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Environmental stress-corrosion cracking of fiberglass: Lessons learned from failures in the chemical industry

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Abstract

Fiberglass reinforced plastic (FRP) composite materials are often used to construct tanks, piping, scrubbers, beams, grating, and other components for use in corrosive environments. While FRP typically offers superior and cost effective corrosion resistance relative to other construction materials, the glass fibers traditionally used to provide the structural strength of the FRP can be susceptible to attack by the corrosive environment. The structural integrity of traditional FRP components in corrosive environments is usually dependent on the integrity of a corrosion-resistant barrier, such as a resin-rich layer containing corrosion resistant glass fibers. Without adequate protection, FRP components can fail under loads well below their design by an environmental stress-corrosion cracking (ESCC) mechanism when simultaneously exposed to mechanical stress and a corrosive chemical environment. Failure of these components can result in significant releases of hazardous substances into plants and the environment.

In this paper, we present two case studies where fiberglass components failed due to ESCC at small chemical manufacturing facilities. As is often typical, the small chemical manufacturing facilities relied largely on FRP component suppliers to determine materials appropriate for the specific process environment and to repair damaged in-service components. We discuss the lessons learned from these incidents and precautions companies should take when interfacing with suppliers and other parties during the specification, design, construction, and repair of FRP components in order to prevent similar failures and chemical releases from occurring in the future.

Keywords: Environmental stress-corrosion cracking; Fiberglass reinforced plastics; Composites; Acid

1. Introduction

Because of their corrosion resistance, fiberglass reinforced plastics (FRP) are often used to construct tanks, piping, scrubbers, beams, gratings and other components for use in corrosive environments. However, fiberglass is susceptible to failure by environmental stress-corrosion cracking (ESCC) similar to traditional metal components [1,2]. In ESCC, the simultaneous presence of a corrosive environment (typically acid) and applied load combine to cause premature failure of the fiberglass. Failure due to ESCC can occur in components loaded at levels much below their design load. The ESCC failure mode occurs in fiberglass because many types of glass fibers, which are the primary load-supporting component of an FRP matrix, are susceptible to attack by acids. ESCC will typically occur faster in components under higher loads, at higher acid concentrations, and at higher temperatures.

1.1. Susceptibility of glass fibers to corrosion

A variety of glass fibers are manufactured with varying resistance to acid attack. Descriptions of the common uses of a major manufacturer's glass fibers are listed in Table 1.

The relative performance of these types of glass fibers in an acid environment is reflected in the 1-day weight loss data shown in Table 1. These data do not measure stress-corrosion cracking performance, which is the performance under load in an acid environment; however, the choice of glass fiber does affect the performance of a fiberglass structure operating in an acid environment. Recent testing of different commercial glass fibers has shown a relationship between the uniform corrosion resistance of glass fibers and the ESCC resistance of laminates made from those fibers [5]. In many traditional FRP structures, a less chemical resistant fiber, such as E-glass, is used as the

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Table 1
Description of glass type applications and susceptibility to acid attack [3,4]

Glass type	Application	One day weight loss in 10% H_2SO_4 (%)
E-glass	General purpose fibers	39
ECR-glass®	Used where acid corrosion resistance is desired	6.2
S-2 glass®	Used for reinforcement in composite structural applications which require stability under extreme corrosive environments	4.1
C-glass	Used for chemical stability in corrosive acid environments	2.2
A-glass	General purpose fibers	0.4

main structural fiberglass and a more chemical resistant fiber is used in a corrosion barrier. Some authors recommend that for long-term durability of FRP structures, corrosion resistant fibers should be used throughout the entire matrix [5].

1.2. Protection of glass fibers

Because many types of glass fibers that are used in FRP are susceptible to attack by acid, successful long-term application of FRP in an acid environment under stress requires that the fibers be protected from exposure to the acid. A protective resin-rich layer approximately 100–250 mil thick is often used on the surfaces of the structure adjacent to the acid and the structural fibers, such as E-glass fibers, are located in the interior of the structure beneath this protective layer [6]. The outer layer of the protective layer typically consists of approximately 95% resin and is reinforced by surfacing veils of a corrosion resistant glass fiber. Beneath the external protective material, an intermediate resin-rich layer, typically about 75% resin, containing an acid-resistant chopped glass reinforcement mat may be utilized as a transition layer between the resin-rich external layer and the less acid resistant internal structural glass. The satisfactory performance of the fiberglass structure in an acid environment depends on maintaining the integrity of the protective layer.

Major manufacturers of resins for fiberglass components typically provide guides or free technical support with recommendations of the resin and protective layer thickness to use in specific chemical and temperature operating environments [6]. Where previous experience with FRP structures in specific operating conditions is not available, resin manufacturers will often supply fiberglass coupons made from different resins and glass fibers for testing in a specific environment. However, true resistance to ESCC can only be measured by testing samples in a chemical environment under representative loads.

1.3. Determination of ESCC failure mode by examination of fracture surfaces

Correct diagnosis of failures caused by ESCC is important to prevent future similar failures from occurring. Fracture surfaces created by ESCC have distinctive characteristics on both macroscopic and microscopic scales aiding in the identification of ESCC as a failure mode. In Fig. 1, comparison fracture surfaces are shown for a beam from an FRP grating that failed due to an ESCC mechanism and an exemplar beam that was intentionally mechanically overloaded. The ESCC fracture surface in Fig. 1a is relatively smooth and planar, and the fracture surfaces of the individual fibers are in the same plane as the fracture surface of the resin matrix. In contrast the mechanically overloaded

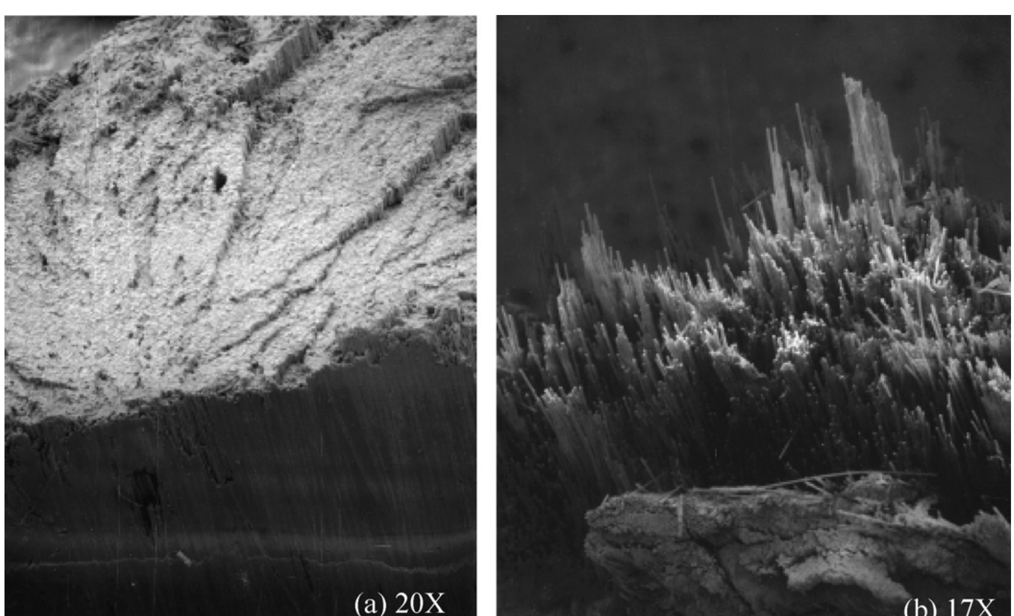

Fig. 1. (a) On the left is a photomicrograph at 20× magnification of the ESCC fracture surface of a failed beam in an FRP grating. The fracture exhibits a smooth, planar surface and the glass fibers are even with the surface of the resin matrix, characteristic of ESCC. (b) On the right is a photomicrograph at 17× magnification of the fracture surface of an exemplar grating beam that was mechanically overloaded. The fracture surface exhibits an irregular surface and broom like structures from fiber pull out from the resin matrix, characteristics of mechanical overload.

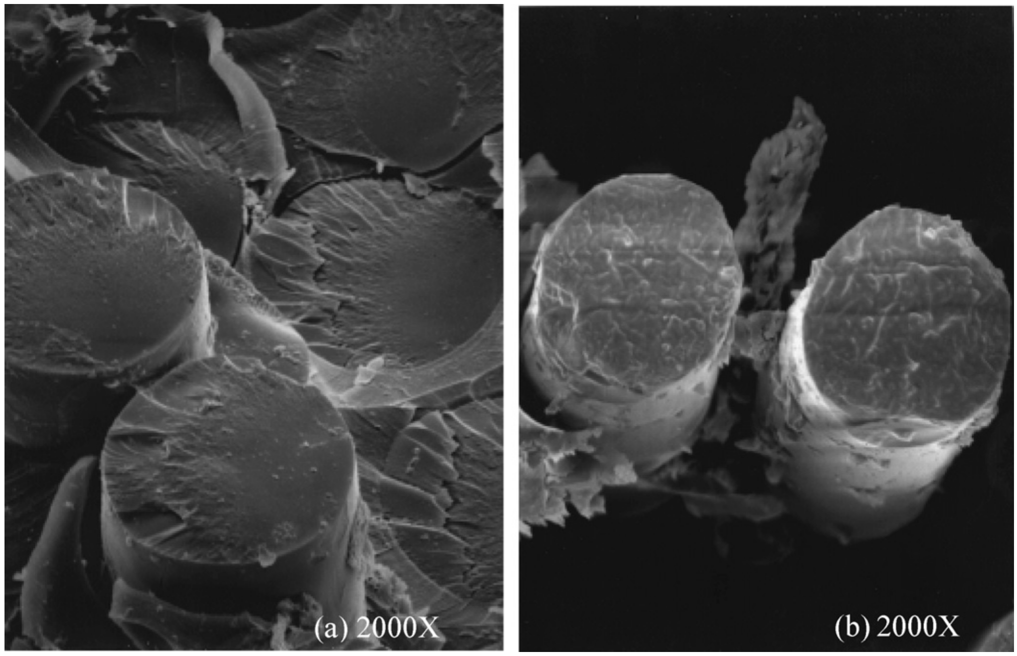

Fig. 2. (a) On the left is a photomicrograph of individual fibers from the ESCC fracture surface in Fig. 1a at 2000× magnification. The ESCC failure mode creates fracture surfaces on individual fibers with mirrored smooth regions and hackles. The mirrored regions are visible on the right hand side of fibers in the figure. The hackles, present on the left hand side of fibers in the figure, transition into river lines in the resin matrix. (b) On the right is a photomicrograph of individual fibers from the mechanically overloaded fracture surface in Fig. 1b at 2000× magnification. This failure mode creates rough fracture surfaces on the fibers and the fracture surfaces appear significantly above and below the resin matrix fracture surface due to fiber pull out.

fracture surface in Fig. 1b is rough with broom like structures due to glass fiber pull out from the resin matrix. The fracture surfaces of most individual fibers are distributed significantly above and below the fracture surface of the resin matrix.

Higher magnification images of these fracture surfaces are shown in Fig. 2 where fracture surfaces on the individual fibers are visible. The fibers on the ESCC fracture surface in Fig. 2a shows two regions on each fiber: a flat mirror region, which results from slow crack growth in the fiber, and a second region of rougher radial ridges (hackles), which are caused by rapid crack growth when the fiber finally fails. The hackles transition into river lines within the resin matrix. In contrast the fracture surface on the mechanically overloaded fibers in Fig. 2b do not have mirror regions but only rough surfaces indicative of fast fracture growth. In ESCC fracture surfaces, the mirrored regions are larger where the stress was lower during crack propagation and smaller where the stress was higher [7]. For a propagating crack, stress is typically lowest near the origin of the crack and becomes larger as the crack propagates and the original load is being supported by a smaller cross-section of material [8]. Hence, larger mirrored regions will typically be found near the fracture origin. As will be shown in one of the case studies, fibers that continue to be exposed to an acid environment after fracturing may exhibit axial, longitudinal, and radial cracking [9].

2. Case studies

The following two case studies investigated by the authors provide examples of failures that occurred due to ESCC of FRP composites. Lessons learned from these failures can prevent future failures from occurring. Examples of other failures of fiberglass due to ESCC are reported in the literature [8,10–14].

2.1. *Failure of fiberglass grating exposed to sulfuric acid*

Chambers containing biological media converted dilute sulfur containing gases in a plant's exhaust stream to sulfuric acid (H_2SO_4). A schematic of a chamber is shown in Fig. 3. Process gases traveled up through the filtration media, which was supported by a grating constructed from FRP I-beams. The I-beams, which were approximately 7 ft long and 2 in. tall, were subjected to bending stresses as shown in Fig. 3 due to the weight of the media. Water was sprayed into the top of the chamber resulting in an aqueous stream of about 1 wt.% H_2SO_4 trickling over the I-beams. Within 3–4 months of startup, all chambers put into service had failed.

2.1.1. *Identification of ESCC as the cause of failures*

After opening the chambers and removing the biological media, it was found that the fiberglass gratings had failed at midspan as shown in Fig. 4. The fracture surfaces were examined with a scanning electron microscope (SEM) and were previously shown in Figs. 1a and 2a. These fracture surfaces had the distinct characteristics of ESCC and so it was necessary to determine why these beams had been susceptible to ESCC.

2.1.2. *Lack of corrosion barrier at maximum stress region*

Cross-sections of undamaged regions of beams were examined and a photomicrograph of the bottom or tension flange of a beam is shown in Fig. 5. The beams were created using a pul-

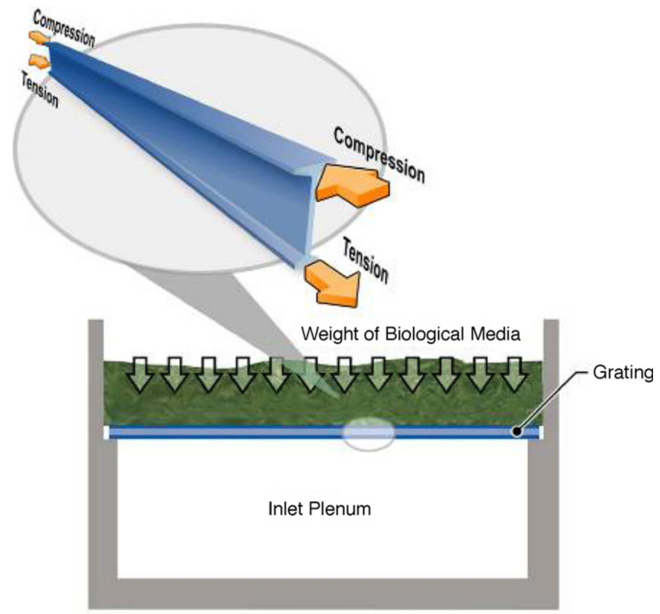

Fig. 3. Schematic of biological media chambers. Process gases flow up through the biological media. An aqueous stream containing H_2SO_4 drains down over the beams. FRP I-beam gratings support the biological media.

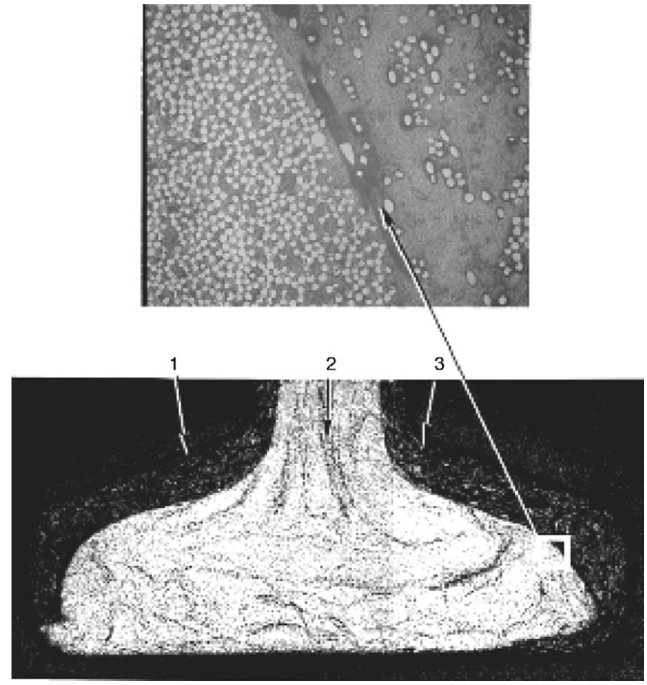

Fig. 5. Photomicrograph of bottom tension flange of I-beam. Resin lean region is labeled 2 and contains structural glass fibers. Resin-rich layers labeled 1 and 3 are present on top and sides of tension flange, but not on bottom of tension flange.

Fig. 4. Grating beams fractured at mid-span in biological media chambers.

trusion process and consisted of a central section of continuous structural E-glass fibers aligned along the length of the beams. A protective resin-rich layer labeled 1 and 3 in the figure was present on the sides of the beam and the top of the bottom tension flange. However, this resin-rich layer was not present on the bottom of the tension flange or the top of the compression flange. Fig. 6 schematic shows the location of the protective layer on the beams. The lack of a protective layer on the bottom of the tension flange was significant because this is the maximum tension stress location in the loaded beams. As a result,

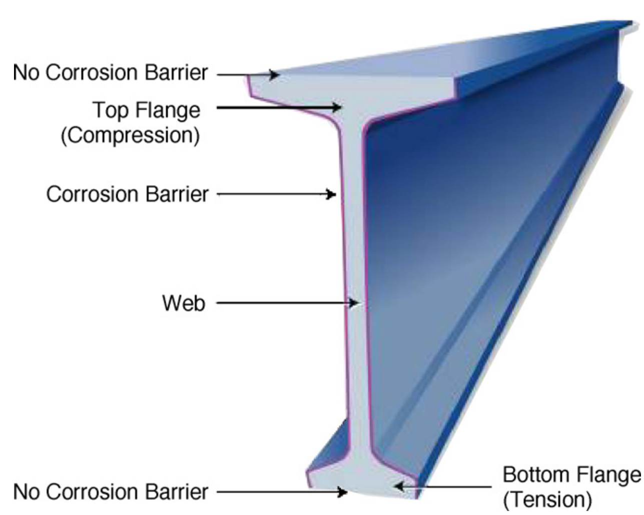

Fig. 6. Schematic of cross-section of grating beams. Corrosion barrier is present on web of beams but not on the upper surface of the top flange or lower surface of the bottom flange.

Fig. 7. Apparatus used to test beam pairs under load in various aqueous environments. Six sets of beam pairs were enclosed in flexible hose and weights were hung from the beams to load beam pairs in a four point bending configuration. Aqueous solutions were circulated through the flexible hose with peristaltic pumps.

the structural E-glass fibers under tension were exposed to acid causing ESCC to occur. Although the resin rich protective layer was also missing on the top flange of the I-beam, this flange was not subject to ESCC because it was in compression.

2.1.3. Testing of exemplar beams

The manufacturer of the beams claimed that ESCC occurred not because of the lack of a corrosion barrier on the bottom of the tension flange, but because the presence of dilute CS_2 in the process streams caused the resin layer to swell, allowing acid to attack the structural fiberglass. While some resins are not compatible with concentrated CS_2, the resin manufacturer confirmed that the specific resin used in the beams would be suitable for the application because it would not be adversely affected by the presence of CS_2 at the low concentrations present in the chambers. The average CS_2 concentration was less than 1 ppm in the aqueous phase and approximately 180 ppm in the vapor phase. To confirm that the dilute CS_2 played no role in the failures, beam pairs (two attached beams) were tested under two loads in aqueous solutions containing CS_2 alone, CS_2 and H_2SO_4 combined, and H_2SO_4 alone at concentrations representative of the chambers. The apparatus used in the testing is shown in Fig. 7.

Results from these tests are shown below in Table 2. The test results showed that beams loaded with a mid-span stress of 11,000 psi and exposed to H_2SO_4 failed in 4.5 days regardless of whether CS_2 was present. Similarly beams loaded with a lower mid-span stress of 8400 psi and exposed to H_2SO_4 failed in 9.5–10 days regardless of whether or not CS_2 was present. The tests demonstrate that ESCC occurs more rapidly in beams under greater loads even though the loads were significantly below the beams' ultimate strength. By comparison the beams in the chamber were designed to support a load corresponding to a stress of 10,500 psi and were reported to have a failure stress of 100,000 psi. Beams that were only exposed to CS_2 did not fail during 52 days of testing and showed no signs of deterioration. These tests confirmed that the presence of CS_2 was not necessary to cause the beams to fail and that the beams would fail due to exposure to H_2SO_4 alone under load due to the lack of a corrosion barrier on the bottom of the tension flange. Furthermore, the presence of dilute CS_2 did not accelerate the failures refuting the beam manufacturer's claim that CS_2 swelling the resin had allowed the ESCC to occur.

2.2. Failure of an FRP tank containing a hydrochloric acid solution after repair of a tank leak

A 43,000 gallon tank, 37 ft tall and 14 ft in diameter was in use in a tank farm to store acid solutions with HCl concentrations ranging from 5 to 17%. After 1.5 years of use a small leak from the tank shell was observed approximately 8–10 ft from the bottom of the tank. The tank manufacturer was called to patch the tank and reported finding a $3'' \times 6''$ star-crazing in the interior 100-mil corrosion liner in the area of the tank leak. The damage was claimed to have been caused by a point loading impact. During a later inspection it was noted that several star-crazing marks and linear scratches in the corrosion liner were present in other regions of the interior of the tank. Chopped mat and resin patches were field bonded to both the tank interior and exterior to arrest the leak. It was reported that the technician applying the patch had not completely ground out the crack, but merely scuffed the surface of the area of the leak before applying the patch. The locations of the interior and exterior patches are shown in Fig. 8. The inner patch covered a larger area of the tank than the outer patch.

After the repair, the tank was brought back on line and sometime after being refilled, a leak was observed near the region of the patch. The tank subsequently collapsed onto an adjacent building as shown in Figs. 9 and 10. The collapse caused approx-

Table 2
Test results from beams tested in three aqueous solutions under two loads

Beam pair	Aqueous solution	Mid-span stress (psi)	Average CS_2 concentration (ppm)	Average H_2SO_4 concentration (wt.%)	Failure time (days)
1	CS_2	11000	0.9	0	No failure after 52 days
2		8400	0.9	0	No failure after 52 days
3	CS_2 and H_2SO_4	11000	0.5	0.9	4.5
4		8400	0.3	1	9.5
5	H_2SO_4	11000	0	1	4.5
6		8400	0	1	10

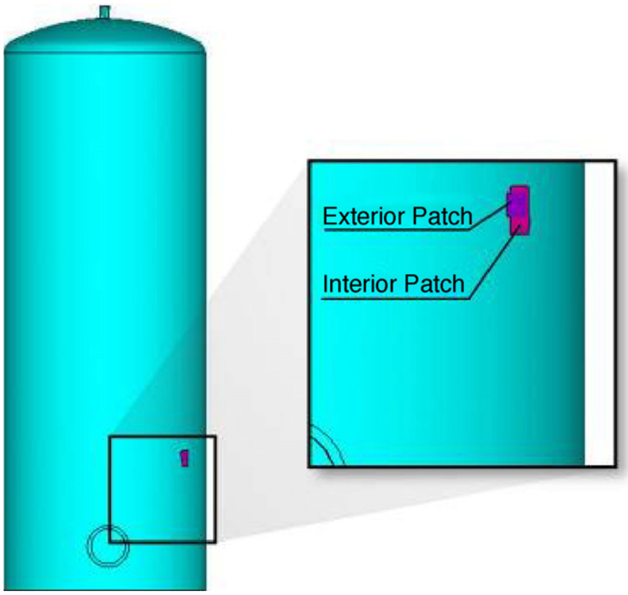

Fig. 8. Location of interior and exterior patches on tank.

imately 32,000 gallons of an acid solution to spill out into the surrounding areas and tank farm sump.

2.2.1. Description of the failure

Inspection of the tank after the collapse indicated that an approximately 1 ft tall vertical fracture had formed through the previously applied patch. During the collapse, a 1-ft band on either side of the fracture surface "unzipped" circumferentially. Photographs of the two sides of the initial vertical fracture on the tank are shown in Figs. 11–13. The upper patch in Fig. 13 shows a branch point indicating the crack first traveled vertically, before

Fig. 9. Collapsed tank in tank farm.

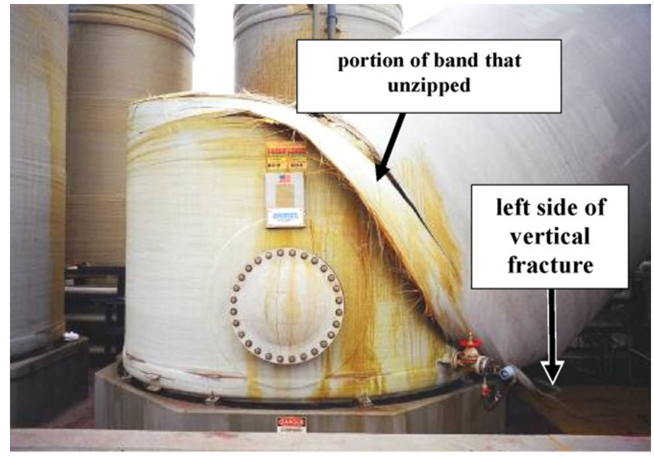

Fig. 10. Close-up view of bottom of collapsed tank. The band that unzipped circumferentially and the post incident location of the left side of the fracture surface are visible in the photograph. The right side of the fracture surface is not visible in this photograph.

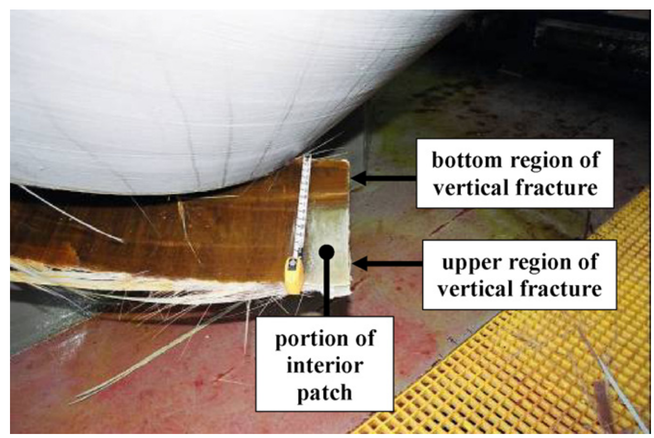

Fig. 11. Left side of initial vertical fracture. The interior side of the band of the tank wall that unzipped circumferentially, is visible in the photograph. Portion of interior tank patch is visible on upper region of vertical fracture.

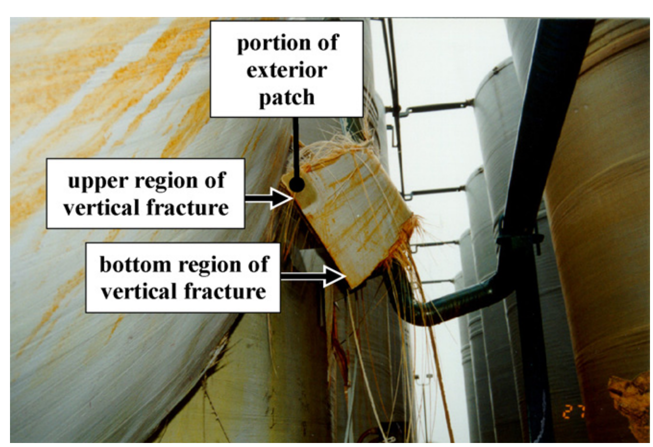

Fig. 12. Right side of initial vertical fracture still attached to tank. The exterior side of the band of the tank wall that unzipped circumferentially is visible in the photograph. Portion of exterior tank patch is visible in upper region of the initial vertical fracture.

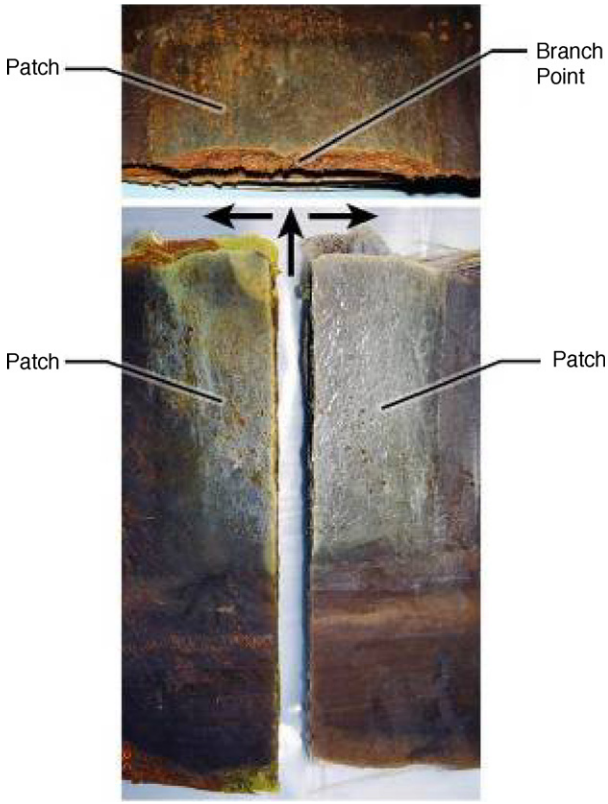

Fig. 13. Interior view of mating portions of tank wall near initial vertical fracture. The three portions of the original interior patch are visible near the intersection of the three surfaces. The vertical fracture surfaces in the figure correspond to the vertical fracture surfaces shown in Figs. 11 and 12. Note that the upper patch shows crack branching, where the vertical crack transitioned into two horizontal cracks. This clearly demonstrates that the origin of the cracking is in the vertical portion of the fracture. The black arrows indicate the branching of crack propagation at the top of the fracture surfaces.

branching and traveling horizontally. The relatively smooth, planar vertical fracture surfaces that lacked fiber pull out suggested ESCC as a failure mechanism. SEM examination of the fracture surfaces was used to further investigate the cause of the failure.

2.2.2. SEM examination of vertical fracture surface

Samples removed from two regions with and without patches along the length of the vertical fracture surface on the right side of Fig. 13 are shown in Fig. 14. SEM images at increasing magnification are shown for each region in Figs. 15 and 16. The fracture surfaces exhibit characteristics of ESCC as described in the figure captions. However, each region has unique characteristics indicating crack growth from inside the patched region, region 1, and propagating beyond the patch through region 2. The fractured fibers in region 1 show significant cracking indicating they received long-term exposure to acid after their initial failure. Region 2 shows characteristics of ESCC, without additional cracking of the fibers indicating it had occurred more recently. Additionally, the location of mirrored regions on the left hand side of fibers in region 2, indicate that the crack propagated from top to bottom of the tank through this region (left to right as oriented in Figs. 14 and 16).

Fracture surfaces indicate failure by ESCC. Cracking within fibers indicates long-term exposure of fibers to an acid environment after the initial failure.

2.2.3. Inappropriate application of patch

A photograph of the tank wall cross-section through the patched region is shown in Fig. 17. Cracks are present in the interior of the tank wall that end abruptly at the location of the interior patch indicating they were present before the patch was applied. This confirms that the technician who applied the patch had not fully ground out the damaged region of the tank before applying the patch.

The purpose of a repair patch as applied to a fluid storage tank is to restore both the fluid tightness and the structural integrity at the location in the shell that has been damaged or suffered a breach of the wall. A repair patch restores fluid tightness by supplying a complete seal around the breach. Structural integrity is restored by adequately creating a new path for the forces transmitted through the tank wall that circumvents the region of the breach. The patch applied to the tank only temporarily restored the fluid tightness of the tank without addressing the underlying structural damage to the tank.

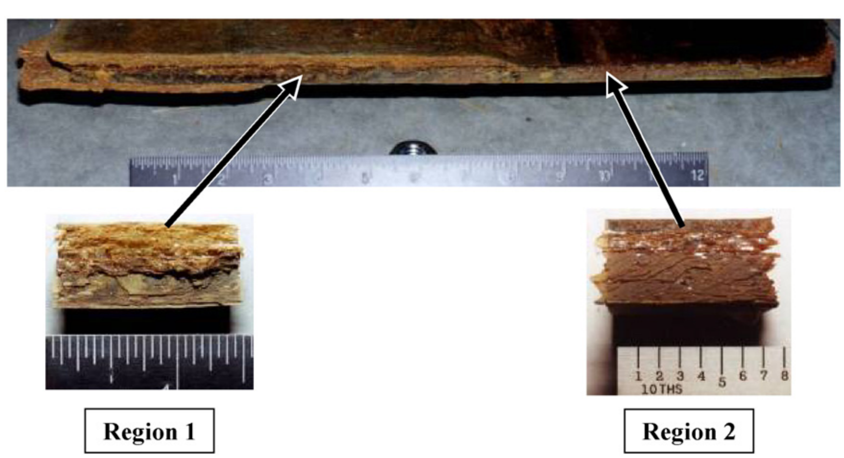

Fig. 14. Cross-sectional view of the vertical fracture surface on the right side of Fig. 13. Location of samples taken from two regions along the length of the fracture surface. Portions of interior and exterior patches are visible on the left hand side of the photograph. The orientation of the samples in the photographs correspond to the top of the tank being to the left and the interior of the tank being up in the figure.

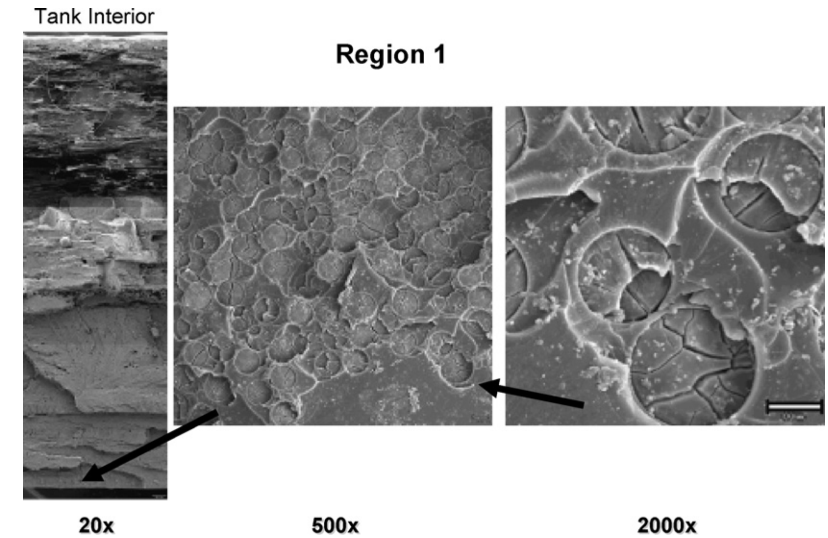

Fig. 15. View of fracture surface in the patched region, region 1, near the origin of the ESCC cracking. Image on left is the entire cross-section of the wall, images in middle and right are higher magnification views of regions indicated with arrows. The orientation of the samples in the photographs correspond to the top of the tank being to the left and the inside of the tank being up in the figure.

The presence of the through wall leak in the FRP tank indicated that acid had penetrated the tank's corrosion barrier and was able to attack the structural fiberglass in the tank wall. In repairing the leak, it is critical to fully grind out the damaged region of the tank wall in order to reveal the extent of the damaged region and to remove all of the damage. Because the technician did not grind out the damaged region of the tank, he was not fully aware of the extent of the damage and applied an inadequate tank patch. After the repair, the undetected crack was able to continue to propagate vertically due to the circumferential tension stress in the tank leading to the collapse of the tank.

The preferred method for patching a damaged composite is to use a tapered or scarfed joint [15]. Scarfing the tank wall exposes, and provides a means for the repair patch to adhere directly to, a complete cross-section of the tank wall reinforcement. This provides a relatively direct transfer of the forces in the tank wall into the patch. In the absence of scarfing, the patch merely adheres to the surface of the tank wall, creating an inferior connection with regard to strength and stiffness. Scarfing also serves to reveal the complete cross-section of the damage region, providing a means of assessment of the full extent of the damage.

2.3. *Lessons learned from case studies and general guidance*

The case studies reviewed in this paper teach several lessons for preventing future failures due to ESCC of FRP composite structures.

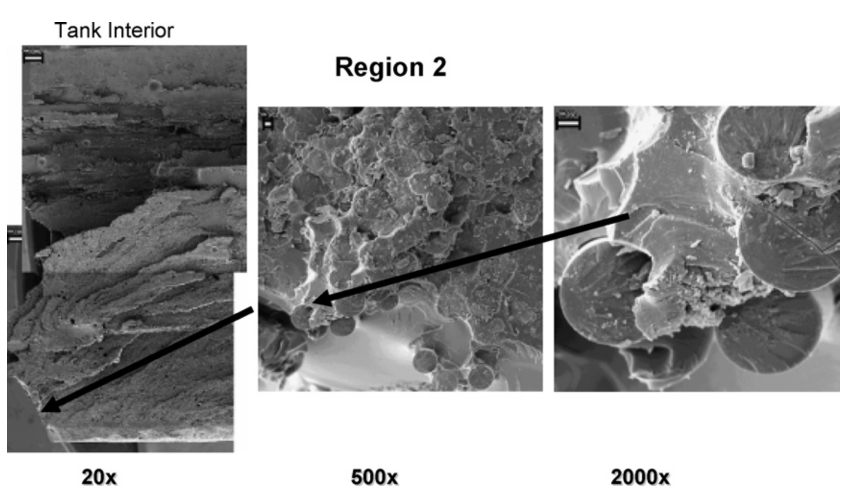

Fig. 16. View of fracture surface in region 2 away from the patch and the origin of the ESCC cracking. Image on left is the entire cross-section of the wall, images in middle and right are higher magnification views of regions indicated with arrows. The orientation of the samples in the photographs correspond to the top of the tank being to the left and the inside of the tank being up in the figure. Fibers show mirrored regions and radial hackling transitioning into river lines in matrix indicating failure by ESCC. Note also that the mirror zones in right hand photograph are on the left side of the fibers, indicating that the origin of the ESCC cracking is to the left of this location on the fracture surface, i.e. the fracture initiated to the left of region 2 in Fig. 14.

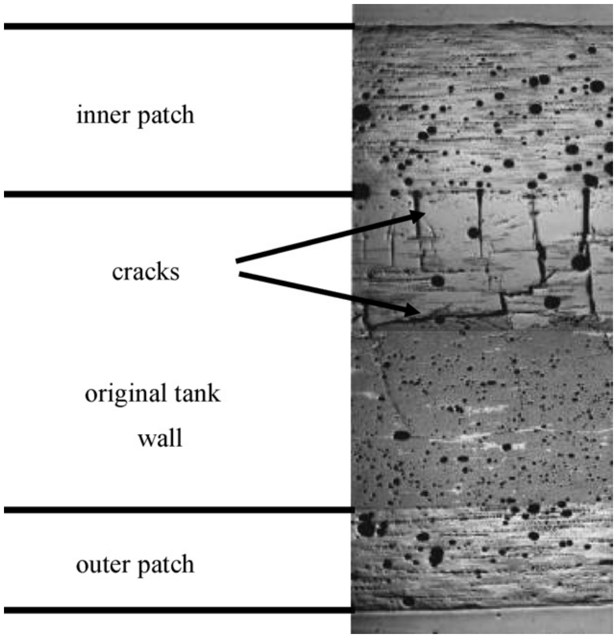

Fig. 17. Cross-section of tank wall. The orientation of the sample in the photographs corresponds to the top of the tank being to the left and the inside of the tank being up in the figure. Cracks were present in the tanks inner corrosion barrier and tank wall before the patch was applied.

- When searching for suppliers of FRP components for corrosive environments, identify suppliers who understand the special protection needs of glass fibers in corrosive environments.
- Ensure that the FRP supplier is fully aware of the stress, temperature, pressure, and chemical environment in which the material will be used and ask them to confirm in writing that the proposed material is appropriate for the application.
- Ask the equipment supplier to describe the corrosion barrier, fiberglass, and resin system that will be used and their basis for determining that they will be adequate for your installation.
- Consider consulting the resin supplier or a knowledgeable consultant to determine if they agree that the corrosion barrier, fiberglass and resin chosen by the equipment supplier will be adequate for the installation.
- Consider performing accelerated testing (higher chemical concentrations and loads) on samples of FRP that will be used in unique chemical environments to ensure the materials are adequate.
- Consider inspecting new FRP equipment when received from the manufacturer to ensure that the specified corrosion barrier is present in critical areas and has not been damaged during shipping or installation. Periodic inspection during the life of the equipment can identify flaws before they lead to catastrophic failure.
- For long-term stability of FRP components in extremely corrosive environments, consider using glass fibers that are resistant to the specific chemical for structural glass as well as corrosion barriers.
- Recognize that when leaks or fractures occur in FRP equipment, acid may have penetrated a significant region of the structural fiberglass beyond any defects visible in the external fiberglass.
- When repairing leaks in FRP tanks or other equipment, fully grind out all damaged regions until no damage or residual acid is present within the wall.
- Use tapering or scarfing to apply patches to repaired areas and ensure the patch has been designed to restore full structural integrity as well as fluid tightness.

3. Conclusions

Fiberglass reinforced plastic (FRP) is often used to construct tanks, piping, scrubbers, beams, grating, and other components for use in corrosive environments. While FRP typically offers superior and cost effective corrosion resistance relative to other construction materials, the glass fibers traditionally used to provide the structural strength of the FRP can be susceptible to attack by the corrosive environment. The structural integrity of traditional FRP components in corrosive environments is usually dependent on the integrity of a corrosion-resistant barrier, such as a resin-rich layer containing corrosion resistant glass fibers. Without adequate protection, FRP components can fail at well below their design loads by an environmental stress-corrosion cracking (ESCC) mechanism when simultaneously exposed to mechanical stress and a corrosive chemical environment. Failure of these components can result in significant releases of hazardous substances into plants and the environment.

Two case studies provide examples of failures that occurred due to a corrosion barrier missing from critical areas of I-beams, damage to a tank wall corrosion barrier, and improper patching of a leak in a tank wall. When purchasing FRP equipment for corrosive environments it is important to deal with suppliers familiar with the special requirements of these applications and to ensure they provide adequate corrosion barriers to protect the fiberglass from failure by ESCC. For long-term stability of FRP components in extremely corrosive environments, using glass fibers that are resistant to the specific chemical for structural glass as well as corrosion barriers.

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