Fractographic Analysis of the Effects of Combined Natural Weathering and Seawater on the Performance of GFRE Pipes

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Abstract

The use of glass fiber reinforced Epoxy (GFRE) pipes for seawater and crude oil handling and transportation in Saudi Arabia is hindered by the lack of reliable engineering data on the durability of these pipes when exposed to harsh Arabian Gulf outdoor environment.

This paper presents a study of the effects of natural outdoor weather and seawater on tensile and creep (stress rupture) behavior of GFRE materials. Exposure of seawater filled pipes to Saudi outdoor environment for periods ranging from 6 to 12 months has resulted in a drastic reduction of tensile and stress rupture strengths of the materials. Fractographic analysis of unexposed specimens showed a combination of mode I and mode II failure characterized by inclined hackles as well as large amount of fiber breakage. High temperature and moisture absorption decreased fiber-matrix interface strength and caused degradation at the fiber level resulting in lower strength and stiffness of the GFRE.

Key Words: Seawater, Tensile, Creep, Fractography, Hackles, Mode I, Mode II.

1. Introduction

Because of their high strength to weight ratio glass fiber reinforced epoxy (GFRE) pipes are now replacing their metallic counter parts for crude oil and seawater transportation. The major problem associated with the full fledge application of these composite pipes is the lack of reliable engineering data regarding their long term durability after exposure to various natural environmental conditions, such as change in temperature, humidity, moisture, alkaline solutions, crude oil and acids.

Most of the research work on GFRE involved experimental studies on the behavior of the composite after conditioning to seawater, natural outdoor weather and distilled water at various temperatures by tensile and creep testing. Sookay et al [1] investigated the durability of glass fiber/epoxy laminates after conditioning to wet and humid climate. The compressive strength was seen to reduce with increase in exposure period. Abdel-Magid et al [2] investigated the tensile and flexural creep behavior of E-glass epoxy composites at room temperature (RT) and 50^{o}C respectively. Both the tensile and creep properties decreased with temperature increase. The SEM analysis of the broken samples revealed that the shear properties of the matrix and the interfacial bond strength between fiber and matrix are important for the creep or stress-rupture strength. Ellyin and Rohrbacher [3] investigated the environmental durability of E-glass/Epoxy composite laminates after conditioning in distilled water at ambient and higher temperatures by conducting tensile tests. The test results indicated that the strength and ductility of the composite material decreased as a result of immersion in water at higher temperatures. Kontou et al [4] observed that the creep strain rate of unidirectional E-glass/Epoxy composite specimens was negligible at room temperature and considerable at higher temperatures. Ellyin [5] found that cycles of dry and wet conditions at high temperatures were the most damaging environment for E-glass/Epoxy laminate specimens. Raghavendra et al [6] found that tensile, compressive and inter-laminar shear properties of E-glass/Epoxy composite specimens showed a reduction in values at hot-wet conditions when compared with its values at RT. The degradation of these composites in the above mentioned conditions was mainly attributed to fiber matrix de-bonding and matrix cracking.

In the present study the effects of natural outdoor weather and seawater on tensile and stress rupture behavior of GFRE pipes is evaluated for exposure periods of 6 and 12 months. Fractographic analysis of the tested specimen is performed using optical microscope followed by scanning election microscope (SEM) to investigate the controlling degradative phenomenon.

2. Materials and Testing Procedures

The composite under study is E-glass/Epoxy (GFRE) composite pipes manufactured from filament winding process and having dimensions of 150 mm inner diameter, 6 mm thickness and 10 m in length. It has an 8 plies structure with the fibers wounded at an angle of ±54.5°; this winding angle exhibits optimum mechanical properties of circumferential (hoop) and longitudinal (axial) strength for the filament wound pipes. The GFRE pipes were machined to lengths of about 1.6 m and filled with Arabian Gulf seawater and after sealing with CPVC end caps, were exposed to natural Dhahran weather with the axis of the pipe parallel to the east-west direction to allow maximum exposure to sunlight.

After exposure for desired periods, ring specimens (Fig. 1) were machined from the pipe section to a width of 22.86 ± 0.254 mm and single side double edge notches were cut in order to obtain the reduced cross section area having a gage width of 13.97 ± 0.127 mm. The notches were machined in the exposed area of the pipe. All the dimensions are in accordance with ASTM D-2290 standard for thermosetting pipes [7].

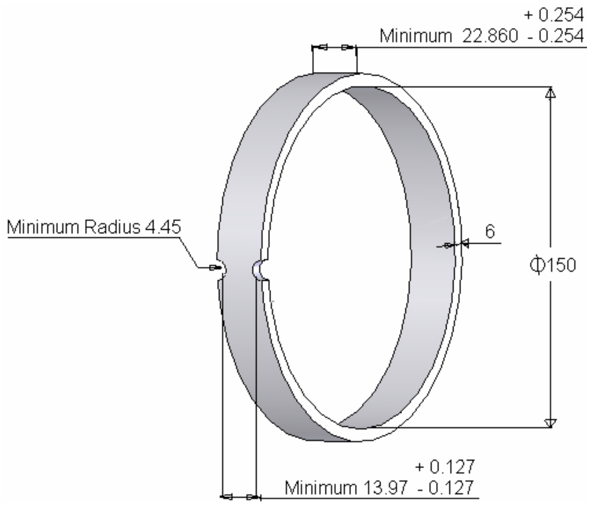


Fig. 1. Test Ring specimen [ASTM D-2290] (All Dimensions in mm).

The GFRE ring specimens were held on the tensile and creep test machines for testing using a split disk fixture which is in accordance with ASTM D-2290 standard. The hoop's tensile test was performed on an Instron 8801 machine having a capacity of 100 KN and equipped with a hydraulically actuated self aligning and griping system. All tensile tests were carried out in position increments with a cross head speed maintained at 3 mm/min. The stress rupture tests were carried out on two types of test machines namely; ATS lever arm creep tester and homemade creep/stress rupture tester. The ATS lever arm creep tester machine has a maximum load bearing capacity of 40 KN with a maximum mechanical advantage of 20:1. This ratio of mechanical advantage can be varied depending upon requirement. Whereas the homemade stress rupture testing machines were designed for a mechanical advantage of 10:1. The conditioned specimens were loaded at stress levels ranging from 60% to 90% of ultimate tensile strength (UTS) of as received (baseline) material. Tensile and creep tests results of as received GFRE samples were taken as reference.

Fractographic analysis is performed by optical microscope followed by scanning electron microscope (SEM).

3. Results and Discussion

3.1 Tensile Test Results

The average tensile strength of six as received GFRE ring specimens was found to be 327.23 MPa with a standard deviation of 20.53 MPa. The natural outdoor weathering of seawater filled GFRE pipes resulted in reduction of tensile stress, strain and stiffness for both 6 and 12 months. The average tensile stress, strain and stiffness decreased by 9%, 28 % and 3.6%, respectively after 6 months of exposure. Further degradation of these properties is observed after 12 months of exposure reaching 12% for stress, 32.7% for strain and 5% for stiffness.

The reduction in tensile properties of GFRE pipes is due to the effects of UV, temperature and humidity on outer surface and seawater and temperature on the inner surface. Plasticization of the composite was seen in minor amount indicated by the reduced stiffness values with increasing exposure period. This would have been due to continuous contact with seawater and humidity of outdoor Dhahran weather. It should be mentioned that the humidity in Dhahran can reach 100% with temperatures up to 50°C. A similar trend of decrease in tensile strength was also observed by Ellyin and Rohrbacher [3] for E-glass/Epoxy composites after conditioning to seawater environment.

3.2. Stress Rupture Tests

From stress-rupture test results obtained in this study it could be inferred that when a GFRE sample is loaded at stress levels of 70–90% of ultimate strength, the tertiary creep stage is reached and fracture was seen to occur within a few hours of loading. However, the GFRE material is capable of sustaining 60% of its ultimate strength without fracture for more than 10,000 hours at room temperature. The strength of as received GFRE pipe samples extrapolated to 50 years was found to be 155 MPa. This strength retention is much higher than the expected design strength of about 100 MPa.

GFRE pipes filled with sea water and exposed to natural weathering environment for 6 and 12 months revealed drastic reduction in the average specimen life. Time to rupture at 70% of the ultimate strength decreased from an average of 35.5 hours for virgin material to 2.7 and 1.4 hours after 6 and 12 months exposure, respectively. This reduction may have been caused by the combined effects of aqueous environment and high temperature. Similar results of decrease in strength retention were also reported by Yao and Ziegmann [8] after stress rupture testing of GFRE specimens conditioned to water.

4. Fractographic Analysis

4.1 Fractographic Analysis of Tensile Tested Samples

Fractographs of the tensile tested GFRE baseline sample can be seen in figures 2 to 4. Optical observation of the fractured specimen revealed that the fracture initiated at the inner surface of the ring specimen. This is due to the high hoop stresses developed at the internal surface. The fracture appears to initiate from the notch and propagate in a zigzag manner throughout the width of the sample. The cup and cone fractured zone in Fig 2 indicated different fracture mechanisms at the lateral edges and in the middle [9]. The fracture appeared to have initiated (Fig. 2) mainly due to fiber breakage (C) for the first 2-3 plies. This was followed by laminate splitting (B) and interficial debonding (D) till the fracture reached the outer surface. The outer surface of the specimen displayed broom type failure that including matrix shear excessive longitudinal laminate splitting and interficial debonding and fiber breakage on the outer 2 plies at the end of failure. Figure 8 is an SEM view of area C of Fig. 2. It clearly shows broken fibers adhered with resin matrix indicating good interficial bonding. The presence of more amounts of broken fibers indicates application of higher loads prior to fracture [9]. The SEM of the outer surface of the specimen (Fig. 4) revealed the presence of a large number of blow holes. These might have resulted from volatile gases trapped during the fabrication of composites [10]. The inclined hackles on the matrix are an indication that failure happened as a result of a combination of Mode I and Mode II fracture.

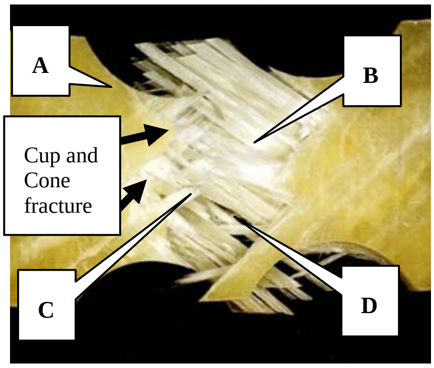


Figure 2. Optical picture of inner portion of the tensile tested virgin GFRE specimen.

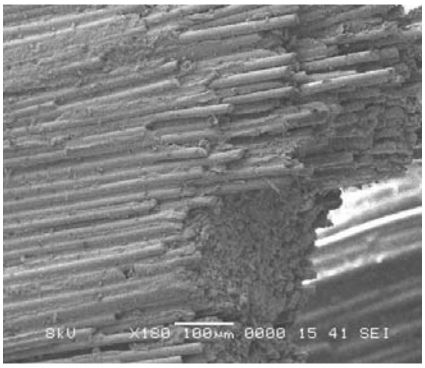


Figure 3: Broken fibers embedded in resin matrix at position C (Fig 2).

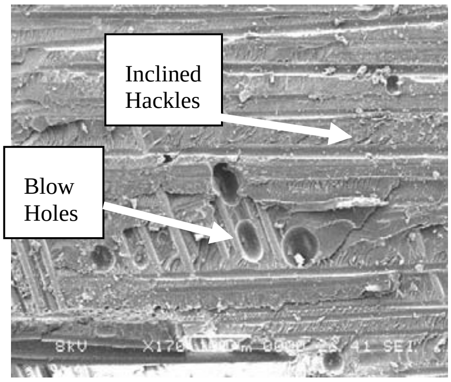


Figure 4. SEM view showing blow holes and inclined hackles

Figure 5 is an optical view of inner gage portion of the GFRE specimen conditioned to combined natural outdoor weather and seawater for 12 months. The fracture initiation from the inner surface and propagation was seen to be virtually identical to the failed GFRE baseline sample with the exception that more amount of fiber breakage and matrix shear along with less laminate splitting. The SEM picture (Fig. 6) taken at position C of Fig. 5 revealed neat broken fibers with minimum matrix adherence indicating weak interficial bond strength. Since the inner portion of specimen was exposed to seawater, it is possible that the water molecules in seawater might have ingressed through the epoxy resin due to the presence of organic groups in the backbone of the epoxy resin [2]. The water molecules in contact with the glass fiber could have caused leaching out of water soluble alkaline components such as Na^+ and other elements present in glass fiber resulting in fiber level degradation [3].

Figure 7 is the SEM view at the inner portion of the specimen (Fig 5-D) indicating groove marks on matrix along with inclined hackles, blow holes and transverse cracks. The presence of blow holes increases the porosity of epoxy matrix resulting in increased water ingress into the sample till the fiber level. The groove marks on the matrix are the result of pulled out fibers indicating weak interficial bond strength after conditioning to seawater as a consequence of fluid attack on the fiber matrix interface. Again, the presence of inclined hackles indicates shearing of matrix and failure due to mixed mode.

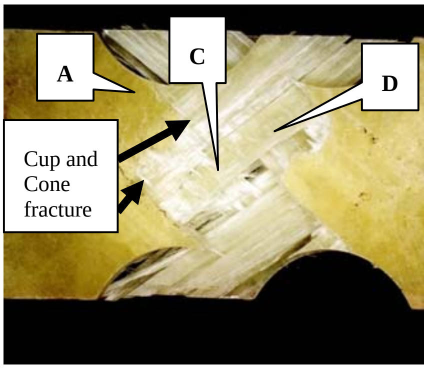


Figure 5. Optical picture of inner portion of the tensile tested weathered GFRE specimen.

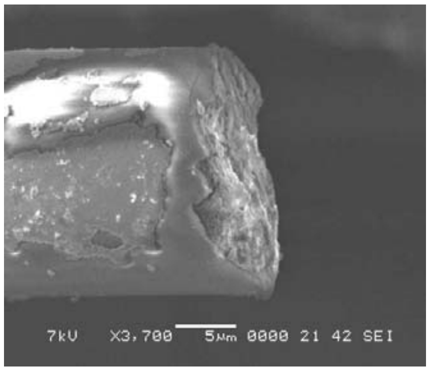


Figure 6: Fiber level damage at position C in Fig 5.

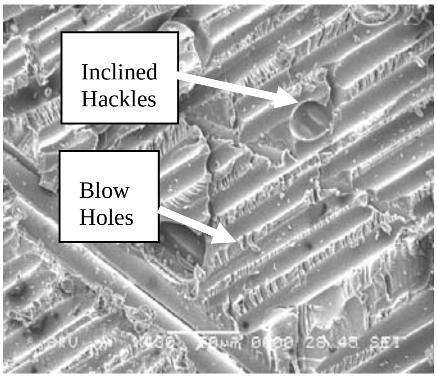


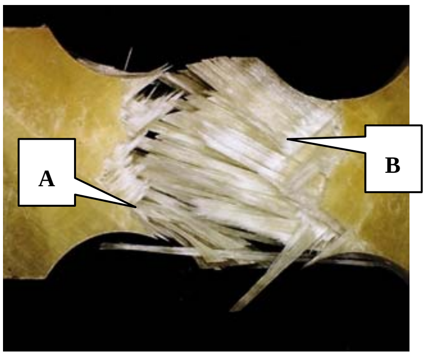
Figure 7. SEM view showing blow holes and inclined hackles in area D (Fig. 5).

4.2. Fractographic Analysis of Creep Tested Samples

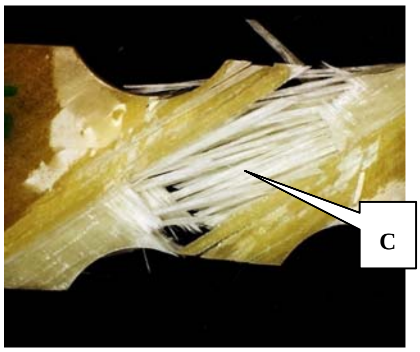
The average time to rupture at 70% of UTS for four GFRE baseline samples was found to be 35.5 hours. The fracture initiation and propagation mechanisms from inner surface (Fig 8 (a)) to outer surface (Fig 8 (b)) of GFRE baseline sample was found to be similar to that of the tensile tested GFRE baseline sample, in addition to more amount of fiber breakage.

Figure 9 is the SEM view taken at position A on the inner surface revealing broken fibers partially buried in resin matrix. Thus, indicating good interfacial bond strength as noted before in the tensile tested GFRE baseline sample. The presence of blow holes in large number could be seen on the inner surface of specimen (Fig 8-B) as shown in Figure 10. The inclined hackles towards the fiber pullout direction depicts failure would have occurred due to combination of matrix shearing and also due to combination of mode I and mode II fracture mechanisms. The fracture propagation from inner to outer portion was seen to be mainly due to laminate splitting (Fig 8 (b)-C).

Figures 11 (a) and 11 (b) show optical views of inner and outer surfaces of stress-rupture tested GFRE sample after exposure to combined natural outdoor and seawater conditions for 12 months. The fracture initiation and propagation mechanisms are similar to those of the tensile tested samples. Figure 12 reveals the presence of blow holes on the inner surface of specimen (Fig 11 (a)-B), hence increasing the porosity of epoxy matrix and resulting in increased water ingress into the sample till the fiber level. The presence of hackles on the matrix would have resulted due to mixed mode failure. The outer surface of specimen (Fig 12 (b)-C) indicated that failure was mainly due to interficial debonding. Neat fibers embedded in resin matrix could be seen in Figure 13 indicating reduced interficial bond strength due to prolonged exposure to outdoor environment for 12 months.

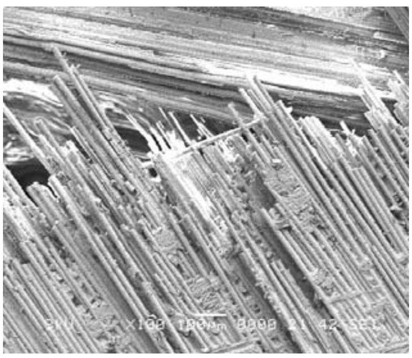


(a) Inner portion of pipe



(b) Outer portion of pipe

Figure 8. Optical pictures of the creep tested GFRE baseline sample (1X).



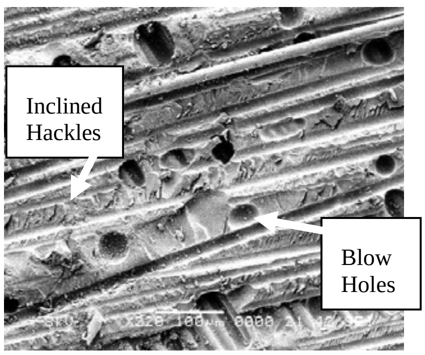


Figure 9. Broken fibers buried in matrix at position A in Fig 8 (a)

Figure 10: Blow holes and inclined hackles at position B in Fig 8 (a).

(a) Inner portion of pipe

(b) Outer portion of pipe

Figure 11. Optical pictures of the creep tested GFRE samples exposed to combined natural weather and seawater for 12 months (1X).

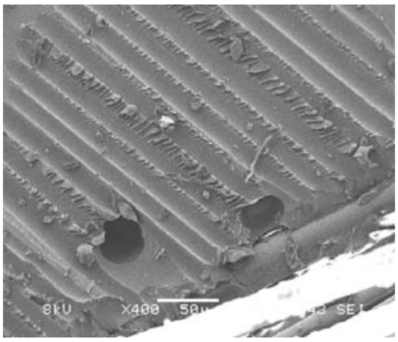


Figure 12. Blow holes along with inclined hackles at position B in Fig. 11(a).

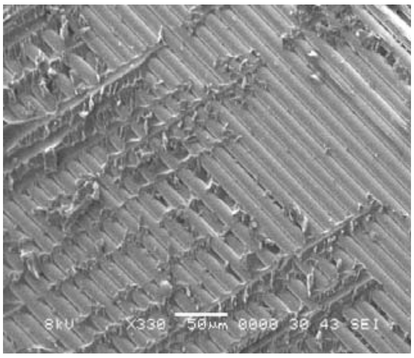


Figure 13. Neat fibers deviod of resin at position C in Fig.11(b)

5. CONCLUSIONS

Analysis of results of tensile and creep tests on GFRE pipe sections conditioned to combined natural outdoor and seawater environments led to the following conclusions:

i. The combined natural outdoor weathering and seawater exposure of GFRE specimens resulted in drastic reduction in the average tensile strength and fracture strain after 6 and 12 months of exposure. This decrease in strength can be attributed to the continuous contact of the epoxy matrix with sun heated seawater, which by nature is more susceptible to water and hence causing damage till fiber level.

ii. The GFRE pipe specimens filled with seawater and exposed to natural outdoor weather revealed drastic reduction in average time to rupture after 6 and 12 months of exposure. This reduction in strength is mainly due to epoxy matrix susceptibility to water.

iii. The fractographic analysis of the tensile and stress rupture tested GFRE specimens revealed cup and cone type of fracture. The optical pictures showed that failure was mainly due to fiber breakage and laminate splitting. The SEM analysis also confirmed the existence of broken fibers with good amount of adhered matrix indicating good interficial bonding. The presence of blow holes has probably helped moisture ingress in the material.

iv. The strength retention of as received GFRE pipe samples after 50 years was found to be 155 MPa. This strength retention is much higher than the expected average design strength of 100 MPa.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

1. Sookay N.K., Klemperer C.J. von, Verijenko V.E., "Environmental testing of advanced epoxy composites", *Composite Structures*, Vol. 62, pp. 429-433, 2003.

2. Abdel-Magid Beckry, Lopez-Anido Roberto, Smith Glenn, Trofka Sara, "Flexure creep properties of E-glass reinforced polymers", *Composite structures*, Vol. 62, pp. 247-253, 2003.

3. Ellyin F. and Rohrbacher C., "Effect of Aqueous Environment and Temperature on Glass-Fibre Epoxy Resin Composites", *Journal of reinforced plastics and composites*, Vol. 19, No. 17, pp. 1405-1427, 2000.

4. Kontou. E, "Tensile Creep Behavior of Unidirectional Glass-Fiber Polymer Composites", *Polymer Composites*, pp. 287-292, 2005.

5. Ellyin Fernand, "Durability of Glass-Fiber Reinforced Polymer Composites in Aqueous and High Temperature Environments", *Polymers & Polymer Composites*, Vol. 12, No. 4, pp. 277-288, 2004.

6. Raghavendra M, Manjunatha C, Jeeva Peter M, Venugopal C.V, Rangavittal H. K, "Effect of Moisture on the Mechanical properties of GFRP Composite Fabric Material", *International Symposium of Research Students on Material Science and Engineering*, India, 2004.

7. ASTM D 2290, “Standard Test Method for Apparent Hoop Tensile Strength of Plastic or Reinforced Plastic Pipe by Split Disk Method”, *Annual Book of ASTM Standards*, Vol. 08, No 01, 04, June 10 2000.

8. Yao. J, and Ziegmann. G, “Equivalence of moisture and temperature in accelerated test method and its application in prediction of long-term properties of glass-fiber reinforced epoxy pipe specimen”, *Polymer Testing*, Vol. 25, pp. 149–157, 2006.

9. Ginty C. A., Chamis C. C., “Fracture characteristics of angle-plied laminates fabricated from overaged graphite/epoxy prepeg”, Fractography of modern engineering materials: composites and metals, ASTM STP 948, J. E. Masters and J. J. Au, Eds., *American society for testing and materials*, Philadelphia, pp. 101-130, 1987.

10. Hibbs M. F., Bradley W. L., “Correlation between micro-mechanical failure processes and the delamination toughness of Graphite/Epoxy systems”, Fractography of modern engineering materials: composites and metals, ASTM STP 948, J. E. Masters and J. J. Au, Eds., *American society for testing and materials*, Philadelphia, pp. 68-97, 1987