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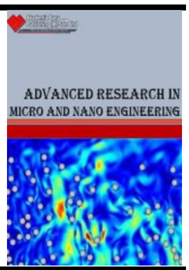

A Novel Investigation on Microstructural Analysis of a Rupture GRE Composite Pipe for Underground Fire Water System

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ABSTRACT

This study investigates how underground fire water systems using Glass Reinforced Epoxy (GRE) pipes are affected by aging. Over time, GRE pipes can degrade, potentially reducing their expected lifespan. The study compared damaged and undamaged areas of pipes to assess how damage impacts their structural integrity. Microscopic and chemical analyses (SEM and EDX) were performed on pipe samples from different locations of the pipe. Mechanical testing (following ASTM standards) was conducted beforehand. Analyses revealed that as damage increased (closer to damaged area), the levels of carbon (C) and oxygen (O) changed significantly with the weight percentage exposed 45.62% difference and 11.62% difference, respectively, as compared with the undamaged area. This suggests that the epoxy resin degrades with age, releasing these elements. Additionally, the presence of foreign objects around the pipes can affect how they bear loads. These findings highlight the importance of understanding how aging and surrounding conditions can influence the performance of GRE pipes in real-world fire water systems.

1. Introduction

Glass Reinforced Epoxy (GRE) pipes, consisting of resin and glass fibers, find extensive use in the oil and gas industries due to their strength, corrosion resistance and durability lasting up to 50 years with low maintenance [1]. They offer advantages over carbon steel, including corrosion resistance, energy-efficient manufacturing, and enhanced flow capacity in smaller diameters. In firewater protection systems, GRE pipes exhibit reduced susceptibility to corrosion and high thermal resistance.

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Despite these benefits, reported failures, often linked to manufacturing defects and harsh environments, highlight limitations. GRE pipes may not be suitable for high-temperature applications or specific corrosion resistance needs. ISO standards like ISO14692 and AWWA M45 provide guidelines for fiberglass pipe issues [2].

The manufacturing process involves combining E-glass and C-glass in a filament-wound plastic design, blending fiberglass strength with chemical resistance. This process includes winding composite pipes on rotating mandrels using wet or reimpregnated tape winding techniques, with epoxy resin reinforced by E-glass strands at a 45° angle and a chemically resistant C-glass liner [3,4]. Glass fiber alignment maximizes strength in both longitudinal and circumferential directions, providing double strength in the hoop direction. Some products use a single winding angle, while others employ alternating layers.

However, uneven material distribution significantly impacts mechanical properties [5]. Variations in resin or hardener content can lead to poor performance and early degradation. Incorrect winding techniques, such as excessive tension, may reduce stiffness and weaken fiber bonds. The curing process at the installation site can cause uneven wall thickness. While epoxy is commonly used, formulations may have limitations, making careful consideration of material distribution, winding techniques, and epoxy properties essential for optimal mechanical performance [6].

Studies by Khan *et al.,* [7] and Haubler *et al.,* [8] focus on Glass Fiber/Epoxy (GFE) composite laminates and optimizing tensile strength in polymer composite pipes, respectively. Understanding GRE pipes' impact resistance is crucial, with factors like energy levels and fiber arrangement influencing strength [9]. Water aging and impact loading decrease burst strength in E-glass fiber/epoxy pipes [10,11]. In lightweight armor development, the nonlinear relationship between energy absorption and laminate thickness is emphasized [12]. Flexural strength is vital for GRE pipes facing severe conditions, and Azizian *et al.,* [13] suggest composite pipes are favorable, especially in earthquake-prone regions, providing insights for improved installation designs, failure prevention, and cost reduction.

Abdul Majid *et al.,* [14] investigation into hydrothermal aging reveals significant weaknesses in compressive strength, emphasizing the detrimental effects of resin separation, microcracking, and degraded fiber-matrix bonding. Timely addressing microcracks is crucial to prevent catastrophic failures, especially when material layers are vulnerable to applied loads [15,16]. Additionally, studies employing ASTM D2290's SDT method [17] shed light on the impact of notches or holes, indicating reduced load capacity and localized failure due to stress concentration.

Furthermore, Liao *et al.,*'s examination of resin content in composite pipes [5] underscores challenges in maintaining industry standards, influencing fracture localization in GRE pipes. In the context of underground applications, Hassan *et al.,*'s study on GRP behavior [18] emphasizes vulnerability to environmental factors such as abrasive particles and chlorine exposure.

Epoxy resin coatings, widely used for corrosion protection in buried pipelines, face a potential challenge – water-induced degradation. Alrudayni's research [19] highlights this issue, revealing weakening of the adhesive bond between resin molecules wet-adhesion loss and destabilization of the entire resin matrix. Similarly, Fitriah *et al.,*'s study confirms this degradation process [32]. Water exposure leads to progressive damage, starting with debonding between the epoxy and reinforcing fibers. This creates voids within the coating, allowing even more water to enter. Subsequently, the epoxy itself leaches away, resulting in weight loss and compromising the coating's effectiveness.

Despite compliance with established standards, the underground pipeline utilized for industrial pool water transport frequently experiences ruptures, leading to material degradation and shorter-than-expected lifespan, resulting in persistent flooding incidents. The objective is to assess the impact of damage on the structural integrity of the GRE pipe through mechanical testing, including tensile, impact, and flexural tests following ASTM D3039, ASTM E23, and ASTM D790 standards. Specimens are extracted evenly from damaged and undamaged sections of the GRE pipe, and analysis is conducted using Scanning Electron Microstructure (SEM) and Energy-Dispersive X-ray (EDX). Figure 1 shows a summarized experimental design.

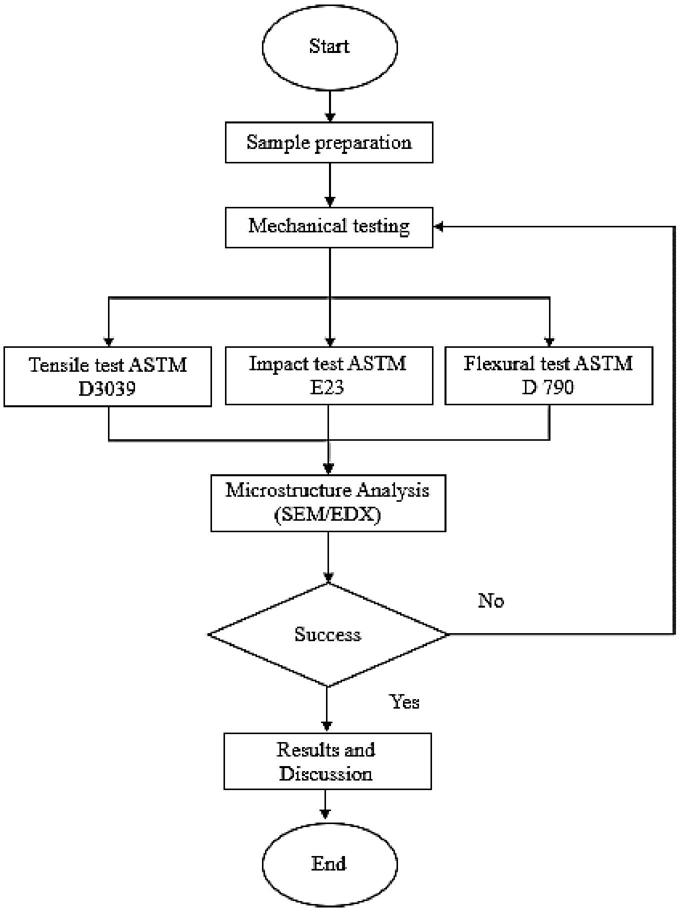

Fig. 1. Methodology process flowchart

2. Methodology
2.1 Sample and Material

In this study, a sample pipe from the Bonstrand 2420-FM series, designated as 1N103BQ confirming to ISO14692 standards, was used. The GRE pipe, with a design pressure of 18.4 Bar, design temperature of 75°C, epoxy resin construction, adhesive-bonded joint, 5mm thickness, 800mm length, and 150mm nominal diameter, meets industry standards with tailored specifications for durability and corrosion resistance. The pipe can withstand a maximum pressure of 16 Barg and operates within a temperature range of up to 75°C. An adhesive-bonded joint securely connects its components for structural integrity. The GRE pipe subjected to the study was divided into parts and set in the back, right, and left directions, as shown in Figure 2.

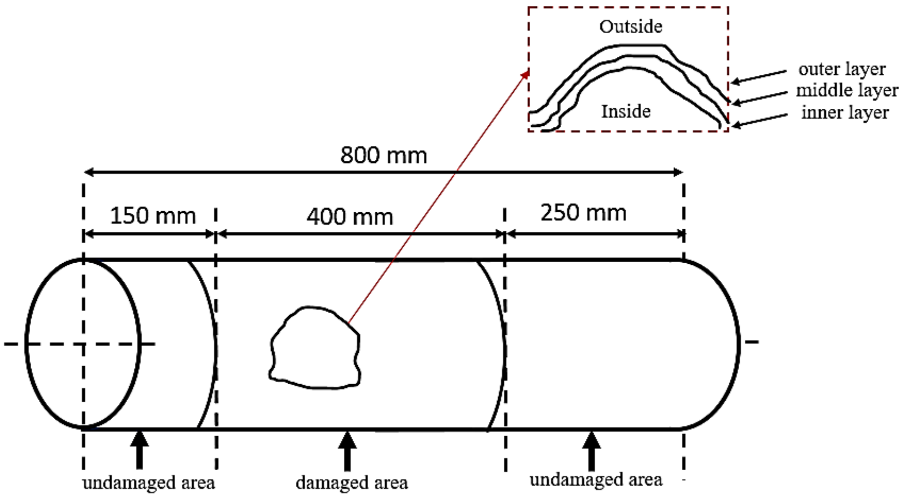

Fig. 2. Illustration area divided on the pipe

Specimens measuring 10 mm x 10 mm x 5 mm, matching the pipe's average thickness of 5 mm, were extracted for analysis. They were labeled A (furthest in the undamaged area), B (further in the damaged area), C (closer in the damaged area), and D (at the damaged area), as illustrated in Figure 3. The samples selected for analysis were derived from the mechanical tests with the highest values, including tensile, impact, and flexural tests, aiming to understand the failure mechanisms and their implications.

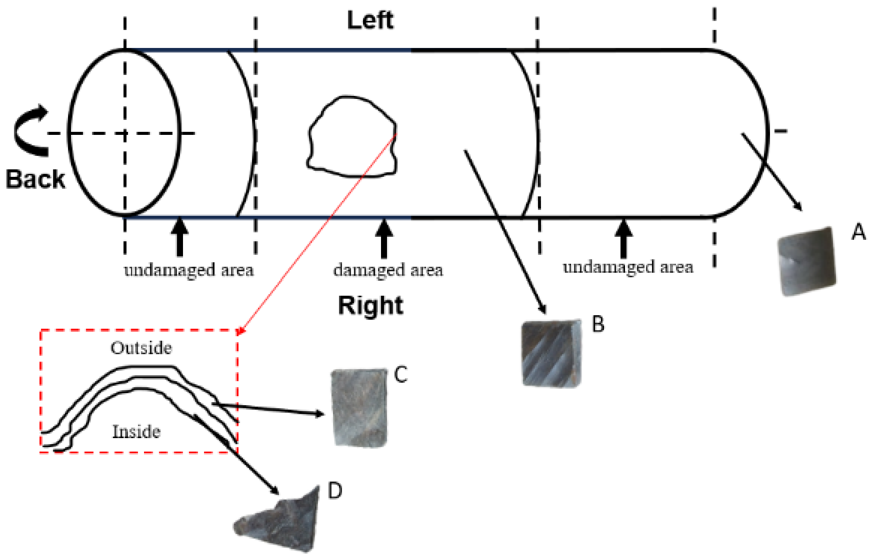

Fig. 3. Illustration specimens taken from the pipe

2.2 Method of Microstructural Analysis

The SEM and EDX analyses were conducted on the exterior surface layer of the specimens labeled A, B, C, and D. For the EDX analysis, specimens B, C, and D were tested to provide detailed compositional information. Figure 4 shows the machine for EDX and SEM.

Fig. 4. SEM and EDX analyses occupying the same machine

The SEM images were acquired using a JEOL JSM-6380LA series SEM machine. The SEM analysis was performed at a voltage of 15 kV with magnifications of 50x, 500x, 1000x, and 3000x. Each magnification test involved examining different sections, including top views, cross-sections, and bottom views. To ensure clear and non-fuzzy images, a thorough coating of gold was applied to the samples.

3. Results and Discussions

3.1 Scanning Electron Microscope (SEM)

The microstructure results pertain to the external surface of the GRE pipe, where the outer layer serves as a 100% epoxy resin barrier. This barrier is crucial for preventing the internal layer from being exposed, thereby maintaining the structural reinforcement provided by the glass fiber.

Prior to inspection, the condition of the buried GRE pipe was assessed. The results revealed backfill, and on-site inspections are essential for understanding GRE pipe performance, particularly in addressing issues related to improper embedment. Soil observations in reveal Class I backfill soil stiffness, requiring the use of granular materials—primarily crushed rock and gravel with <15% sand and ≤5% fines [2]. Ensuring fully filled voids through proper compaction is essential for optimal pipe support.

This compaction level minimizes settlement, deformation, and uneven support, ensuring long-term pipeline performance [20]. Backfill and bedding are essential for stable loads in sewer pipes, with proper compaction reducing stress and displacement. Research highlights a more significant reduction in stress (20%) and displacement (40%) for backfill compared to bedding [21]. Inadequate compaction may lead to settling, causing deformation and compressive stress on the pipe diameter. On-site observations also reveal foreign materials, such as non-metallic pipe elbows, wood, metal strips, and metallic wires, affecting backfill composition. Proper backfilling is crucial for GRE pipe support, emphasizing the need for more sand in cases with foreign materials. These findings highlight the importance of proper backfill material selection and quality control during GRE pipe installation to ensure optimal support and prevent stress concentrations.

To understand the impact of foreign materials on structure and integrity, Scanning Electron Microstructure (SEM) analysis is essential. Figure 5(a) and Figure 5(b) provide baseline micrographs of neat epoxy resin with fibers, captured from a fractured surface. In Figure 5, an in-depth exploration of the microstructure focuses on the outer layer at varying distances from the failure point.

Microcracks are observed, with Figure 5(c) showing fewer microcracks than Figure 5(d), indicating structural degradation in the GRE pipe.

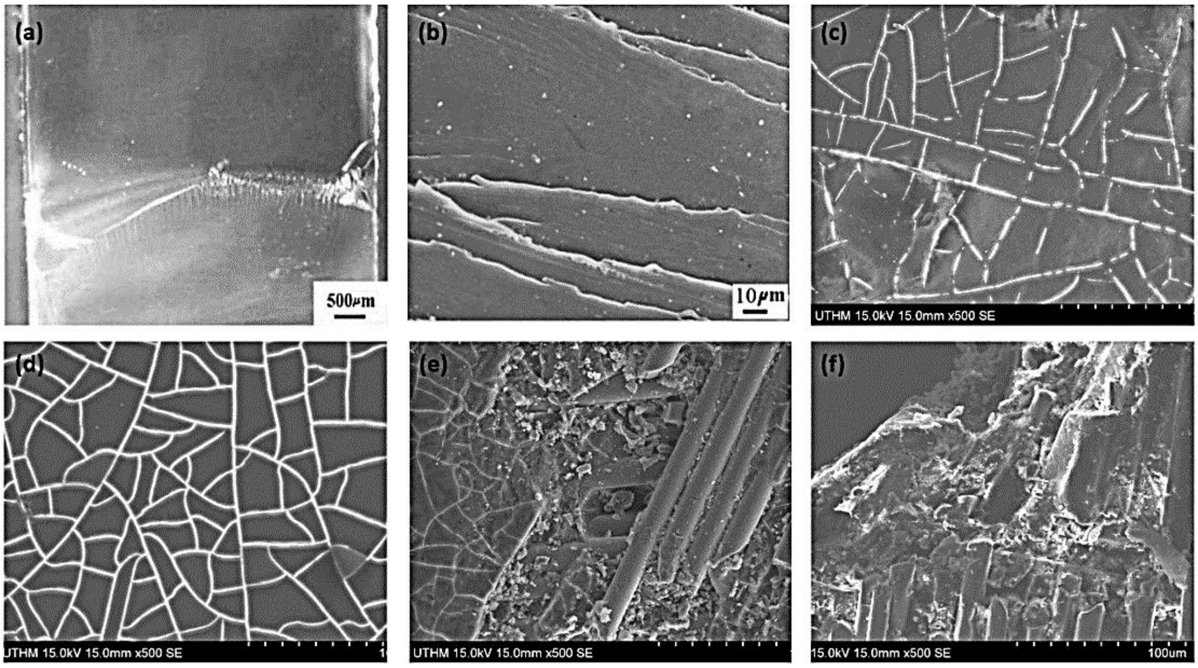

Fig. 5. (a) Optical micrographs of neat GRE [22], (b) SEM micrographs of neat GRE [22], (c) Undamaged furthers to failure, (d) Damaged furthers to failure, (e) Damaged close to failure, and (f) Damaged at the failure

Further examination of Figure 5(c) reveals an optimal condition farthest from failure, while Figure 5(d) showcases the damaged area, yet it is still farther from complete failure. Both figures illustrate the presence of microcracks on the surface. Additionally, matrix degradation is observed in the form of flake formation. Fiber damage is also noted, indicated by changes in fiber circularity, as shown in Figure 5(e) and Figure 5(f). Despite an initial inspection at X3000 magnification showing no obvious defects in the baseline micrographs, compare with magnification of X500 already exposes microcracks, offering insights into the GRE pipe's internal structure and defect progression.

In Harris *et al.,*'s study [23], microcracks are noted to reduce the strength and stiffness of composite materials, making them more susceptible to additional damage and eventual failure. Micro-cracks may initiate extensive damage, including delamination and inter-ply cracking, especially under subsequent loading or pressure conditions. In filament-wound composite pipes, even minor impacts leading to micro-cracking significantly reduce failure pressure by 60%, impacting structural integrity, safety, and long-term performance of composite piping systems.

Examining the fiber at point C mentioned, Figure 3 shows an adhesive failure mode, mainly seen in Figure 6(a). Adhesive failure happens when the adhesive material used is weaker than the materials being bonded. Microcracks, visible at these points in Figure 6(b), can propagate and eventually lead to larger matrix cracks, commonly observed in SEM images.

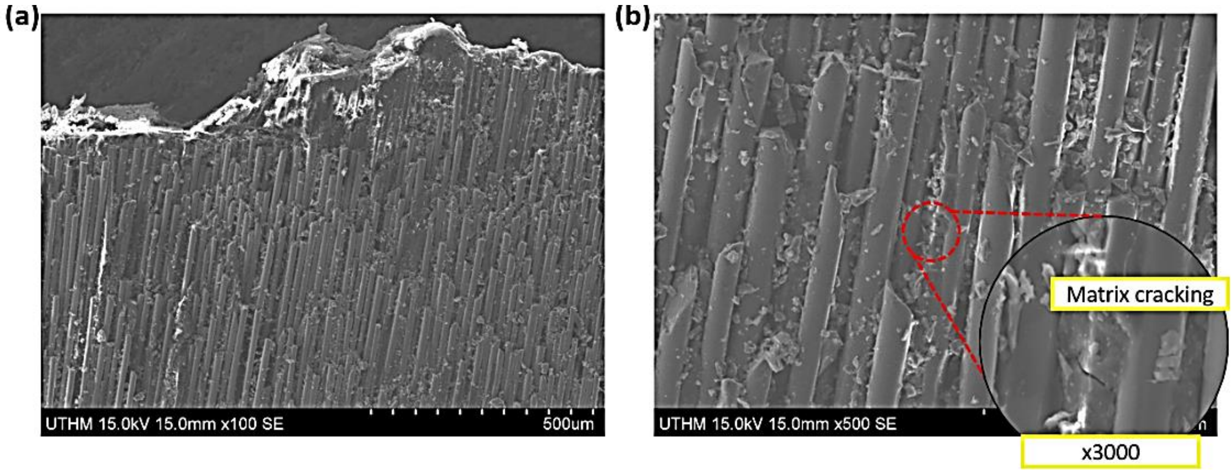

Fig. 6. (a) Point C damaged area, and (b) Matrix cracking

Microcracks in the matrix can be caused by factors like localized stress concentrations, material defects, and environmental conditions [33]. In Figure 7, as the applied tensile load increases during testing, these microcracks can spread and lead to matrix cracking. This process ultimately impacts the mechanical properties and structural integrity of the composite material. It's worth noting that all the tensile specimens showed the same whitening phenomenon.

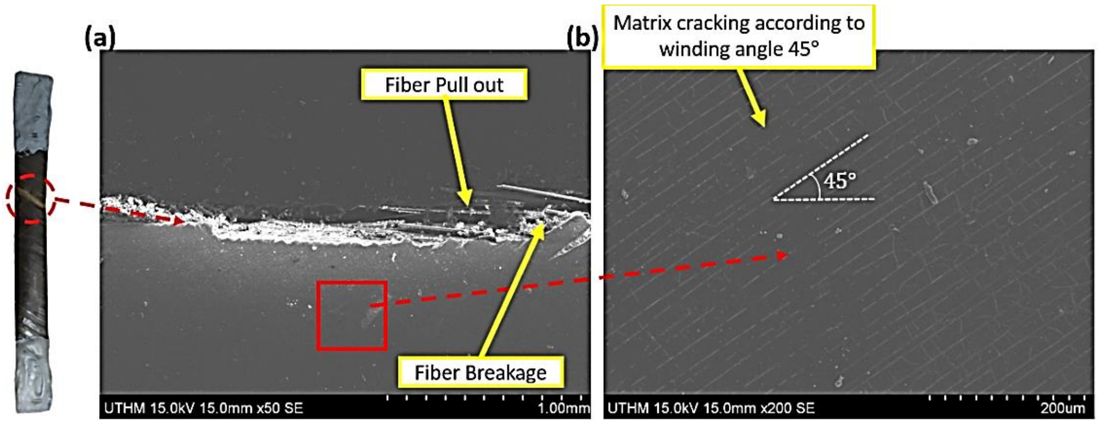

Fig. 7. (a) Failure modes on tensile sample, and (b) SEM whitening area tensile failure

The whitening observed on the surface, indicating early failure in the filament winding direction, has been identified by Hawa *et al.,* [10]. This whitening results from debonding and delamination processes within the multi-layer bonded structure of the pipe. Structural degradation leads to radial expansion due to hoop stress, exposing the interlayer structure to shear stress parallel to the fiber winding direction [5,34]. Radial cracks are formed and propagate based on the fiber winding angle in the layer stacking [35]. SEM analysis on the tested pipes revealed more intense observations of fiber breakage failures, delamination, and pull-out failures. Additionally, the glass fibers exhibit brittle failure behavior with a net fracture along the ply [17]. Identifying whitening during tensile testing can be a valuable tool for early detection of potential failure in the filament winding direction, allowing for preventative maintenance or pipe replacement before catastrophic failure. It's essential to determine the appropriate winding angle for both hoop and longitudinal strain [24].

Figure 8 shows pores on the inner surface of a pipe liner, whether metallic or polymer, acting as a barrier against the inner fluid [25]. The current design assumes that the inner layer, serving as a fluid barrier, remains unaffected by potential matrix breaking in the outer pipe layer [8]. Pore formation in protective coatings can result from various factors, including soil conditions like moisture, temperature, pH, salt concentration, soil resistivity, soluble ions, and the presence of microbes, all influencing the state of buried pipes.

Differential aeration corrosion is a significant concern for underground pipelines due to varying oxygen levels in the soil causing potential differences and current flow [19]. A study by Hassan *et al.,* [26] found pores in the composite material matrix, indicating a ductile fracture behavior. Defects like cracks, pinholes, or voids in the protective coating can allow water penetration, leading to issues like swelling, softening, or loss of adhesion [14]. The research explores various fracture behaviors, including fiber pullout, fiber breakage, and matrix cracking. Unlike studies reporting external pore formation [19,26], this research identified internal pores, suggesting potential differences due to factors like pipe materials manufactured or burial conditions.

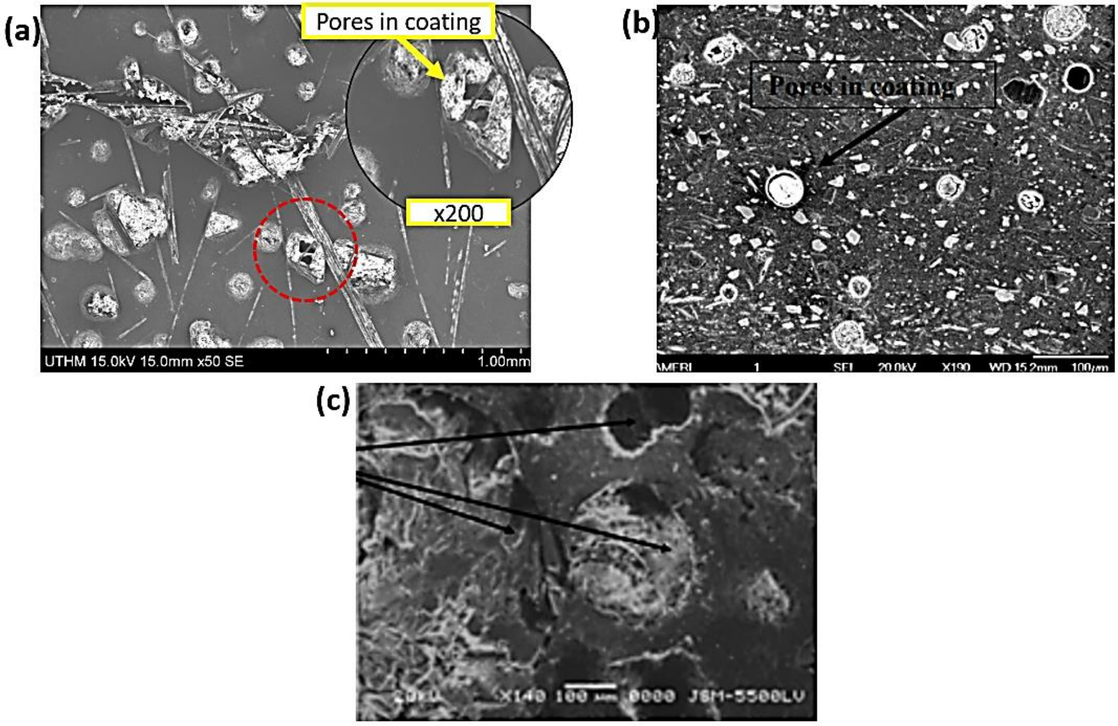

Fig. 8. (a) Pores occurred on the surface of liner; (b) Hybrid epoxy external coating pore [19]; (c) Deep pores in the external matrix [26]

Figure 9 shows a flexural failure with delamination cracks in the structural layer, as seen in the cross-sectional view in Figure 9(a). The study indicates that delamination in the GRE pipe under investigation is due to the applied tensile load surpassing the material's interlaminar strength [27].

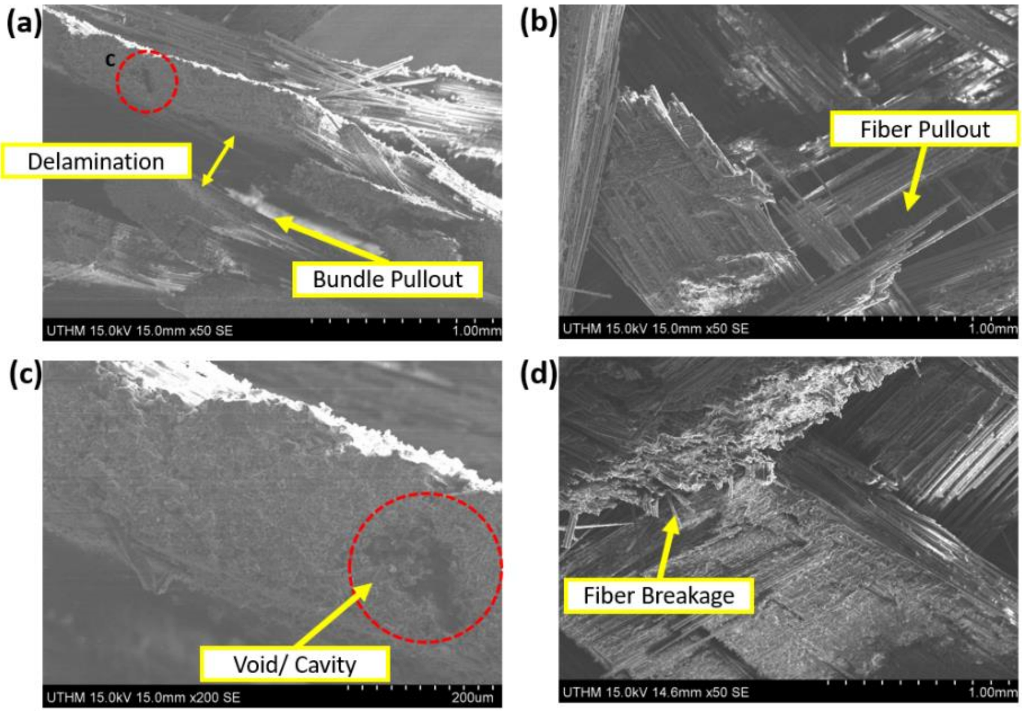

Fig. 9. (a) Cross section SEM; (b) Outer surface SEM; (c) Magnification cross section SEM; (d) Inner surface SEM

Localized porosity and insufficient resin filling lead to the formation of cavities and cracks at the fiber/resin contact [5]. At the failure section, Figure 9(c), the glass fiber layer displays brittle fracture characteristics, with pulled-out fibers creating holes. The fibers, once pulled out, show minimal resin covering, indicating a weak bond with the resin [5]. Failure primarily occurs at the fiber-resin interface, resulting in layer separation and a rough surface.

Factors such as manufacturing defects, impact damage, and fatigue can make the applied tensile load exceed the material's interlaminar strength. Voids or inclusions in the material can create stress concentrations and trigger layer separation. When force is applied, stress concentrates at the fiber-matrix interface, potentially causing fiber pullout. In a composite, when stress is applied, it is distributed among the bundled fibers. If the stress at the interface is too much for the matrix, the fibers may collectively pull out because they are mechanically connected, sharing the load.

In Figure 9(b), compression results in a failure mode characterized by fiber pullout, indicating a weak bond between fibers and the matrix. This impacts the composite material's mechanical properties by reducing stiffness and strength [27]. Fiber pullout can initiate further damage, as separated fibers create stress concentrations, contributing to crack propagation.

In Figure 9(d), tension results in the failure mode of fiber breakage, characterized by complete fracture of fibers due to the applied tensile load. In the GRE pipe during the tensile test, fiber breakage occurs when the load exceeds the ultimate strength of the fibers [27].

Figure 10 shows the results of an impact test. In Figure 10(a), fiber pull-out is observed, where the fiber separates from the matrix without breaking. This can happen due to weak bonding or the presence of voids or defects in the composite structure, as seen in Figure 10(b). Figure 10(c) depicts fiber/matrix debonding, where the fiber separates from the matrix due to bonding interface failure. This can occur due to inadequate adhesive bonding or the presence of contaminants or defects at the bonding interface. Figure 10(c) shows fibers detaching from the matrix due to moisture presence, caused by moisture-induced porosity and voids, leading to reduced joint strength [28].

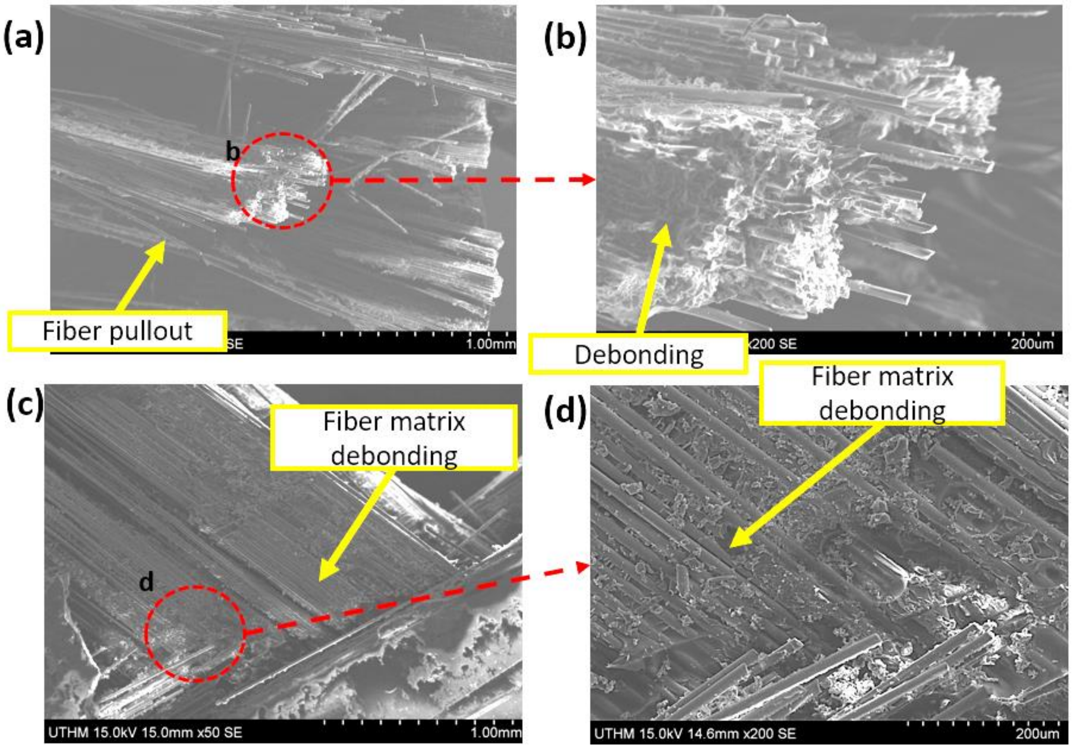

Fig. 10. (a) Cross section SEM; (b) Magnification cross section; (c) Outer surface SEM; (d) Magnification outer surface

To summarize this section data/results/investigation:

(a) Backfill investigations
- Microscopic examination showed a 100% epoxy resin barrier on the external surface of the GRE pipe.
- On-site inspection revealed backfill contains foreign material with non-metallic elbows, wood, and metal pieces.
- Soil observations indicated Class I backfill soil stiffness.

(b) Microstructure analysis on failure modes
- Figure 5(c): Undamaged furthest to failure (presence of microcracks observed)
- Figure 5(d): Damaged furthest to failure (more microcracks observed compared to Figure 5(c))
- Figure 5(e): Damaged closer to failure (presence of microcracks and fiber damage)
- Figure 5(f): Damaged at the failure point (presence of microcracks, fiber damage, and matrix degradation)
- Microcracks are visible in Figure 6(b) at the point of adhesive failure.
- Adhesive failure (Figure 6) was observed at the point of fiber-matrix bonding.
- Whitening observed during tensile testing (Figure 7) indicates early failure and potential delamination.
- SEM analysis of tested pipes revealed fiber breakage, delamination, and pull-out failures

(c) Pore occurrence
- Figure 8 shows pores on the inner surface of a pipe liner, regardless of whether it's metallic or polymeric.

- Unlike previous studies, this research identified pores on the inner surface, potential due to burial conditions (Figure 8(b), Figure 8(c)).
- After mechanical testing observation
- Delamination cracks (Figure 9) were observed due to the applied tensile load exceeding the material's interlaminar strength.
- Weak bonding between fibers and the matrix can cause fiber pullout (Figure 9(b)) and reduce the composite's strength.
- SEM images (Figure 10) show fiber pull-out, debonding, and detachment due to weak bonding, voids, or moisture presence.

3.2 Energy Dispersive X-ray (EDX)

EDX analysis is essential for understanding the composition of materials. Table 1 displays the percentage of chemical elements at various locations.

Table 1
Percentage of chemical elements at different locations

	Chemical element	Weight %	Atomic %
B	C	69.47	75.19
	O	30.53	24.81
C	C	23.85	34.76
	O	42.15	46.11
	Al	5.13	3.33
	Si	17.17	10.70
	Ca	11.7	5.11
D	C	13.62	20.70
	O	50.76	57.94
	Al	5.76	3.90
	Si	19.81	12.88
	Ca	10.81	4.58

Understanding degradation in GRE over time is crucial. Findings reveal that carbon (C) in GRE pipes comes from the resin matrix, while silicon (Si) comes from glass fibers [5]. Both epoxy resin and glass fibers contribute oxygen (O) elements [29]. While glass fibers are corrosion-resistant, the resin in fiberglass pipes may still degrade due to specific chemicals and environmental factors. Carbon and oxygen are common in many resins, like epoxy [19]. Table 1 shows that specimen B has only C and O elements, indicating no additional composition. As it gets closer to failure, carbon decreases, and oxygen increases. During aging, epoxy resin bonds can break, releasing compounds like CO, CO2, CH4, and CH2O [30].

Locations C and D likely experienced oxidation, with D showing a significant decrease in carbon and an increase in oxygen, aligning with oxidation indications. As it approaches failure, there's a 45.62% decrease in carbon and an 11.62% increase in oxygen at location C compared to B. Similarly, at location D, carbon decreases by 55.85%, and oxygen increases by 20.23% compared to B.

Oxidation of epoxy resin can occur due to heat, oxygen, and moisture exposure, especially in high-moisture soil [2]. Oxidation by-products can degrade the resin, reducing adhesion and mechanical properties. The outer tube surface is more susceptible to oxidative degradation than the inner wall [5]. Swelling from water absorption in internal pores can also downgrade mechanical properties, making epoxy more ductile and compromising structural integrity and load-bearing capacity [31].

Examining the microstructure of GRE pipes reveals that the external surface significantly affects defect initiation and progression. The internal side, used for water transport, is less impacted due to epoxy resin protection and glass fiber reinforcement. On-site inspections underscore the importance of proper backfilling and compaction for stable support. Improperly disposed of foreign materials during installation raises concerns about backfill impact. SEM analysis exposes microcracks, matrix degradation, and fiber damage, influencing the pipe's structural integrity. Microcracks, observed in SEM, can reduce strength and stiffness. EDX analysis indicates changes in chemical composition, suggesting possible degradation. The study highlights the complex interplay of external factors, including soil conditions, drainage, foreign materials, and chemical reactions, contributing to GRE pipe degradation.

4. Conclusions

In conclusion, microstructure analysis, particularly using Scanning Electron Microstructure (SEM), revealed the existence of microcracks, adhesive failures, and various failure modes like fiber pullout, fiber breakage, and matrix cracking. The observed microcracks in the SEM images suggest that the composite material may exhibit brittle behavior. However, occurrences of fiber pullout and matrix cracking suggest a more ductile response in the composite material.

The objective of the study aimed to evaluate the impact of damage on the structural integrity of the GRE pipe. Through detailed mechanical testing and microstructure analysis, it became clear that introducing damage significantly influences the material's response to external forces. Damaged positions showed reduced tensile strength, impact resistance, and flexural strength, along with distinct failure modes like adhesive failures and microcracks.

The observed elongation in the tensile test suggests some ductility. This behavior aligns with typical failure patterns where initial force distribution might be influenced by factors like fiber orientation. On the other hand, impact testing reveals a mixed ductile-brittle response. Fiber pullout without breakage and limited debonding suggest potentially weak interfacial bonding between the fiber and matrix.

The flexural test strengthens this concern. The presence of fiber breakage, fiber bundle pullout, and visible voids with minimal resin coverage post-pullout all point to a weak matrix. These combined observations highlight the challenges posed by weak interfacial bonding and matrix strength in maintaining structural integrity under bending stresses.

EDX analysis reinforced the understanding of degradation processes. Specimens at locations C and D exhibited significant changes in carbon and oxygen content, hinting at potential oxidation or degradation processes, consistent with the effects of oxidation on epoxy resin triggered by environmental factors like heat, oxygen, and moisture.

The findings highlight the intricate interplay between behavior, microstructure, and environmental factors in the performance of GRE pipes. The presence of foreign materials in Class I backfills raises concerns about stress concentrations, casting doubt on achieving effective compaction. This mix of factors jeopardizes backfill stability and load uniformity, posing a threat to GRE pipe structural integrity.

Future research could focus on quantifying the extent of degradation and its correlation with specific environmental factors such as heat and moisture over time. Utilizing advanced characterization techniques like X-ray Photoelectron Spectroscopy (XPS) or Fourier-Transform Infrared Spectroscopy (FTIR) could provide deeper insights into chemical changes within the resin matrix. Additionally, developing models that consider damage, fiber orientation, and environmental factors (heat, moisture) and time would be valuable for predicting the performance of GRE pipes.

Testing different backfill materials for their impact on stress distribution and load uniformity around GRE pipes would be equally important for optimizing underground firewater systems.

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