Microstructural Investigation of Glass Fiber Reinforced Polymer Bars

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Abstract

Microstructural characteristics are crucial in understanding and predicting the behavior of glass fiber reinforced polymer (GFRP) bars used for concrete reinforcement. Considering the lack of extensive GFRP microstructural knowledge, the main purpose of this study is to provide a documentation of GFRP microstructure and demonstrate its contribution in the durability of GFRP bars. Scanning electron microscopy (SEM) imaging was performed at different magnification levels on the cross-section of four different commercially available pristine GFRP bars. As a result of differences in the production method by pultrusion, each pristine bar presented a unique microstructural pattern including voids, defects, and fiber distribution. Two of the bars which demonstrated the most different patterns were exposed to accelerated conditioning in alkaline solution. The horizontal shear test was performed and the results were compared with the pristine bars. The difference in microstructural patterns was found to significantly contribute to GFRP durability. These results can be used as a benchmark for the microstructure of commercially available pristine GFRP bars and serve as a base for monitoring possible changes after any conditioning or testing.

Keywords: A. Glass fibers, A. Polymer-matrix composites (PMCs), B. Microstructures, D. Electron microscopy

1. Introduction

Over the past two decades, glass fiber reinforced polymer (GFRP) bars have been demonstrated to be a practical alternative for black, epoxy-coated and stainless steel rebars in reinforced concrete structures (RC) especially in applications where durability and corrosion resistance are required such as bridge decks and marine structures. In addition, the use of GFRP is suitable for buildings that include equipment sensitive to electromagnetic fields such as magnetic resonance imaging (MRI) units or bases for large motors [1].

GFRP consists of glass fibers as load carrying elements and resin which transfers and distributes the load among the fibers. Microstructural patterns of the fibers and resin play a crucial role in understanding the GFRP behavior. Different manufacturing parameters (such as pulling speed and dye temperature) lead to various GFRP microstructural patterns including the presence of voids and defects at various locations of GFRP cross-section. These patterns present combinations of continuous defects and disconnected voids and lead to a unique pattern for each GFRP bar. Understanding the GFRP microstructural pattern may allow predicting the GFRP behavior under different loading conditions and states of stress.

Considering the lack of GFRP microstructural knowledge, the main purpose of this study is to provide a documentation of GFRP microstructure and investigate its possible contribution on GFRP durability. It is recognized that this study was limited to small sample size, a single bar diameter and four GFRP manufacturers; however, its findings are significant and provide a clear path for investigations to follow.

In the present study, four commercially available GFRP bars with a nominal diameter of 12.7 mm (0.5 in.), equivalent to No. 4 steel rebar, were investigated. First, samples were properly polished and prepared for the microscopic examination. Next, scanning electron microscopy (SEM) was employed at different magnification levels to capture different aspects of the microstructural pattern including i) existing defects and voids in the matrix; ii) fiber-matrix interface; and, iii) fiber distribution in the matrix. Panorama images of the entire cross-sections were provided to give a proper comparison between different bars.

As part of this study, the possible effect of microstructural patterns on GFRP durability was investigated[2][3][4]. Two types of the bar that demonstrated the most different microstructural patterns were exposed to accelerated conditioning. The horizontal shear test was performed and the results were compared with the pristine bars. Finally, SEM imaging was performed on conditioned bars to provide additional evidence of the effect of the microstructural patterns on durability.

2. GFRP Samples

Four different GFRP barsall produced by pultrusion were selected in this study. Even Though, the production process is nominally the same, different process parameters and bar constituents including different surface characterestics have a profound effect on the result of the products.The selected GFRP bars were: Ribbed deformed surface (GFRP-A), fine sand coated with helically wrapped fibers (GFRP-B), double twisted fiber wrapped (GFRP-C) and coarse sand coated (GFRP-D). Table 1 provides the nominal and measured cross-sectional areas for all GFRP bar types. The nominal area is based on a circle with a nominal diameter of 12.7 mm (0.5 in.). The average measured area was computed based on a work by Claure and coworkers [5]following a standard test method for density and specific gravity (relative density) of plastics by displacement, ASTM D792[6].

Table 1: GFRP nominal and measured cross-sectional areas

Surface Texture	GFRP	Area (mm ²)		Diameter (mm)	
		Nominal	Measured	Nominal	Measured
Ribbed surface	A	126.5	130.5	12.7	12.6
Fine sand coated & helically wrapped fiber	B	126.5	137.8	12.7	12.7
Double twisted fiber wrapped	C	126.5	152.6	12.7	13.9
Coarse sand coated	D	126.5	162.6	12.7	14.7

3. Sample Preparation

Several GFRP coupons for each bar type were cut in an approximate thickness of 7 mm. The specimen surfaces were prepared by sanding using different grit levels (i.e., 180, 300, 600 and 1200) of sand paper and employing dedicated grinding and polishing equipment. Fine polishing completed the specimen preparation using a wet-polishing agent and polycrystalline diamond paste. Prior to imaging, specimens were placed in an oven at 60 °C for 24 hours to remove the moisture produced during polishing procedure. Samples were properly cleaned using an air-blower which also saved the SEM chamber from being contaminated and provided higher quality images. A visual inspection of all coupons using a magnifying lens confirmed that similar patterns existed and remained consistent for each bar type. Thus, only three samples for each GFRP bar were randomly selected for further examination by SEM. Since GFRP was a non-conductive material, an ion sputtering device was used to coat the samples with gold prior to SEM examination. A photo of representative samples prior to microscopic examination is shown in Figure 1.

Figure 1: Representative prepared samples (from left: GFRP-A, B, C, and D)

4. SEM Imaging

SEM was conducted at low (0-200x), medium (200-1000x) and high (>1000x) magnification levels. Low magnification images provided a general pattern of the cross-section by covering a higher area in each image. This level was used to measure the length of the existing defects. The images captured in this level were stitched together to prepare the panorama image of the entire sections. Medium level magnification images provided a more detailed view of the section and eventually high-level magnification imaging was mainly conducted to monitor the single glass fibers. Three samples for each GFRP bar were studied under SEM and since similar results were observed for the three, only representative images of one of these samples for each GFRP bar is shown in this paper.

5. Results and Discussions

GFRP-A presented a continuous defect at the edge (Figure 2). A large void with the length of 1.4 mm was observed at the center of the bar (Figure 3). The rest of cross-section did not show any voids or defects and the resin properly impregnated the fibers (Figure 4).

Figure 2:A defect at the edge of GFRP-A at magnification levels of 80x (left) and 150x (right)

Figure 3:Detailed view of a void in the center of GFRP-A at magnification levels of 55x (left) and 140x (right)

Figure 4: Area with no void of GFRP-A at magnification levels of 350x (left) and 1200x (right)

GFRP-B demonstrated a continuous defect along the edge as shown at different magnification levels in Figure 5. These defects were not the result of sample preparation since similar patterns were observed in multiple samples of GFRP-B. Disconnected voids were observed at the entire cross-section. The void numbers increased at the vicinity of the edges compared to the central portion of the section (Figure 6).

Figure 5: A continuous defect at the edge of GFRP-B at magnification levels of 40x (left) and 110x (right)

Figure 6: Distributed voids in the cross-section of GFRP-B: close to the edge (left), close to the center (right)

GFRP-C presented both connected defects and disconnected voids in the cross-section including the central portion and edges while no defect was observed at the surface of the bar (Figure 7-9). The continuous defect pattern across the entire section may be problematic for durability applications. This is especially true for the defects close to the edge, since deterioration of the reinforcement due to diffusion of chemical and environmental attacks initiates at the boundaries.

:Figure 7: Continuous defects at the edge (left) and at the center (right) of GFRP-C

Figure 8: Detailed view of a defect close to the edge of GFRP-C at magnification levels of 80x (left) and 230x (right)

Figure 9: High magnification image of a void (left) and area with no void (right) of GFRP-C

GFRP-D demonstrated disconnected void patterns in the cross-section including both at the central portion and edges (Figure 10). The number of voids reduced in the center compared to the edges. Figure 11 provides higher magnification images of the voids and the no void area.

Figure 10: Disconnected void patterns at the edge (left) and center (right) of GFRP-D

Figure 11: Higher magnification image of the areas with voids (left) and area with no void (right) of GFRP-D

Finally, Figure 12 provides a panorama image of the entire section for all the GFRP bars. It serves for a proper comparison purpose and demonstrates that each bar had a unique microstructural pattern. The observed defects in all GFRP bars were continuous along the length of the bar providing an evidence that the defects are most likely related to the pultrusion and curing process. The defects can be reduced if not entirely avoided by implementing quality control and using proper manufacturing parameters such as pultrusion speed, dye temperature and curing time. It is also possible that some of the secondary constituents such as fillers and additives may improve the quality of the final product.

Figure 12: Panorama images of GFRP-A (a), GFRP-B (b), GFRP-C (c), and GFRP-D (d)

6. Alkaline Conditioning

GFRP-A and GFRP-C were selected among the four types and exposed to an alkaline solution with the pH of 12.5-13 for a duration of 1000 hours. The alkaline solution was prepared following the ACI-440.3R by mixing 118.5 gr of Ca(OH)_2 , 0.9 gr of NaOH and 4.2 gr of KOH in one liter of distilled water[7]. A temperature of 60 °C was chosen to accelerate the exposure effect[4]. Five samples for each bar with the length of 0.5 m were placed in the environmental chamber. The GFRP ends were sealed with epoxy to reduce the effect of moisture absorption through the ends. Next, the horizontal shear strength was

determined following the ASTM D4475, since this test method provides a good assessment of the mechanical properties at fiber-resin interface and is a proper indication of durability and quality control. Five conditioned coupons with the length of 50.8 mm were tested for each bar type. The same number of coupons was taken from pristine bars and tested as benchmark. GFRP durability in alkaline environment was not the main focus of the study and thus a limited number of coupons was tested. The conditioning of the bars and the following horizontal shear tests were performed to simply provide evidence on the possible effect of microstructural pattern on GFRP durability. Test span was set as three times of bar diameter following the standard [8]. The load was applied in displacement control mode at a rate of 1.27 mm/min. The test fixture is shown in Figure 13.

Figure 13: Horizontal shear test fixture

Table 2 reports the average results, where S_p refers to the horizontal shear strength of the pristine, and S_c refers to horizontal shear strength of the conditioned sample. The same notation is used for the failure load (P). Based on the results, after 1000 hours of exposure to alkaline solution at 60 °C, the horizontal shear strength of GFRP-A decreased only by 1% compared to the pristine values while GFRP-C demonstrated 15% reduction in horizontal shear strength.

Table 2: Result of the horizontal shear tests on pristine and conditioned GFRP samples

Type	P_p			P_e			S_p (MPa)	S_e (MPa)	Ratio (S_e/S_p)
	No. of Samples	Average (kN)	CoV (%)	No. of Samples	Average (kN)	CoV (%)			
GFRP-A	5	12.19	12.2	5	12.07	4.0	64.16	63.55	0.99
GFRP-C	5	10.45	9.2	5	8.91	3.5	54.93	46.90	0.85

Note: 1 kN= 0.2248 kip; 1 psi=0.0069 MPa

The results obtained from the horizontal shear test was consistent with the expectations from the SEM imaging. The entire cross-section of GFRP-C was covered with continuous defects and disconnected voids and they were expected to dramatically affect its durability. GFRP-A was characterized with no voids and defect patterns and demonstrated a proper resistance to alkaline conditioning. Additional evidence was provided by performing SEM imaging on conditioned samples to investigate the change in microstructure at the vicinity of the existing defect patterns. Figure 14 shows the conditioned and intact regions and the alkaline surface penetration in GFRP-A. The microstructure of GFRP-A did not include any defect or void pattern, as a result, no localized damage was observed as the alkaline front penetrated from the edge toward the core of the cross-section.

Figure 14: Conditioned and intact regions in GFRP-A

Conversely, SEM images of GFRP-Ccross-section presented extensive damage in the vicinity of the existing defects. Figure 15 shows a region of the alkaline conditioned GFRP-Cpresenting the damage in the resin and glass fibers. Additionally, Figure 16shows excessive fiber degradationin the vicinity of an existing defect. The damaged area was not clearly separated by a front line from the intact region confirming one more time the higher vulnerability of this bar type.

Figure 15: A conditioned region of GFRP-C presenting an extensive damage in resin and glass fibers

Figure 16: A conditioned region of GFRP-C presenting an extensive damage in the fibers in the vicinity of existing defects

7. Conclusion

SEM imaging was performed at different magnification levels on four different commercially available GFRP bars produced by pultrusion. Only one diameter size was investigated. Each bar presented unique microstructural patterns due to different manufacturing parameters and possibly constituent materials, including fillers and additives, used by the manufacturer. The effects of these patterns on GFRP durability was investigated by accelerated conditioning in alkaline solution of two of the bars. Horizontal shear tests were performed as an indicator of durability. SEM imaging was conducted on the conditioned samples to provide additional data. According to the results from the SEM imaging and mechanical testing, the following observations are made:

- Each GFRP bar demonstrated a unique signature of existing defect and void patterns characterized by a combination of disconnected voids and continuous defects at different locations in the cross-section most likely due to different manufacturing parameters such as pulling speed, dye temperature and curing time and constituent materials.
- GFRP-A presented the lowest existing void/defect compared to other GFRP bars. There was a single void in the center of the bar and a macro defect at the edge. GFRP-B demonstrated both disconnected voids mostly at the vicinity of the edges as well as the defects along the edges of the bar. GFRP-C presented the most voids and defects in the cross-section compared to other GFRP bars. It mostly included continuous defects over the entire area. GFRP-D presented disconnected voids all over the cross-section while there were fewer voids at the center of the bar compared to the edges.
- All the observed defects were continuous along the length of the bar confirming that the defects are the results of manufacturing. These defects can possibly be reduced by implementing better quality control practices and using proper manufacturing parameters.

- After 1000 hours of exposure to accelerated conditioning in a high pH alkaline environment at 60 °C, GFRP-A did not experience any change in its horizontal shear strength (less than one 1%) while GFRP-C demonstrated 15% loss of this mechanical property.
- Different penetration of alkaline front was observed in GFRP bars with different microstructural patterns. Additionally, extensive deterioration was noticed at the vicinity of existing defects and voids.
- GFRP microstructural pattern plays a critical role in mechanical properties and durability of GFRP bars.

The results of this study can be used as a benchmark for microstructure analysis of commercially available pristine GFRP bars and serve as a base for monitoring possible changes after conditioning or field use.

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272 **9. References**

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