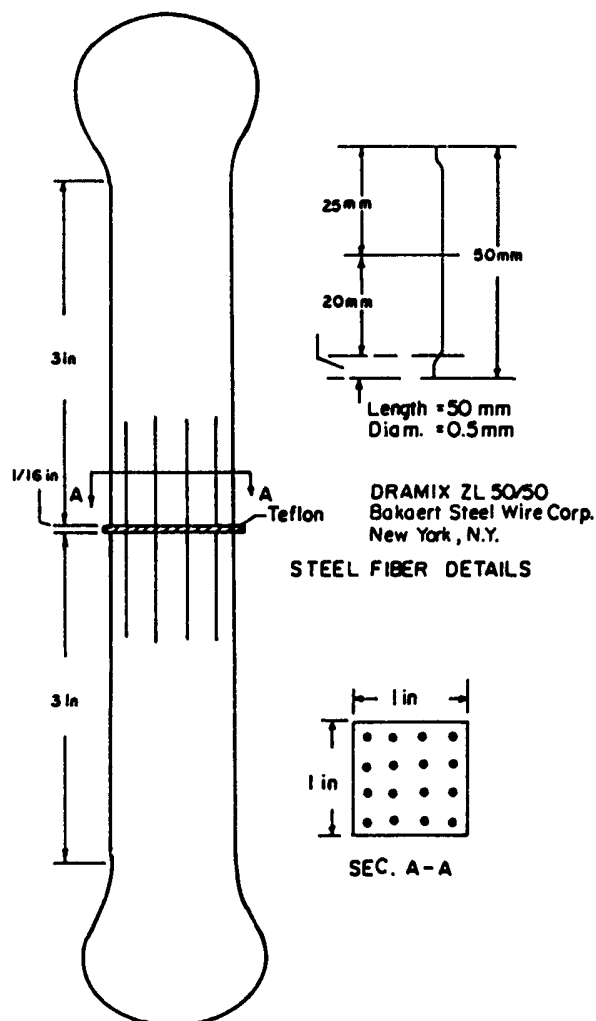


Fig. 2—Pullout test specimen

*DRAMIX ZL (50/50), manufactured by Bakaert Steel Wire Corporation.

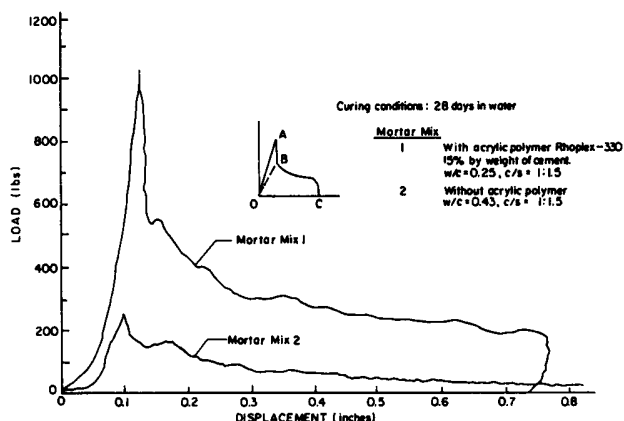


Fig. 3—Load-displacement curves from fiber pull-out test

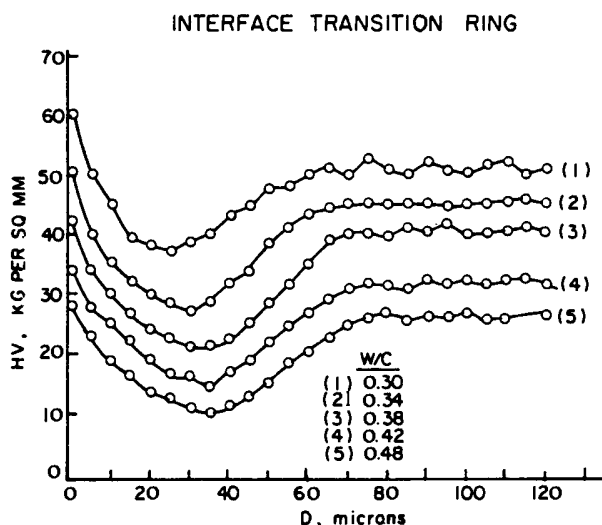


Fig. 4—Relationship between microhardness HV of the matrix (without polymer) and distance D from the fiber

The addition of the acrylic polymer might have caused a change in the structure of the material near the fiber matrix interface. Two possible changes are: (1) since the type of acrylic used is an active surface agent, during the mixing operation, it may have reduced the water-film thickness present around the fibers, and (2) because the acrylic polymer particle size (500 to 5000 Å) is much smaller than sand and cement particle size, it can fill smaller voids and thus prevent the formation of much of the microcracking that occurs in the matrix material and along the fiber-matrix interface.

INTERFACE TRANSITION RING

To study the fiber-matrix interface it is necessary to consider an annular region surrounding the fiber (interface transition ring). This is necessary because the properties in the matrix material in this region are different from those in the remainder of the matrix material. A conceptual model describing the formation of this special region begins during the mixing operation. At this time, a film of water forms on the surface of the

fiber. During mixing, cement particles move into the water film, but the concentration of particles remains smaller than the rest of the bulk material.

The direction of cement particle movement is towards the surface of the fibers. Thus, the lowest concentration of cement particles is closest to the fiber surface. The concentration gradually increases as the distance from the fiber surface increases. Cement solutes diffuse simultaneously with cement particles into the boundary film of water. When the matrix has hardened, further migration of cement particles is prevented and the interface transition ring is formed.

The structure and strength of the material in the interface transition ring is influenced by the thickness of the boundary film of water around the fiber. In general, the thinner the water film, the stronger the material. The thickness of the boundary film of water is generally reduced when the water-cement ratio is reduced. Since addition of a acrylic polymer to the mix allows a reduction in the water-cement ratio (with the same workability), it was anticipated that this would result in a stronger matrix material in the interface transition ring and a larger effective bond strength.

To substantiate this hypothesis, two series of experiments were conducted. In both series of tests, the strength of the material in the interface transition ring was estimated by measuring the microhardness of the material at locations in an annular region around a fiber. The width of this region was approximately 100 microns. In the first test series, the matrix material was cement. Matrices with water-cement ratios of 0.30, 0.34, 0.38, 0.42 and 0.46 were tested. In the second series of tests, the acrylic polymer was added to the mix.

The microhardness was measured with a microhardness tester. A Vickers indentation shape was used. The microhardness was measured five to seven times at each point. The distance between the measured points was two times the length of the diagonal of indentation. The measurements were made at locations where the particles were hydrated.

The results of these two series of tests are presented in Fig. 4 and 5. In Fig. 4 the microhardness values of the specimen without acrylic polymer are presented. Near the surface of the fiber, the microhardness ranged from approximately 27.5 kg/mm² (0.391 lb/in.²) for a water-cement ratio of 0.46 to over 60 kg/mm² (8.53 lb/in.²) for a water-cement ratio of 0.30.

The microhardness of the weakest point in the interface transition ring is an important value, as a failure in the region of the fiber-matrix interface may occur there. For the 0.5 mm diameter smooth steel fiber used in these studies, the weakest point was between 25 and 35 microns from the surface of the fiber (see Fig. 4). At this point values of microhardness ranged from 10 kg/mm² (1.42 lb/in.²) for a water-cement ratio of 0.46 to 37 kg/mm² (5.26 lb/in.²) for a water-cement ratio of 0.30.

The width of the interface transition ring is approximately 80 microns. For distances greater than 80 microns from the surface of the fiber, the microhardness

Table 2 — Microhardness of matrix materials

Water-cement ratio	Admixture (percent by weight of cement)	Curing conditions, days		Microhardness, kg/mm ²			Thickness of interface transition ring, microns	Distance from surface of fiber to weakest point, microns
		water	air	Adjacent to fiber	Weakest point	Outside transition ring		
0.30	None	28	—	60.0	37.5	50.0	50	25
0.34	None	28	—	50.0	27.5	45.0	60	30
0.38	None	28	—	42.0	27.25	40.5	65	33
0.42	None	28	—	33.75	14.1	31.5	70	35
0.46	None	28	—	27.5	10.1	25.5	75	36
0.25	Acrylic polymer (15 percent)	—	28	91.1	70.0	84.0	45	20
0.25	Acrylic polymer (15 percent)	7	21	85.0	65.0	88.0	52	25
0.30	Acrylic polymer (10 percent)	—	28	62.4	33.0	54.0	64	27
0.35	Water reducer (0.5 percent)	28	—	49.0	28.5	46.5	66	28
0.43	None	28	—	33.5	12.2	33.0	70	35

was constant for a given water-cement ratio. The values ranged from 25 kg/mm² (3.56 lb/in.²) (water-cement ratio of 0.46) to 50 kg/mm² (7.11 lb/in.²) (water-cement ratio of 0.30). Data from the first series support the hypothesis that the strength of matrix material in the interface transition ring is increased as the water-cement ratio is decreased.

In the second series of tests, 15 percent acrylic polymer, by weight of cement, was added to the mix. For a given workability, addition of the acrylic polymer permitted a significant reduction in the water-cement ratio. For example, the workability obtained without acrylic polymer, with a water-cement ratio of 0.43, can be achieved with a water-cement ratio of 0.25 if 15 percent acrylic polymer, by weight of cement, is added.

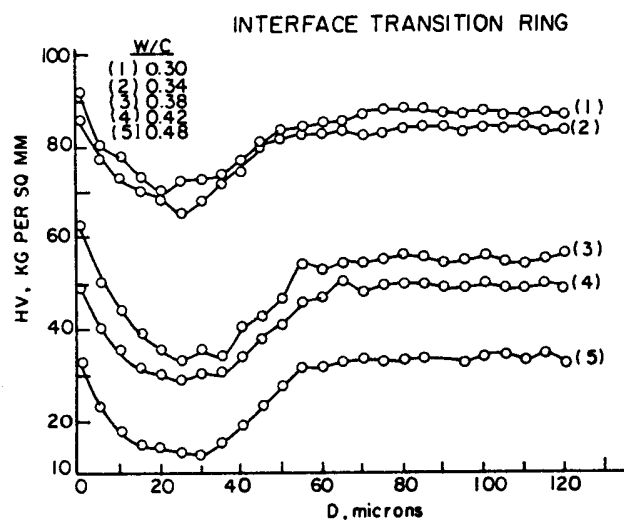
The microhardness of the matrix material near the surface of the fiber (see Fig. 5) varied from 33.5 kg/mm² (4.74 lb/in.²) to 91 kg/mm² (12.9 lb/in.²) for water-cement ratios ranging from 0.43 to 0.25, respectively. The microhardness values at the weakest point in the transition zone also increased when the water-cement ratio was decreased. Use of the acrylic polymer made mixing of materials with much lower water-cement ratios possible and yielded substantially higher values of microhardness.

Values of microhardness for matrix materials with and without acrylic polymer are summarized in Table 2. For corresponding distances away from the fiber surface and water-cement ratios, values of microhardness for matrix materials with and without acrylic polymer are almost equal.

A similar study using a water-reducing agent* yielded similar results (see Table 2). Note, the measured micro-

hardness values for a mortar-matrix material (water-cement ratio of 0.35) with water-reducing agent were close to those for a mortar-matrix material with acrylic polymer (water-cement ratio of 0.34).

A study was made to determine the influence of the fiber spacing on the magnitude and distribution of the



Sample	w/c	Admixture (percent by weight of cement)	Curing time, days	
			Water	Air
1	0.25	Acrylic polymer	—	28
2	0.25	Acrylic polymer	7	21
3	0.30	Acrylic polymer	—	28
4	0.35	Water-reducing agent (0.5 percent)	28	—
5	0.43	None	28	—

Fig. 5—Relationship between microhardness HV of the matrix (with polymer) and distance D from the fiber

*Mighty 150 (manufactured by ICI Americas Inc.).

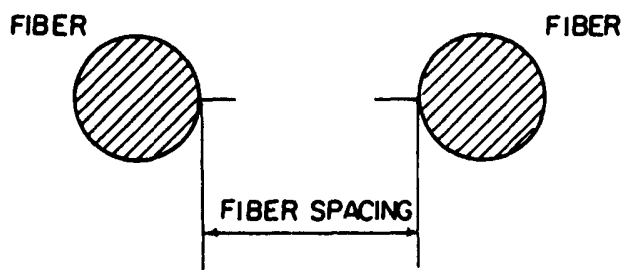


Fig. 6—Fiber spacing

microhardness values in the transition ring. In this study, two fiber spacings were used. They were 100 and 250 microns, measured from the adjacent edges of the two fibers (see Fig. 6). The fiber diameter used in this study was 0.5 mm (500 microns).

In Fig. 7 and 8, microhardness values are shown for the 100 micron fiber spacing. To show the influence of

fiber spacing, measurements were taken on specimens containing one fiber and specimens with two fibers. As indicated in Fig. 7 and 8, the minimum values of microhardness were higher when there were two fibers of diameter 0.50 mm spaced at 100 microns than when there was a single fiber. Note that 100 microns is approximately twice the width of the interface transition ring for a single fiber.

Two matrix materials were used in this study. The first material was portland cement having a water-cement ratio of 0.43. The second material was portland cement with a water-cement ratio of 0.25. Fifteen percent acrylic polymer, by weight of cement, was added to the second material. Both materials had the same workability. Microhardness values for the material with acrylic polymer were more than three times as large as those for the material without acrylic polymer. This was approximately the same percentage increase as was obtained in the studies reported earlier in this paper.

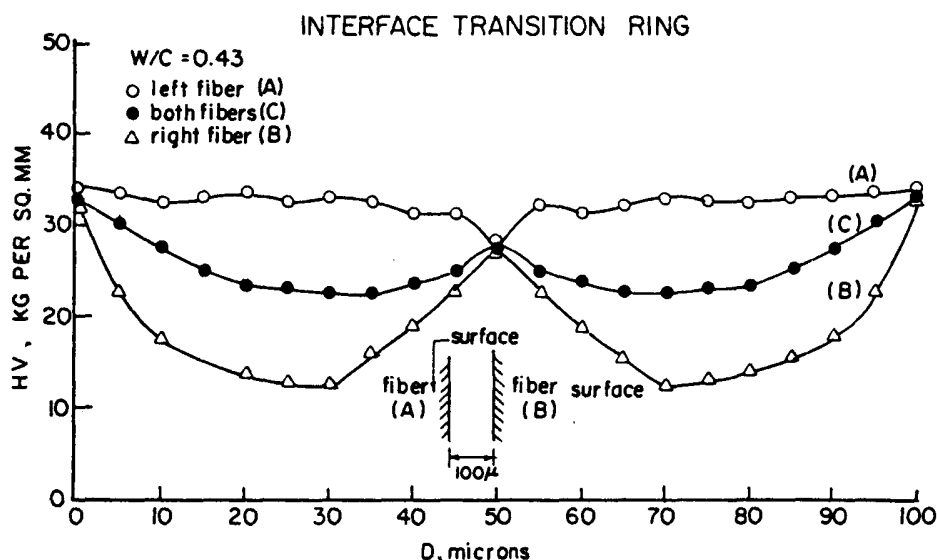


Fig. 7—Interface transition rings (without acrylic polymer)

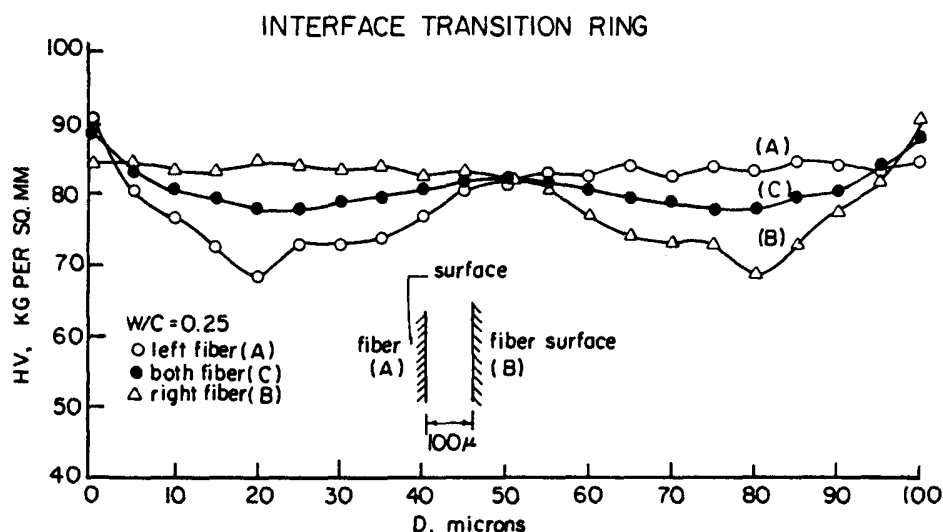


Fig. 8—Interface transition rings (with acrylic polymer)

A similar study was conducted with a fiber spacing of 250 microns. This spacing is more than twice the width of the interface transition ring. As expected, the values of microhardness in the interface transition ring of one fiber were not influenced by the adjacent fiber (Fig. 9 and 10). The microhardness values for the material with acrylic polymer were again significantly higher than those for the material without acrylic polymer.

MICROSTRUCTURAL FEATURES

To find a physical explanation for why the addition of acrylic polymer and reduced water-cement ratio produces a material with higher tensile strength, bond

strength, and microhardness, the microstructure of the material was studied with a scanning electron microscope. Fig. 11 through 16 are photographs taken with the scanning electron microscope.

In Fig. 11 through 14, the fiber-matrix interface is shown perpendicular and parallel to the longitudinal axis of the fiber. Cements both with and without the acrylic polymer were included. Notice that for the material without acrylic polymer, there was substantial cracking at and near the fiber interface.

In Fig. 15 and 16, the surface of the fiber is shown after the fiber-matrix interface had been broken. For the material without acrylic polymer, the surface of the

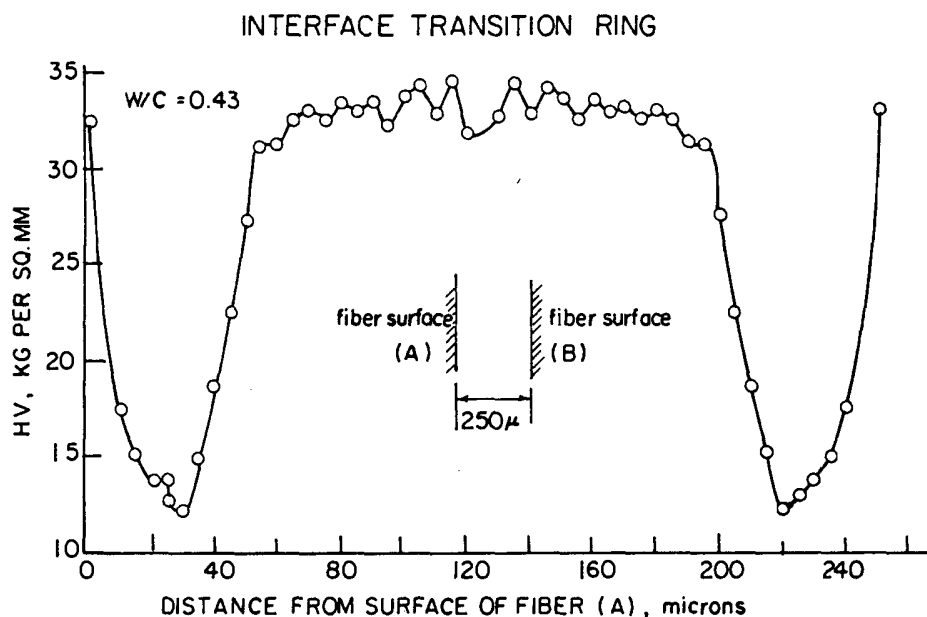


Fig. 9—Interface transition rings (without acrylic polymer)

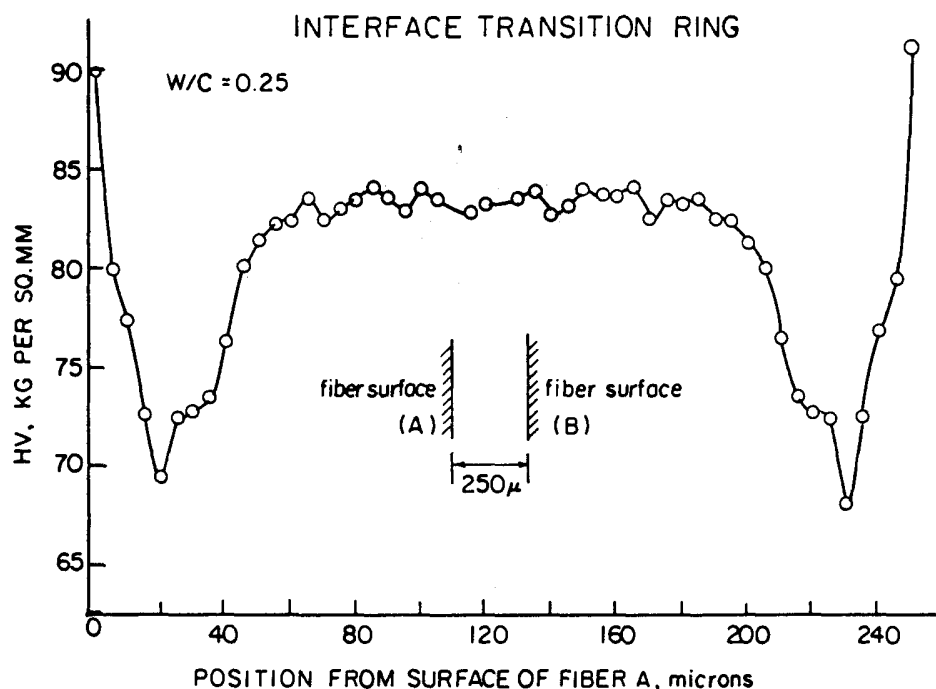


Fig. 10—Interface transition rings (with acrylic polymer)

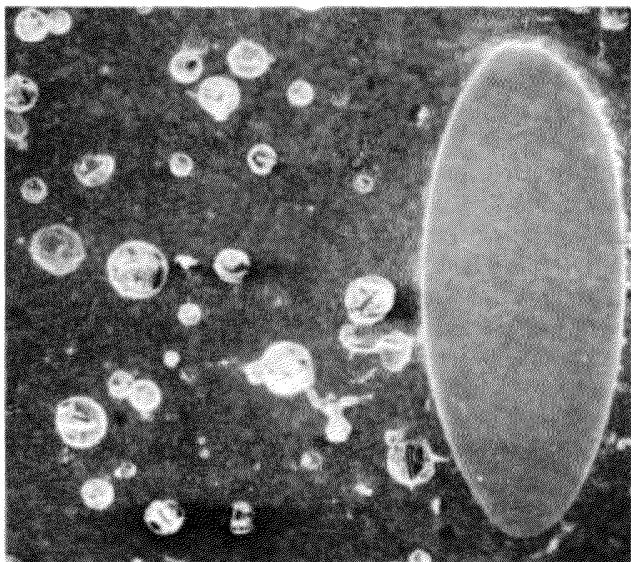


Fig. 11—Interfacial features (cement matrix, water-cement ratio—0.25, polymer—Rhoplex E-330 [15 percent by weight of cement]) (50X magnification)

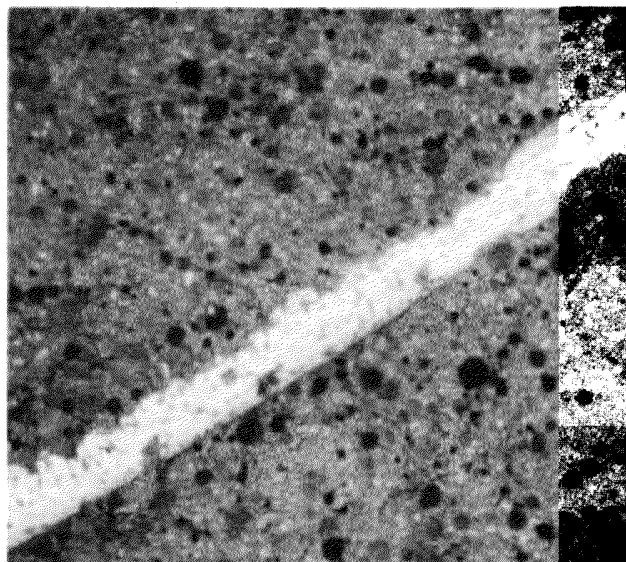


Fig. 12—Interfacial features (cement matrix, water-cement ratio—0.25, polymer—Rhoplex E-330 [15 percent by weight of cement]) (50X magnification)

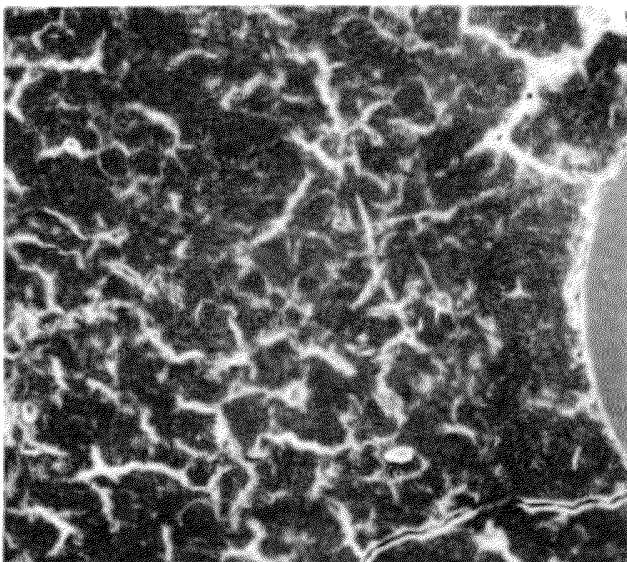


Fig. 13—Interfacial features (cement matrix, water-cement ratio—0.43, without polymer) (150X magnification)

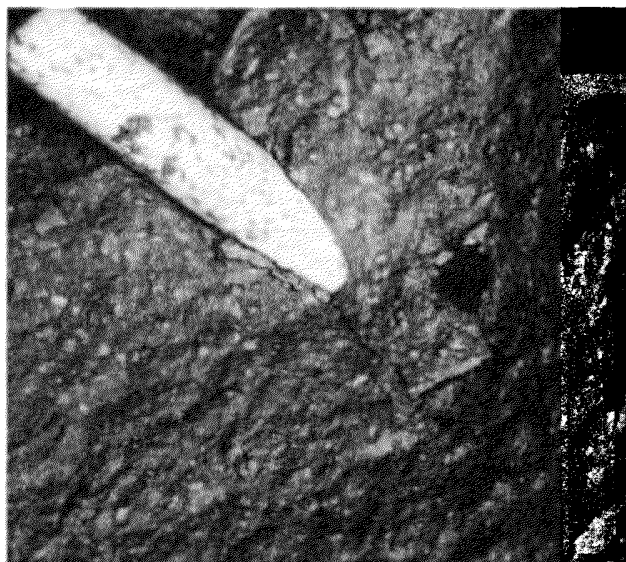


Fig. 14—Interfacial features (cement matrix, water-cement ratio—0.43, without polymer) (50X magnification)

fiber was relatively clean (free of cement particles). For the material with acrylic polymer, however, the surface of the fiber had cement particles adhered to it. This of course indicated a higher bond strength.

CONCLUSIONS

1. Reducing the water cement ratio increased the tensile strength and microhardness of mortar and the bond strength between mortar and steel fibers. Increases in strength were approximately the same (370 percent) for a reduction in water-cement ratio from 0.43 to 0.25.

2. Adding acrylic polymer or water-reducing agent made possible the manufacture of materials with low water-cement ratios and high tensile strength, bond strength, and microhardness.

3. Cement materials with acrylic polymer exhibited less cracking in the region of the fiber-matrix interface. This may have been due to the filling of small cracks by the acrylic polymer due to its small particle size.

4. After the fiber-matrix bond had been broken, cement particles were still bonded to the fiber when the cement contained the acrylic polymer. For cement without acrylic polymer, the fiber was relatively smooth

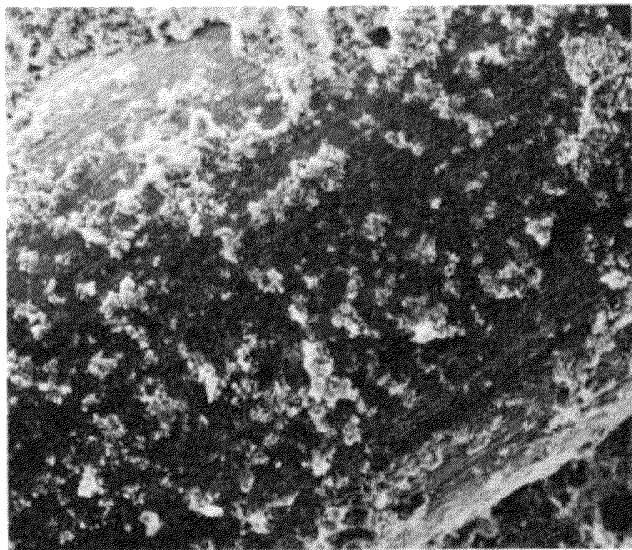


Fig. 15—Fiber surface (cement matrix with 15 percent by weight of cement of Rhoplex E-330, water-cement ratio—0.25) (400X magnification)

after the fiber-matrix interface had been broken. This indicated a higher bond strength for cements with acrylic polymer.

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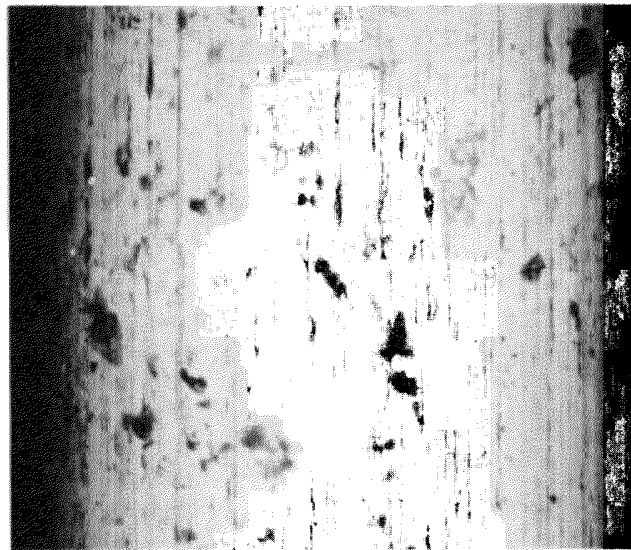


Fig. 16—Fiber surface (cement matrix without acrylic, water-cement ratio—0.43) (150X magnification)

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