

## COMPARISON BETWEEN THE CORROSION MECHANISM AND CRACK FORMATION OF BASALT AND GLASS FIBERS IN AGGRESSIVE MEDIA

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### Abstract

*In this study, strength behavior and crack formation mechanism of E-glass and basalt fibers exposed to sulfuric acid environment for different immersion times were investigated. In order to study the strength degradation, fibers were immersed in sulfuric acid and Strength reduction of fibers versus immersion times was studied. The corrosion mechanism of fibers exposed to acid, were examined by quantitative XRF (X-Ray Fluorescence) method. Moreover, intact and degraded fibers were examined by SEM and EDX (Energy Dispersive x-ray Microanalysis) methods to show the relation between the reaction of fiber elements and acid environment. The results showed the higher corrosion resistance of basalt in comparison with E-glass fiber.*

### 1 Introduction

Polymer composites are widely used in chemical and petroleum industries. Brittle fracture resulting from corrosion is very current phenomenon of these materials. Acidic media degrades fiber strength and therefore lowers fracture resistance of composites which lead them to failure in very lower mechanical stresses than their ultimate strength. Gratings and tubes used in the chemical and pipeline industries [1] and high voltage composite insulators made by polymer composites [2] are examples of components which are degraded in the corrosive environments. The importance of this issue, caused many investigations were carried out on the corrosion of polymer composites. Due to the degradation and failure of fiber of reinforced polymer composites exposed to corrosive media, many investigations have been devoted to explain the mechanism of fiber corrosion [3-6]. In most of research, strength degradation of composite has been evaluated using the mechanical or Acoustic emission test [7] and since yet, crack formation mechanism of fibers had not been introduced.

In this study, strength behavior and crack formation mechanism of E-glass and basalt fibers exposed to sulfuric acid environment were investigated. In order to study the strength degradation, fibers were immersed in sulfuric acid and Strength reduction of fibers versus immersion times was studied. The corrosion mechanism of fibers exposed to acid, were examined by quantitative XRF (X-Ray Fluorescence) method. Moreover, intact and degraded fibers were examined by SEM (Scanning Electron Microscope) and EDX (Energy Dispersive

x-ray Microanalysis) methods to show the relation between the reaction of fiber elements and acid environment. The ion-depletion-depth model was used to study the fracture process. In order to study the crack formation mechanism, the EDX results were used to investigate the effects of ion depletion on the crack initiation and the role of Al, Ca and Fe ions in creation of surface cracks on fibers for different immersion times.

## 2 Experiments procedure

The fiber bundles were kept in 5% sulfuric acid with initial pH of 0.33. Thereafter, the fiber bundles were removed from the solution and rinsed with de-ionized water and finally they were dried in the air. At this stage, initial intact fibers and degraded fibers were examined by yarn tensile tests in order to study the change in tensile strength. In order to study the corrosion mechanism and crack formation of fibers, some intact and degraded fiber bundles were examined by quantitative XRF based on the ASTM C982-97 standard. After that, some intact and degraded fiber bundles were examined by SEM and EDX Analyses to investigate the chemical mechanism of element changes in glass fiber and crack formation of degraded fibers.

## 3 Results and discussion

### 3.1 Strength degradation behavior

The mechanical properties of fibers are given in Figure 1 based on the tensile test results on seven specimens. The strength of degraded fibers was normalized relative to properties of intact fibers which are 2158 MPa for E-glass and 2280 MPa for basalt fiber. It can be seen that after 500 h of immersion time, the strength of fiber reduce 51%. Compared to the strength behavior of E- glass fiber in the same media for 500 h, the strength of glass fiber reduces 62%. It can be concluded that the strength degradation of E-glass fiber is more severe than that of basalt fiber.

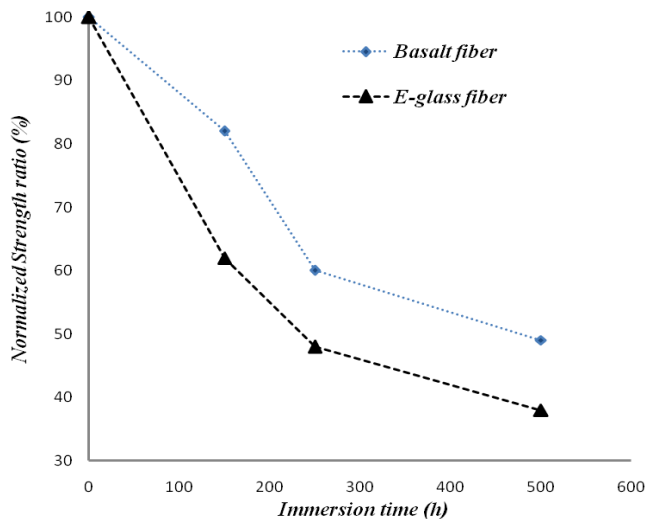


Figure 1. Tensile strength of E-glass and basalt fibers vs. immersion time

### 3.2 Corrosion mechanisms

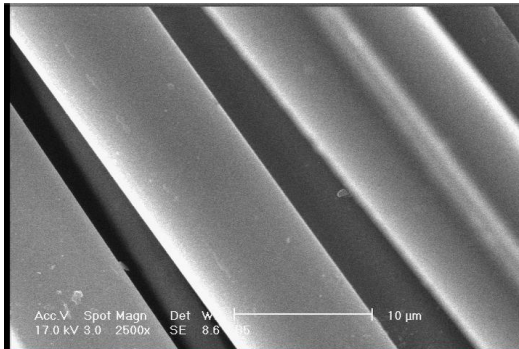
In order to study the corrosion mechanism of intact and degraded fibers exposed to acidic media within 200 h, quantitative XRF analysis was then carried out. The tests were performed by PW 2404 apparatus made by Philips Company. Main chemical compositions of intact and degraded fibers are shown in Table 1 and 2. The results indicated that the  $\text{Ca}^{2+}$ ,  $\text{Al}^{3+}$  and  $\text{Fe}^{3+}$  ions were the main non-siliceous ions involving in the corrosion of basalt fiber and the leaching of  $\text{Ca}^{2+}$  and  $\text{Al}^{3+}$  were the main cause of E-glass corrosion. In this stage, the EDX analysis of fiber surface was the best way to investigate the corrosion behavior of the fibers. The SEM images of intact fibers before exposure to acid are illustrated in Fig. 2. In this case, there is no crack and deposit on the basalt fiber surface. The EDX results of the intact fiber surfaces (Fig.2) are given in Table 3.

Component percentage	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	Br	Sr	SO <sub>3</sub>	L.O.I*
Intact glass fiber (%)	0.77	3.26	12.2	58.9	18.6	0.26	1.17	0.08	0.00	4.70
Degraded fiber (%)	0.57	2.66	9.51	46.3	14.4	1.01	1.00	0.05	3.64	20.8

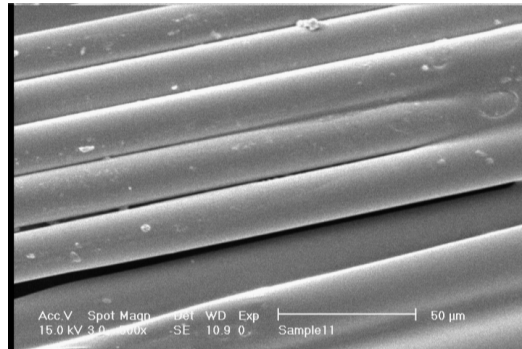
Table 1. Quantitative XRF results of E-glass fiber (\* L.O.I means loss on ignition)

Component percentage	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	TiO <sub>2</sub>
Intact fiber (wt %)	1.80	4.90	16.40	49.10	8.40	14.80	1.20	3.40
Degraded basalt fiber (wt %)	1.60	4.50	14.20	55.80	6.80	12.60	1.40	3.10

Table 2. Quantitative XRF results of basalt fiber



(2.1)



(2.2)

Figure 2. SEM image of intact basalt (Figure 2.1) and glass (Figure 2.2) fiber

Related Fig. number	Element content (wt%)							
	Na	Mg	Al	Si	K	Ca	Fe	Ti
2.1	2.25	3.28	12.81	51.63	3.43	9.05	15.23	2.32
2.2	0.0	2.28	12.66	54.10	1.07	34.95	2.13	0.0

Table 3. The EDX results of the intact fiber surface (Element with the weight percent less than 1% are missed)

The SEM images of glass fiber specimens exposed to sulfuric acid for 96 h showed that after 96 h of fiber immersion in sulfuric acid, there was no crack in glass fibers; however, the deposit was slowly generated on the fiber surface. The EDX results showed that the content of Fe ions remarkably increased and that of Ca reduced over the time. By increasing the immersion time, irregular cracks appeared on the glass fibers surface. The SEM images of glass fibers exposed to acid for 192 h are illustrated in Figure 3. The Figure clearly indicates the irregular cracks on the glass fiber surface.

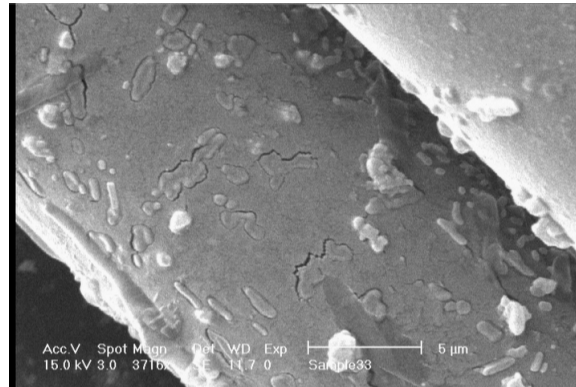


Figure3. SEM images of glass fibers immersed for 192 h

The EDX result of point around the micro-cracks on figure 3 is shown on table 4. The results showed that in the E-glass fiber, the leaching of Fe<sup>3+</sup> ions caused micro-cracks generation around some areas of fiber surface with higher contents of Fe<sup>3+</sup> ions.

Related Fig. Number	Element content (wt%)					
	Mg	Al	Si	K	Ca	Fe
3	2.97	11.64	56.3	0.54	22.25	6.32

Table3. The EDX results of point around the micro-cracks on figure 3

In the basalt fiber after 192 h of immersion time, Ca<sup>2+</sup> and Fe<sup>3+</sup> ions were leached out from the fiber surface. Some amounts of these ions were entered in the solution and some of them were deposited on the basalt fiber surface. However, there is no crack generated on fiber surface in this time. By increasing the immersion time the high amount of Fe<sup>3+</sup> deposits were generated on the fiber surface. It indicated that Fe<sup>3+</sup> ions were leached out from the interior layers of basalt fiber to its exterior layers. By increasing the immersion time to 1000 h, the SEM observations showed the various micro-cracks generated on the longitudinal direction on the glass fiber (figure 4). In this time catastrophic failure is seen on some glass fiber as shown on figure 5. However, the surface of basalt fiber is covered by dominant spiral cracks (figure6).

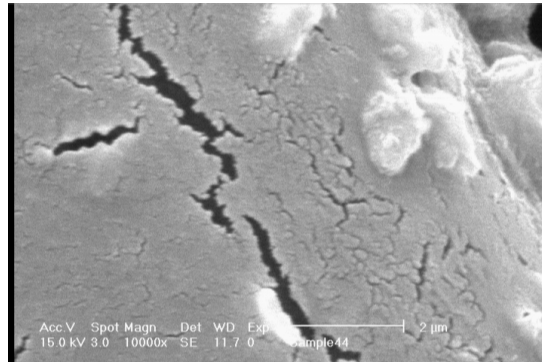


Figure4. Micro-cracks generated on the glass fiber surface after 1000 h exposure time

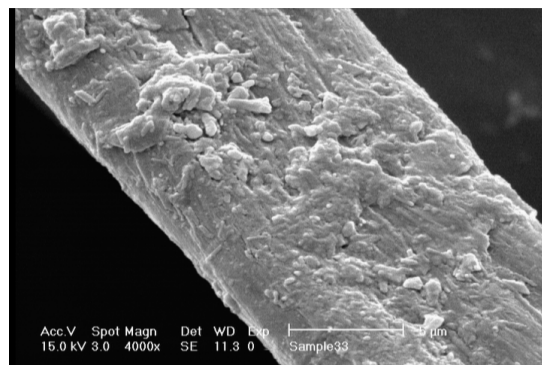


Figure5. Damaged glass fiber after 1000 h exposure time

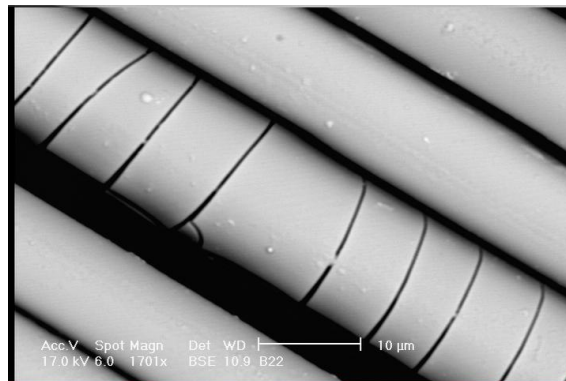


Figure6. Spiral cracks on basalt fiber after 1000 h exposure time

#### 4 Conclusions

1. The strength degradation of E-glass fiber is more severe than that of basalt fiber in sulfuric acid environment.
2. The results indicated that the  $\text{Ca}^{2+}$ ,  $\text{Al}^{3+}$  and  $\text{Fe}^{3+}$  ions were the main non-siliceous ions involving in the corrosion of basalt fiber and the leaching of  $\text{Ca}^{2+}$  and  $\text{Al}^{3+}$  were the main cause of E-glass corrosion.
3. In the basalt fiber after 192 h of immersion time,  $\text{Ca}^{2+}$  and  $\text{Fe}^{3+}$  ions were leached out from the fiber surface. Some amounts of these ions were entered in the solution and

some of them were deposited on the basalt fiber surface. However, there is no crack generated on fiber surface in this time.

4. After 1000 h of immersion time, the SEM observations showed the various micro-cracks generated on the longitudinal direction on the glass fiber.

### **References**

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