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Performance of Glass Fiber-Reinforced Polymer Reinforcing Bars in Tropical Environments—Part II: Microstructural Tests

by Abhijit Mukherjee and S. J. Arwikar

In the first part of this study, the structural scale tests on the synergistic effects of moisture, temperature, alkalinity, and stress level on the performance and durability of glass fiber-reinforced polymer (GFRP) reinforcing bars in concrete have been discussed. In this part, investigations on microstructural studies, carried out to find out the nature, quantum, and mechanism of deterioration in the conditioned reinforcing bars, are reported. Micrographic investigations were carried out using a scanning electron microscope (SEM) to visualize the changes in the microstructure. The other tests that have been carried out are energy-dispersive x-ray analysis (EDX) and inductively coupled plasma mass spectrometry (ICP-MS) to determine the chemical changes in the composite.

Keywords: bars; fibers; hot weather; polymers; testing.

INTRODUCTION

In the first part of the paper, the structural scale test of glass fiber-reinforced polymer (GFRP) reinforcing bars in concrete in a tropical environment was discussed. The structural scale tests revealed that, although the strength of the beams went up during environmental conditioning, the reinforcing bars were found to have lost up to 65% of their strength. Another interesting observation was that, although the strength of the reinforcing bars was drastically affected, the environmental conditioning did not significantly affect the stiffness of the reinforcing bars. In this part, the microstructural tests to investigate the reasons for such behavior are reported.

At present, no standard durability test method exists, and this makes the results obtained by different researchers difficult to compare. As a result, researchers report degradation of GFRP reinforcing bars varying from 4.9 to 100% depending on the parameters selected for durability tests, namely, alkalinity, temperature, stress, and duration of the tests.[1-11] The investigators, however, agree on the susceptibility of reinforcing bars to alkali attack and this mechanism has been highlighted in all durability tests. The chemical composition of E-glass is 54.3% SiO_2; 15.2% Al_2O_3 and Fe_2O_3; 17.3% CaO; 0.6% Na_2O/K_2O; and 8 to 10% B_2O_3.[12,13] In an alkaline environment, glass is attacked by hydroxide ions causing hydrolysis (Eq. (1)).[14,15]

$$Si–O–Si + OH^- \rightarrow –Si–OH(solid) + Si–O^-(solution) \quad (1)$$

As concrete creates a highly alkaline environment base, hydrolysis of the matrix can be expected to some extent. The models built to predict degradation are based on the diffusion theory. For stressed reinforcing bars in a high-alkaline environment, they do not yield satisfactory results.[11] To estimate and visualize the damages in the GFRP, the tests carried out by various researchers are tensile tests and scanning electron microscope (SEM) tests.[1-11] The SEM micrographs show degradation of glass fibers.[5,11]

In the present investigation, the SEM images have been quantified by energy-dispersive x-ray analysis (EDX), an accessory to SEM, which allows simultaneous nondestructive elemental analysis of the sample at a selected spot of approximately 2 microns wide. An EDX test was performed to find changes in chemical compositions at selected points in reinforcing bars. Though x-ray diffraction (XRD) and x-ray fluorescence (XRF) are preferred tests by metallurgists, these did not yield clarity for the nonmetallic substances like glass and polymer. ICP-MS is a very powerful tool for trace (parts per billion to parts per million) and ultra-trace (parts per quadrillion to parts per billion) elemental analysis. This test was performed to identify the chemical changes in the conditioned reinforcing bars.

RESEARCH SIGNIFICANCE

GFRPs are predicted to have great potential use as reinforcement in concrete. In practice, they have started to get a toehold in tropical regions. The use of GFRP reinforcing bars in construction is hampered by the lack of long-term durability and structural performance data. In this paper, concrete-GFRP beams have been subjected simultaneously to stress and an alkaline environment, as well as temperature and humidity conditions of the tropics. Structural scale behavior such as load-deflection and durability has been studied in Part 1. In Part 2, microstructural scale tests that assess the nature, quantum, and mechanism of degradation are reported.

Alkalinity

First, the alkalinity of the concrete was measured. Three beams were cast and cured in water for 28 days. The beams were allowed to dry for 1 month. Then parts of beams were crushed to be very fine. The sample passing 300 micron sieve and retained on 150 micron sieve was taken for alkalinity testing. The powder was diluted in water in 1:1, 1:5, and 1:10 ratios. The pH values were 12.25, 12.182, and 12.05, respectively. The actual pH value at the hydrates level was expected to be higher than these values.[13] It may be noted that in the concrete mixture, normal portland cement has

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Abhijit Mukherjee *is a professor in the Department of Civil Engineering at the Indian Institute of Technology, Bombay, India. His research interests include composite and smart materials, especially their applications in structures.*

S. J. Arwikar *is an assistant professor in B.L.D.E.A's Dr.P.G. Halkatti College of Engineering and Technology, Bijapur, India. His research interests include the application and durability of composites in concrete structures in tropical environments.*

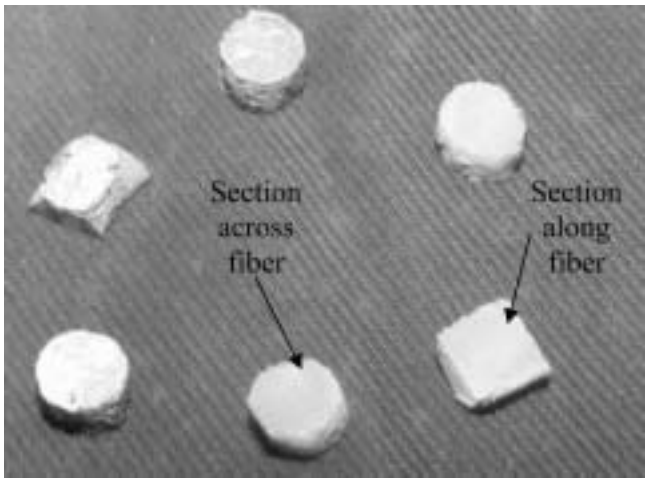

Fig. 1—Specimens from GFRP reinforcing bars after polishing.

been used without any other cementitious materials. As a result, the pH of concrete is rather high and the glass may be susceptible to alkali attack. The mixture proportion used here is commonly used in this part of the world. To study the conditions of the exposed reinforcing bars, the SEM and EDX tests were employed.

MICROSTRUCTURAL TESTS

SEM and EDX tests

Specimens—It may be recalled that, in this investigation, concrete beams with a single GFRP reinforcing bar were cast. The beams were conditioned for accelerated aging for different durations under service loads. The calculated level of stress in the reinforcing bar at service load was 89 MPa. The conditioned beams were tested and subsequently the reinforcing bars were dug out of them. A substantial reduction in the failure stress of the reinforcing bars was observed. The micrographic investigation is aimed at establishing the reasons for degradation. The micrographic test samples were collected from the location not more than 10 mm away from the location of the fracture of the reinforcing bars. Specimens of approximately 9 mm dimensions were cut out from reinforcing bars along and across the direction of fiber as shown in Fig. 1. The surface was polished using sandpaper with grits of 300, 600, 800, and 1200 in sequence. Afterward, fine polishing was done using an emery cloth and a wet-polishing agent. As GFRP is a nonconducting material, the polished specimens were gold-coated. A minimum of six samples for each conditioned reinforcing bar were prepared for the tests. An SEM was used to obtain the micrographs.

Fresh reinforcing bar—Figure 2 shows the micrograph of the original reinforcing bar. The magnification scale is presented by a marked line length. It can be seen that fibers have different diameters, and the average diameter is 20 μm. The packed density of fibers within the vinylester matrix is high and is as per the manufacturer's specification of 70% minimum (fiber content by weight—ASTM D 2584). To find microcracks, air bubbles, and manufacturing defects in the

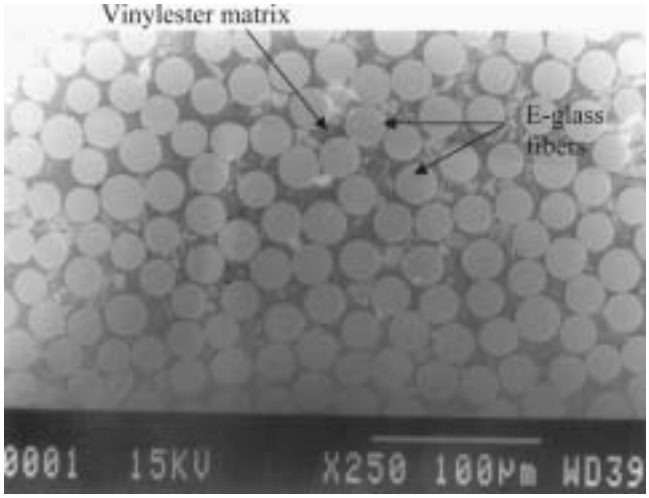

Fig. 2—Micrograph showing distribution of E-glass fibers in original GFRP reinforcing bar. (Note: X250 = magnified 250 times and showing a line scale of 100 micrometers.)

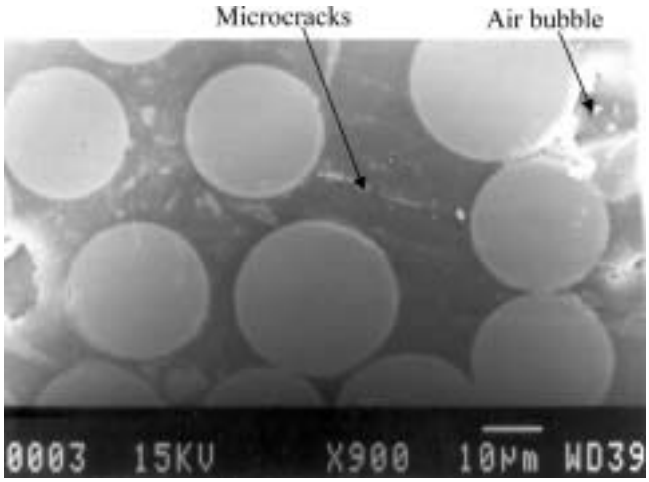

Fig. 3—Micrographs of GFRP reinforcing bar showing size of fiber and defects. (Note: X900 = magnified 900 times and showing a line scale of 10 micrometers.)

original reinforcing bar, it was magnified 900 times further (Fig. 3). Here, it can be seen that there are scattered air bubbles and microcracks in the matrix. Because of the thin exterior coating of matrix on the fibers, these manufacturing defects may form the pathway for ingress of moisture and alkali.

Reinforcing bar conditioned for 3 months at 60 °C—Samples were made out of the reinforcing bars dug out of the conditioned beams. The portions of the reinforcing bars near the cracks of the beams were chosen for the tests. Figure 4 shows the cross-sectional view of the reinforcing bar near the surface of the bar. The voids in the reinforcing bar are identified by white spots. It can be seen that many voids have developed in the reinforcing bar, especially in the exterior 1 mm region of the reinforcing bar. If Fig. 4 is compared with Fig. 2, one can identify that these voids developed due to the conditioning of the reinforcing bar. The voids in the core region are sparse and smaller in size. To have a closer look at the voids, a magnified (112×) view of the reinforcing bar was taken (Fig. 5). From the figure, it can be seen that the voids have been created in the matrix region of the reinforcing bar. The fibers have remained largely unaffected. The matrix is

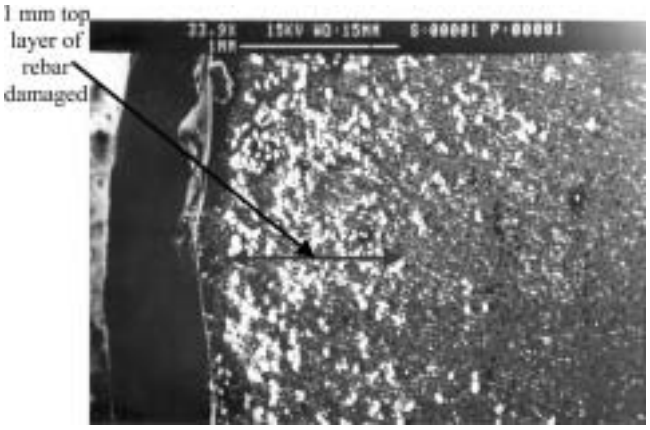

Fig. 4—Damage near reinforcing bar surface (B3-900).

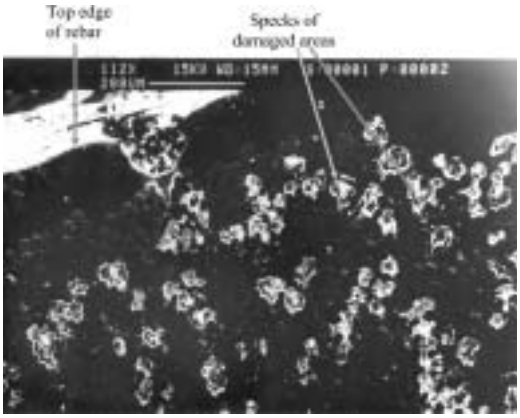

Fig. 5—Enlarged area near surface of GFRP reinforcing bar (B3-900).

Fig. 6—Damaged area on outer layer of GFRP reinforcing bar (B3-900).

clearly degraded on the surface of the reinforcing bar, which is the entry zone for the moisture/alkali inside the reinforcing bar. The typical size of the voids was 30 μm.

The earlier micrographs gave an idea of damage across the fiber. The transverse section of the reinforcing bar is magnified to understand the ingress of the voids along the length of the fiber. A magnified (890×) view near the reinforcing bar surface is shown in Fig. 6. The voids are created along the length of the fiber/reinforcing bar. In this sample, the maximum damaged zone is 250 μm inside from the surface of the reinforcing bar. At this stage, the matrix is damaged much more than the fiber. Further, it can be seen in Fig. 6 that an approximately 600 μm length of the matrix is damaged at a distance of 400 μm from the edge of the reinforcing bar. The major areas near the edge are unaffected. Thus, the damage to the matrix is local in nature.

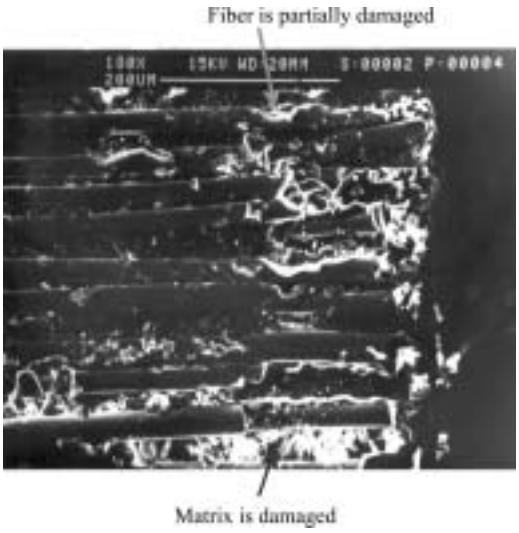

Fig. 7—Damaged area near ruptured surface of reinforcing bar.

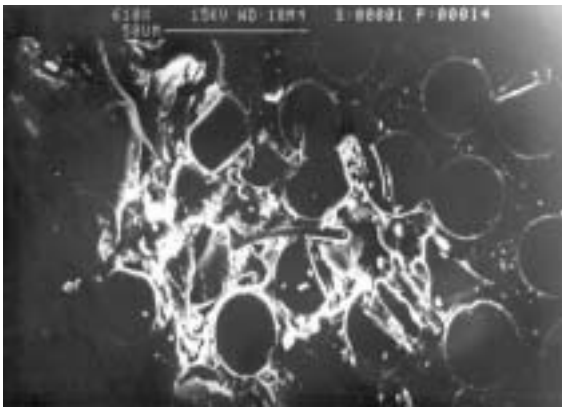

Fig. 8—Enlarged view of single void (B3-900).

To visualize the nature of damage along the length of the fibers, specimens were taken from the ruptured reinforcing bar and a magnified (180×) view is given in Fig. 7. It can be seen that the fibers near to the surface of the reinforcing bar (top of micrograph) have started to corrode. The fibers at the bottom of the micrograph and regions away from the rupture are intact even though the matrix in that region is damaged. Thus it can be said that the matrix is damaged first and then the fibers start disintegrating. It is also seen that the fibers near the ruptured surface are damaged and the fibers away from the ruptured surface are unaffected, signifying that causes of damages are local in nature. To visualize the damage pattern in the voids, a highly magnified (610×) view of only one void is shown in Fig. 8. The size of the void is approximately 60 x 70 μm. Approximately five to six fibers are damaged in this void.

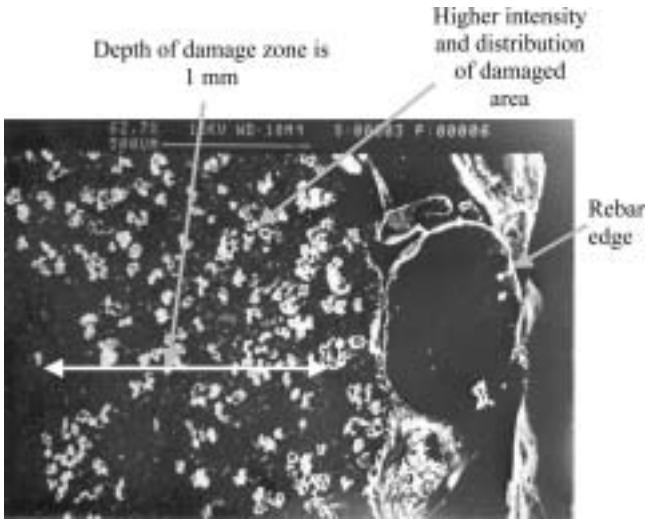

Fig. 9—Damage near surface of GFRP reinforcing bar (B6-900).

Reinforcing bar conditioned for 6 months at 60 °C—The cross section of the reinforcing bar after 6 months of conditioning is magnified (627×) to assess the comparative damages in the matrix and fibers near the surface. Figure 9 gives an enlarged view of approximately 1.5 mm depth near the surface of the reinforcing bar. It can be seen that the nature of the damage is the same as for the 3-month-conditioned reinforcing bar (Fig. 5). Here also, the voids have developed in the exterior 1 mm region of the reinforcing bar. However, there is a distinct increase in the number and size of the voids. Voids as large as 125 μm can be seen in Fig. 9. In the 3-month-conditioned reinforcing bars, the maximum size of voids was approximately 70 μm.

To visualize the distribution of voids along the fiber/reinforcing bar length, a magnified view (646×) near the reinforcing bar surface is shown in Fig. 10. It can be seen that the damage in the matrix is distributed along the length of the specimen. The reinforcing bar strength loss was approximately 56% in 6 months compared with a 42% loss in 3 months. The reinforcing bars lost their strength depending on the percentage of damage to the fibers. In both cases, the matrix was damaged first and then these damaged portions formed the pathway for the ingress of moisture in the reinforcing bar. To substantiate this, an enlarged micrograph is given in Fig. 11. It can be seen in the figure that the matrix around the fiber is damaged and the fiber is intact within the damaged matrix.

Reinforcing bar conditioned for 12 months at 60 °C—Figure 12 shows the cross section of the reinforcing bar after 12 months conditioning at 100× magnification. A 1.5 mm depth near the surface of the reinforcing bar is shown here. It can be seen that the nature of damage is same for the 3-month-conditioned reinforcing bar (Fig. 5) and 6-month-conditioned reinforcing bar (Fig. 9). Here also, the voids have developed in the exterior 1 mm region of the reinforcing bar. The maximum void size that could be seen is approximately 140 μm compared with the 125 μm void size in the 6-month-conditioned reinforcing bar. However, the rate of growth of the voids had reduced. It can be interpreted that the sizes of voids stabilized after 12 months of conditioning, but damage to fibers increased with conditioning time, as shown in Fig. 13. While scanning for damage in the fibers, many areas were found where the fibers were intact even after

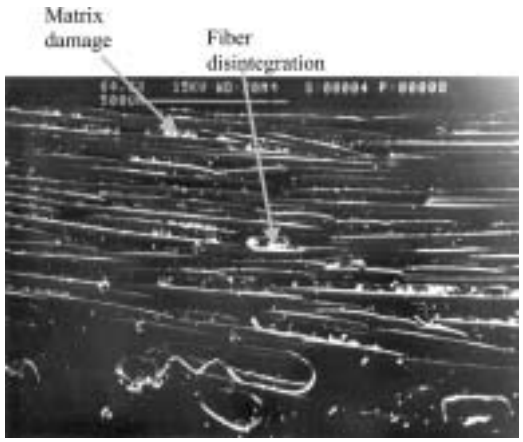

Fig. 10—Micrograph of transverse section of reinforcing bar (B6-900).

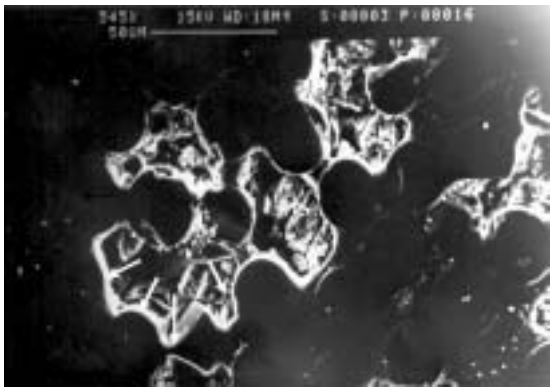

Fig. 11—Damage from matrix progresses to fibers (B6-900).

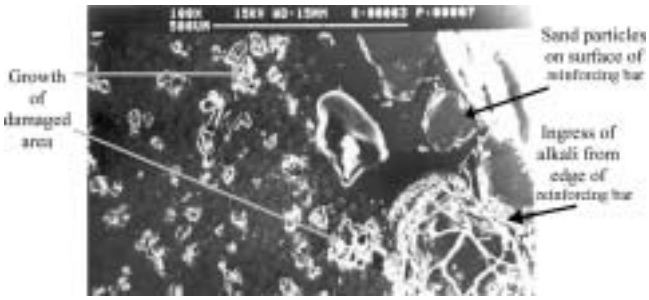

Fig. 12—Micrograph done on surface of GFRP reinforcing bar.

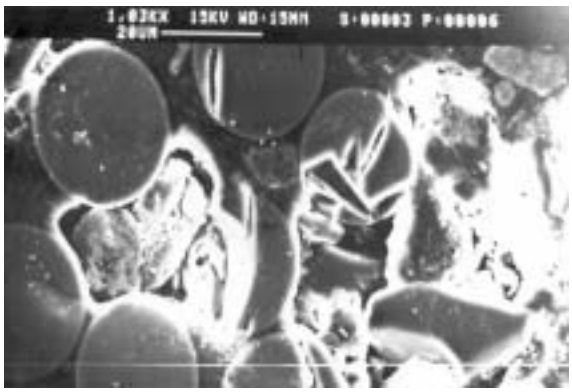

Fig. 13—Micrograph showing damage to fibers after 12 months of conditioning (B12-900).

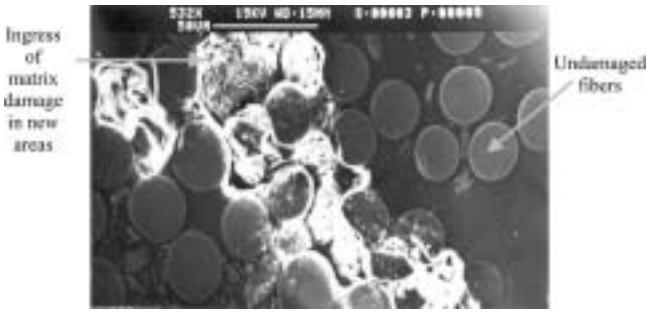

Fig. 14—New area in matrix getting damaged after 12 months of conditioning (B12-900).

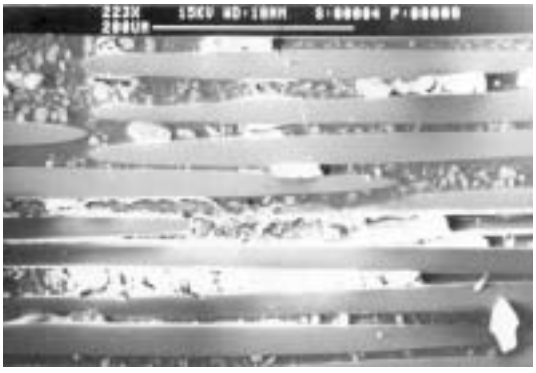

Fig. 15—Micrograph of transverse section showing flakes of matrix (B12-900).

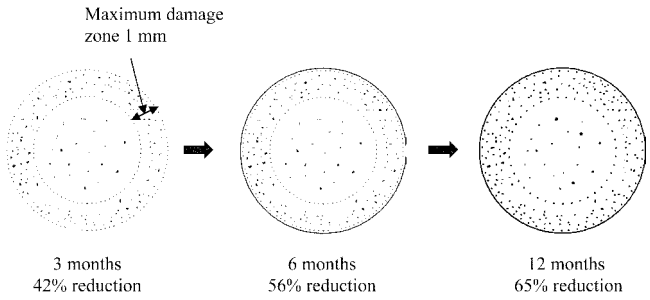

Fig. 16—Progressive damage pattern of 9.5 mm diameter GFRP reinforcing bar due to conditioning at 60 °C and percentage reduction in tensile strength.

12 months of conditioning. In these specific areas, the matrix was unaffected and it protected the fibers (Fig. 14). Thus, it is important to have a matrix resistant to moisture and alkali attack because it is the first line of defense against damage to fibers. In Fig. 14, it can also be observed that new areas of the matrix are getting damaged with progressive conditioning. Further damage to reinforcing bars with conditioning could be expected.

The moisture absorption test was carried out for fresh and conditioned reinforcing bars. The fresh reinforcing bar absorbed 1.2% moisture by weight in 30 days, while the 12-month-conditioned reinforcing bar absorbed 3.5%. This suggested that due to conditioning, the matrix at some regions had disintegrated. To visualize this characteristic of the reinforcing bar, a transverse section is magnified (223×), as shown in Fig. 15. It can be seen that damage of the matrix has resulted in flakes coming off the specimen. In some places, the matrix had become powdery.

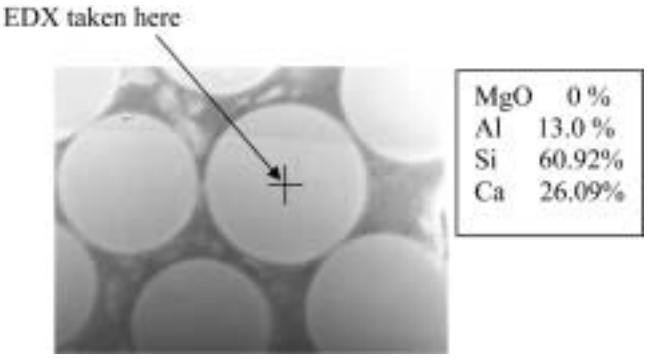

Fig. 17—EDX on original reinforcing bar specimen (B0-900).

The effect of conditioning and pattern of damage are summarized in Fig. 16. The representative diagram of what was seen in the SEM while scanning for damaged zones is given herein. The damaged zones were concentrated near the surface layer of the reinforcing bar (1 mm). The damage front did not proceed appreciably with time, but the number of pockets of damage increased with time. The sizes of the pockets also increased at a decreasing rate. However, the damaged zone was very localized. At the locations where the reinforcing bar had eroded, the damage could be seen; but in the portions away from the edges, there were no damaged pockets at all. It may be noted that strength is determined by local weakness, while the stiffness manifests the overall stress-strain characteristics of the reinforcing bar. Therefore, the stiffness remained largely unaffected by local damage. However, the local damage reduced the strength of the reinforcing bar. SEM revealed the nature and magnitude of degradation of the reinforcing bar. Next, the EDX test was performed on reinforcing bars to assess chemical changes that caused the damage.

Energy-dispersive x-ray analysis (EDX) test

EDX on undamaged E-glass fiber—The SEM tests have shown that the vinylester matrix and E-glass fibers are damaged due to accelerated conditioning. When an incident electron beam hits atoms of the sample, secondary and back-scattered electrons are emitted from the sample surface. The x-rays emitted from the sample atoms are characteristic in energy and wavelength to the element of the parent atom, which is used to identify and quantify the elements. The focus then became to find if any changes in chemical composition took place in the E-glass fibers. To evaluate the chemical changes in the specimens, EDX was done on a few critical samples. Locations where SEM revealed damage and where there was no damage were selected for the test.

To compare the chemical composition of E-glass fibers, EDX was done at the center of the E-glass fiber. The samples were taken from the original reinforcing bar, reinforcing bar conditioned in the tank for 9 months, and reinforcing bar conditioned for 12 months in the tank, as shown in Fig. 17 to 19. Chemical compositions obtained by EDX have been given along with the figures. The silica content in E-glass fiber is 60.92%, 57.49%, and 60.6%. The calcium content is 26.09%, 30.99%, and 25.83%. Thus, there were no substantial chemical changes taking place in E-glass fiber which were not attacked due to the deterioration of the matrix.

EDX on damaged E-glass fiber—The EDX test on undamaged E-glass fiber did not show any composition changes of the fiber. The EDX test was done in the damaged

Table 1—Comparative values, in ppm, of various elements in GFRP reinforcing bars

Sample	SiO_2	Al_2O_3	MgO	CaO	Na_2O	K_2O
Original reinforcing bar	40.15	10.34	2.67	12.36	0.40	Low*
Reinforcing bar from 12-month beam	46.05	11.94	2.82	12.38	0.40	Low*

*Less than 0.1 ppm.

Table 2—Comparative values, in ppm, of various elements in water extract of GFRP reinforcing bars

Sample	Si	Al	Mg	Ca	B	Na	K
Original reinforcing bar	69.39	1.88	16.83	54.21	3.63	Low*	11.86
Reinforcing bar from 12-months beam	168.88	ND*	10.73	201.94	4.82	Low*	12.31

*Less than 0.1 ppm.

zones. The matrix vinylester was an organic substance. Hence, it became easy to assess the elements present in the damaged zones. The EDX test in the damaged zone (Fig. 20) gives the composition of silica as 39.43% and calcium as 1.47%. The values in undamaged fiber are 60.6% and 25.83%, respectively. Similarly, one more test in the damaged zone gave silica and calcium contents as 56.93% and 3.78%. One can infer that silica and calcium content in a damaged area is much less than that in the healthy fiber. This is only possible if the molecular structure of the fiber is broken wherever it comes in contact with some degenerating agent. This agent displaces silica and calcium from the healthy fibers.

This test, however, did not reveal the reason for the molecular breakdown of the E-glass fiber. Hence, a chemical investigation was performed. An ICP-MS test was done to identify the chemical inducing molecular breakdown of the E-glass fiber.

ICP-MS test

The objective of this test is to find free silica and alkali in the specimens and the reason for it. In this test, the elements are extracted into a solvent for analysis. The original reinforcing bar and reinforcing bar cut out from beams conditioned for 12 months were pounded and powdered separately. The powdered specimens were taken up for further investigation to find elements present in the GFRP reinforcing bar.

Fused powdered specimens—0.2 g of sample (powdered reinforcing bar) was fused at 1000 °C with 0.6 g of lithium meta-borate ($LiBO_4$) and 0.2 g of lithium tetra-borate (LiB_4O_7). The solid powder fuses at high temperature. After cooling, the solid mass was dissolved in 30% nitric acid. One hundred mL of the solution was used for the ICP-MS test. The results in percentage are given in Table 1. The results in Table 1 indicate that the E-glass fibers from the original specimen and the conditioned specimen have the same percentage of chemical elements. Conditioning for 12 months did not induce any chemical changes in the E-glass fiber. This has also been corroborated by EDX testing.

Water extract of powdered specimens—To identify the liberated compounds in the reinforcing bar (Eq. (1)), water extract was taken up for further investigation. Twenty-five g of powdered sample was boiled in distilled water in a Teflon beaker for nearly 3 h. Forty mL of distilled water were reduced to 20 mL. This water extract was used for the ICP-MS tests. The results are tabulated in Table 2. From Table 2,

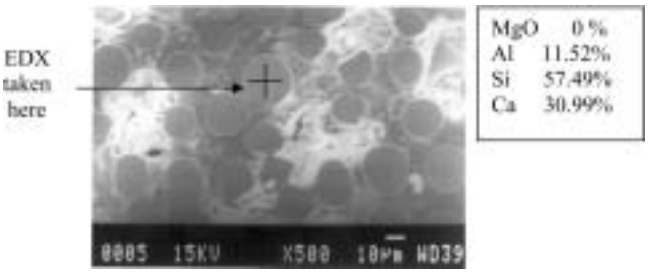

Fig. 18—EDX on reinforcing bar specimen conditioned for 9 months in open tank at 60 °C.

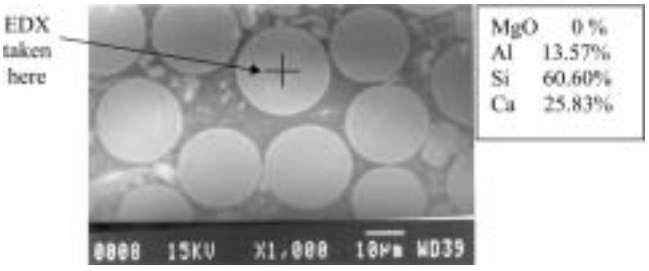

Fig. 19—EDX on reinforcing bar specimen conditioned for 12 months in tank at 60 °C (B12-900).

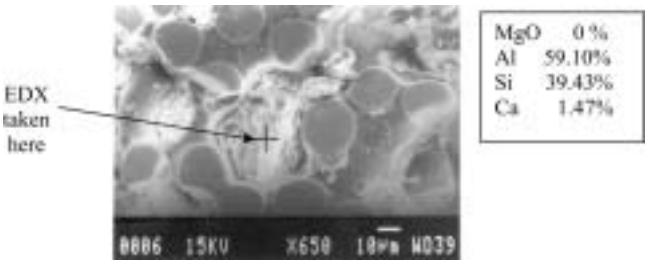

Fig. 20—EDX on reinforcing bar specimen conditioned in open tank at 60 °C for 9 months.

it is seen that free silica in an original reinforcing bar specimen was 69.37 ppm, whereas in a conditioned specimen, it was 168.88 ppm. The increase is more than 200%. This extra silica is liberated from the E-glass fiber.

Again in Table 2, it is seen that free calcium in the original reinforcing bars was 54.51 ppm, whereas in the conditioned reinforcing bars, it was 209.14 ppm. The increase in calcium is enormously high at 284%. This high value of calcium can only come from alkali $Ca(OH)_2$. This alkali is liberated from the concrete due to hydration of cement and clearly is absorbed in the conditioned reinforcing bars with time. Thus, the chemical responsible for the breakdown of the molecular structure of E-glass is $Ca(OH)_2$, or alkali. The reaction suggested is given in Eq. (1). This phenomenon has been termed as alkali attack on glass fibers.

The ICP-MS results showed that alkali is absorbed in the reinforcing bar. As the fibers are protected by the matrix, matrix degradation must precede the damage of the fibers. This is only possible if the vinylester matrix is breaking down due to hydrolysis by alkali. It can be concluded that due to the presence of alkali, high temperature, and stressing of the reinforcing bars, the vinylester disintegrated with conditioning time and failed to protect the E-glass fibers. E-glass fibers eventually degenerated due to alkali attack.

Therefore, the conditioned reinforcing bars progressively lost their tensile strength by 42, 56, and 65% in 3, 6, and 12 months, respectively. As alkali attack is localized in the area of cracks in the reinforcing bars, the elastic modulus of the reinforcing bar did not change more than 6%. The tests revealed that the present reinforcing bar is susceptible to environmental attack, especially in hot and humid tropical conditions. The degradation is triggered by the deterioration of the matrix that allows the attack on the fibers. The matrix may have to be redesigned to develop a more durable composite.

SUMMARY AND CONCLUSIONS

The objectives of the experiments were to visualize degradation, identify the mechanism, and explain the high degradation of the reinforcing bar due to conditioning in a tropical environment. With this view in mind, microstructural tests were conducted.

The SEM observations on original reinforcing bars points to defects in the form of air bubbles and microcracks that could have formed during the manufacturing process and then formed the pathway for the ingress of moisture. SEM tests showed that reinforcing bars get damaged progressively due to conditioning. Damages in the form of voids can be seen in the micrographs. The voids are of maximum numbers near the surface of the reinforcing bars, forming an approximately 1 mm deep annular ring. The voids grow in number and size with conditioning. The maximum void size was approximately 70, 125, and 140 μm with 3, 6, and 12 months of conditioning, respectively. The voids are formed due to the degradation of the matrix. The fibers in the void disintegrated with time, resulting in a loss of tensile strength of the reinforcing bar. Thus, the matrix gets damaged first and then the fibers get damaged due to conditioning. As per the micrographs, the matrix disintegrates completely in 12 months and starts to flake. The absorption of water in the matrix increased dramatically at this stage.

Both EDX and ICP-MS tests showed that no chemical change takes place in the unaffected portions of the fibers. SEM micrographs show the damaged zones are scattered and localized in the reinforcing bars due to conditioning. This could be the reason why the reinforcing bars have not lost their stiffness despite losing their strength. A loss of approximately 6% in modulus of elasticity was noted.

The ICP-MS test on water extract of 12-month-conditioned reinforcing bar shows an excessive increase in the values of calcium (approximately 284%) and silica (approximately 200%) compared with an unconditioned specimen. This excess calcium can only come from the free alkali present in concrete. The excessive silica can come only from the molecular breakdown of the E-glass fiber. This molecular disintegration is observed in the SEM micrographs. This displacement of Si from the molecular structure of E-glass fiber leads to the collapse of the structure of the fiber and is termed alkali attack. This takes place in the regions where the fiber comes in contact with alkali. The micrographs show the notching, and etching of fibers with void formation within the glass fiber. This test reveals that the matrix of the reinforcing bar must be redesigned to resist temperature, moisture, and alkalinity conditions of tropical regions.

The synergistic effect of alkali, temperature, stress has caused high degradation of the reinforcing bars. Stressing of reinforcing bars cracks the matrix, and moisture/alkali is able to penetrate the reinforcing bar through the microcracks. Higher temperatures increase the diffusion rate. The alkali damaged the matrix first and then the fibers.

If reinforcing bars are to be used in concrete, then a suitable environmental reduction factor has to be used in design, depending on the severity of alkali attack. More tests with different temperatures, alkalinity, and prestressing stresses are required to build a model to predict the rate and magnitude of damage to the fibers. The use of ground granulated blast-furnace slag and fly ash in concrete to control free alkali is an area of further research to improve the performance of the GFRP reinforcing bars in concrete.

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