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Fire behaviour of hybrid filament-wound single and adhesively bonded composites tubes under static pressure

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A B S T R A C T

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The present work aims to experimentally investigate the fire behaviour of water-filled E glass reinforced thermoset resin hybrid filament-wound composites tubes under static pressure. Heretofore, fire endurance tests have been conducted on single and adhesively bonded tubes manufactured by CTRA Company. Furthermore, internal pressure tests until failure have been performed on the burnt single and burnt joined tubes in order to quantify their abilities to contain the fluid after being exposed to heat flux. A comparison between the pressure behaviour of exposed to fire (burnt) and non-exposed tubes (single and joined) was also inspected. The identification of the fire-induced damage mechanisms of the tubes was performed through optical microscopy, Scanning Electron Microscopy (SEM) and X-ray tomographic observations. Finally, the thermal analysis was carried-out on burnt specimens in order to better understand the multiphysical phenomenon taking place during the fire endurance tests. The experimental results have revealed that the combustion process of both single and joined tubes was described in four steps namely tube heating, resin degradation, ignition and flame decay. Moreover, it was found that no leakage was witnessed on the tubes (single and joined) outer surfaces during the fire endurance tests. The comparison between the pressure behaviour of the burnt single tube and the burnt joined one has proved that the single tube is much resistant under internal pressure loading than the burnt joined tube. Finally, the fire-induced damage included matrix cracking and delamination between the tube plies which was noticed from microscopic observations.

1. Introduction

Filament-wound Glass Fiber Reinforced Polymer (GFRP) tubes are being used for a wide range of petrochemical and marine applications owing to their strength-to-weight ratio, good durability in sea water in addition to their excellent corrosion resistance and low thermal expansion. These tubes are often required to be adhesively bonded to confirm with industrial specifications. In spite of the numerous advantages of filament-wound composites tubes, a major issue was encountered with respect to the use of these structures, since they perceived which corresponds to their substandard performance in a fire situation. When the composite material made of polymer matrix is exposed to fire, the organic resin decomposes with the release of a large amount of heat,

smoke as well as toxic fumes [1–3] that cause a serious risk to the life of crew members, economy as well as environment. Furthermore, the mechanical properties of the composites structures are rapidly degraded during and after the fire situation.

In this context, with the collaboration of Chaudronnerie Tuyauterie Resin Anticorrosion (CTRA) Company, single and adhesively bonded filament-wound composites tubes have been manufactured for the chemical fluid transportation in the chemical industry and offshore platforms. However, their application remains restricted given the significant gaps in the single and joined filament-wound composites fire behaviour understanding.

The knowledge of fire resistance in filament-wound composite tubes presents a tremendous interest to prevent hazardous effects and any

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damage that may occur during their operation especially bursting. Indeed, the fire resistance of composite material is defined as the specimen's ability to impede the fire spread and maintain mechanical integrity when exposed to heat flux for a predetermined period.

Several research works were performed to investigate the mechanical behaviour of single filament-wound tubes under different loading tests. Some authors were interested in the fatigue behaviour of the GFRP tubes [4–6]. Other works have been carried-out to study their tensile [7, 8] and pressure behaviour [9–12]. Some other works aimed to investigate the mechanical behaviour of adhesively bonded filament-wound tube under tensile [7,13] and pressure [9] loading. Nevertheless, their fire behaviour has not been fully assessed. Thus, almost all the previous studies have been focused on the fire behaviour of composites plates. The fire behaviour studies of composites' materials have been started in 1970 [14–25]. Since then, several works have been devoted to this subject. Some authors were interested in the flammability characteristics of composites' laminates. G.T Egglestone and D. M Turey [2] focused their research on the burning characteristics of glass-reinforced panels with Polyester, Vinylester and Phenolic resin using a cone calorimeter. The obtained results showed that E glass-reinforced Phenolic composites presents a longer ignition time, a lower smoke generation and a reduced heat release in comparison with E glass-reinforced Polyester or Vinylester resin laminates. A.G. Gibson and al [26]. studied the fire behaviour of woven glass laminates using Vinylester and Polyester resins. In their work, it was found that the Vinylester and Polyester laminates ignite after 30s of heat flux exposure. Whereas, the Phenolic laminates ignited after 460s. J.R Brown and al [27]. emphasized the effect of the resin and the glass reinforcement types on the combustion properties of GFRP composite materials. The obtained outcomes showed that the Chopped Strand Mat (CSM) present a lower rate of heat release and lower ignition times beside woven Roving reinforcement. Regarding the matrix, it was found that the Phenolic resin presents higher combustion properties compared to Vinylester and Polyester. Thi Hai Yen Quach and al [28]. investigated the thermal degradation of Carbon fiber/Epoxy filament wound composites' structures with different stacking sequences. It has been proven that the mechanical properties of 90° specimens were rapidly degraded beside $\pm 45^\circ$ samples.

Numerous studies have been completely devoted to investigating the degradation of the composite plates post-fire properties [29–33]. These studies showed that the mechanical characteristics of GFRP composites are temperature-dependent. G.P Gardinier et al. [34] proved that the exposure of glass fiber reinforced polymer composites' plates to a fuel fire induce the reduction of the compressive strength, the flexural strength, and the flexural rigidity. A.P Mouritz and Z. Mathys [35–37] studied the post-fire mechanical properties of glass-reinforced phenolic and vinylester composite laminates exposed to fire in a cone calorimeter. It was noticed that the delamination cracking and char formation were the main fire-induced damage. The delamination depth, as well as the char thickness rose with the increase of the heat flux and the exposure time leading to post-fire flexural compressive and tensile properties reduction. Similar outcomes were found for composites' materials with localized fire damage [38].

Other studies have been undertaken to investigate the thermal barrier effect on the post-fire properties of the composites' materials. U. Sorathia et al. [39] proved that the fire barrier treatments improved the fire performance characteristics of composites' materials. Alfred N. Montestruc et al. [40] studied the intumescent coating and insulation thickness impact on the fire resistance of the dual-wall composite pipe. The experimental outcomes denote that both insulation and intumescent coating enhance the composite fire resistance since they hinder the heat transfer. More recently, Minkook Kim et al. [41] developed a fire retardant glass fabric/carbonized phenolic composite in order to improve the fire resistance as well as the post mechanical properties of glass/Phenolic composite. It has been shown that the post-fire tensile strength of the developed composite (exposed to 1000 °C fire temperature) is 60% higher than the non-treated material.

The fire behaviour of single and adhesively bonded filament wound-composite tubes has been a burdensome topic due to the lack of research carried on that topic. A set of existing standards formulated by organizations like, the International Maritime Organization (IMO) [42] and the American Society for Testing and Materials [43] recommends important steps to carry on fire experimental tests. Indeed, the aforementioned tests in the above standards don't provide any details of the fire behaviour of composites tubes.

Consequently, the present work denotes an insight into the fire behaviour of water-filled E glass-reinforced Vinylester 411 hybrid filament-wound composites tubes. To accomplish this, a series of fire endurance tests have been carried-out on both single and joined composite tubes. In addition, pressure tests until failure have been conducted in order to evaluate the abilities of burnt single and burnt joined tubes to contain the fluid after being exposed to fire. A comparison between the pressure behaviour of the burnt and non-exposed to fire tubes (single and joined) was also made. The fire-induced damages have been identified thanks to post-mortem observations through an optical microscope, Scanning Electron Microscopy (SEM) and X-ray tomographic analysis. Finally, to better understand the chemical and physical phenomena resulting from the tube fire exposure, thermo-gravimetric (TGA) analysis, differential scanning calorimetry (DSC), thermo-mechanical (TMA) and dynamic mechanical (DMA) analysis have been performed on burnt specimens.

The main novelty of this work is coping with (a) water-filled E glass-reinforced thermoset resin hybrid filament-wound composite tubes instead of composite plates commonly analysed under fire test, (b) extend the experimental investigation to cover adhesively bonded structures, (c) and conduct internal pressure tests on burnt structures.

2. Experimental work

2.1. Materials and specimens

The E glass-reinforced Vinylester 411 hybrid tubes were manufactured by CTRA Tunisia Company using filament winding technique. The Vinylester 411 (VE 411) resin referenced under DERAKANE 411–350 Epoxy Vinylester Resin was produced by Ashland America Company. Indeed, the aforementioned resin was frequently used in pipeline fields owing to its low curing temperature (80 °C), better water resistance in addition to its good mechanical properties in a humid environment. The mechanical properties of both Vinylester 411 resin and E glass fibers as provided by the manufacturers are summarized in Table 1. It should be highlighted that the composite tube is made of two types of layers namely anti-corrosion (AC) and mechanical layer (RM) as shown in Fig. 1-c. The first step of the tube manufacturing process consists of the application of anti-corrosion layer following hand layup method around a rotating cylindrical mandrel. This layer subsumes glass fiber mat, laid randomly across each other, impregnated with VE 411 resin. The aforementioned layer, of 2.5 mm thickness, serves as a protection for the tube from corrosion. The second step of the process is to wind a bundle of E glass fiber, impregnated in a Vinylester 411 resin bath around the AC layer at an angle of $\pm 55^\circ$ with respect to the tube axis. This layer, with 2.5 mm thickness, is called mechanical layer and used mainly to withstand the mechanical effort. Following the winding process, the tube fixed on the rotating mandrel, was placed inside an oven to be cured for 2 h at 80 °C (0.6 °C/min). Finally, the tube was assembled to

Table 1
Mechanical properties of E glass fiber and Vinylester resin.

	Tensile strength (MPa)	Tensile modulus (GPa)	Tensile elongation (%)
E glass fiber	1970	78.8	–
Vinylester 411	86	3.2	5–6

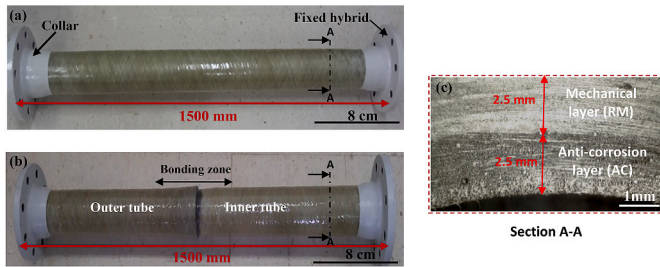


Fig. 1. The filament-wound tube made of E glass/Vinylster 411 resin used for fire endurance tests. With (a) Single tube, (b) Adhesively bonded tube, and (c) The different layers of the tube (cross section).

collars with fixed hybrids (cf. figure1-a).

For the adhesively bonded tube (cf. figure 1-b), composed of an inner and outer tube, it should be mentioned that they were manufactured using the filament-winding technique as described above. For tube bonding, an Araldite 2014-1 adhesive was used. This adhesive, supplied by Huntsman Corporation America, is composed of Epoxy resin and a hardener. The Araldite was applied on the inner surface of the outer tube and bonded with the inner tube. The joined specimens were cured at room temperature for 24 h. It is to be noted that both single and adhesively bonded tubes have an inner diameter of 80 mm and a length of 1500 mm.

2.2. Fire endurance tests

In order to investigate the fire resistance of hybrid filament-wound composite tubes, a series of fire endurance tests were performed on E glass-reinforced Vinylester 411 tubes. Thus, the experimental test was repeated five times in order to evaluate the variability of the experiments. These tests were done following the International Maritime Organization (IMO) resolution A 753 [42]. For this, a device was designed and manufactured especially for this study as witnessed in Fig. 2. The heat source was provided by two rows of five burners. These burners, referenced under Sievert No.2942, have an inner diameter of 29 mm and are connected to two gas cylinders. A pressure sensor of type Swagelock was used in order to record the tube internal pressure during the test. The temperatures of the flame (Thermocouple 1), tube (Thermocouple 2 and 3) and water (Thermocouple 4) were measured continuously along the fire test using four K-type thermocouples (cf. figure 3). These thermocouples were held in place using a device as illustrated in Fig. 2. These thermocouples were connected to a temperature data logger referenced as Testo 177-T4.

The E glass/VE 411 composite tube was completely filled with water. Afterwards, it was placed horizontally on the two V-shaped supports to be exposed to heat flux for 30 min. It is worth noting that the test duration corresponds to the time required for the evacuation of the people when a fire starts. The specimen was maintained at a distance of 125 mm above the burners. The ends of the tube were closed. One of the

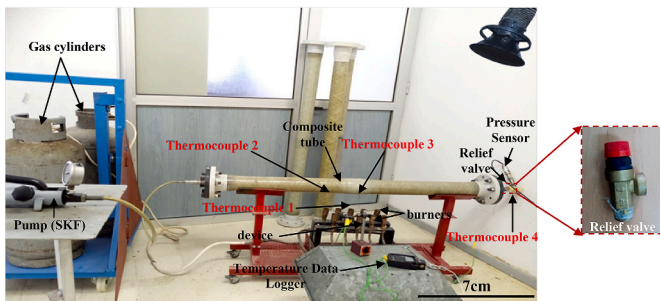


Fig. 2. The experimental device of the fire endurance test.

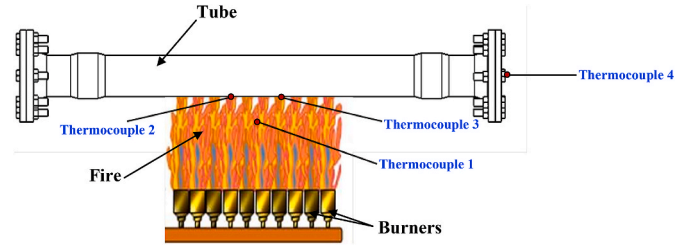


Fig. 3. Schematic view of the tube instrumentation by thermocouples during the fire endurance tests.

ends was connected to a manual pump (SKF). A relief valve was placed on the other end closure to maintain the pressure of the specimen at 3 ± 0.5 bars during the test following IMO resolution instructions.

2.3. Post-fire internal pressure tests

After being exposed to heat flux for 30 min as recommended by IMO resolution A 753, the specimens were cooled to ambient temperature. Subsequently, hydrostatic tests were carried-out on burnt tubes in order to evaluate their ability to contain the fluid. The structure was subjected to its nominal pressure equal to 10 bars following the NF EN ISO 14692-3 standard [23]. It should be highlighted that this pressure should be held for 15 min following the IMO standard instructions. These tests were followed by internal pressure tests until failure in order to quantify the failure pressure of the burnt tube.

2.4. Damage analysis

To identify the fire-induced damage of the E glass-reinforced VE 411 tube, three techniques of characterization were used. Firstly, optical observations were performed through a digital microscope type VHX-1000. Secondly, Scanning Electron Microscope (SEM) referenced as NOVANANOSEM450, was used in order to better understand the damage mechanisms that occur during the fire endurance tests. Finally, a non-destructive technique was performed using an X-ray Micro Tomography Easy Tom 130 machine produced by RX solutions France Company. These observations were conducted in order to quantify the damage mechanisms through the tube thickness. For this, the burnt sample, rotating through 360° , was exposed to the radiation source at a distance of 13 mm. The specimen was scanned for 2 h with 0.01s exposure per projection. The 3D images were reconstructed from 2D images using the Xact 2.0 software.

2.5. Thermal analysis

Along the fire exposure of E glass-reinforced Vinylester 411 composite tube, different multiphysical phenomena have taken place. In order to further understand them, in addition to the aforementioned microscopic techniques, different tests namely DSC, TGA, DMA and TMA were conducted on the burnt specimens. It should be highlighted that, all these tests were repeated 5 times.

2.5.1. DSC and TGA test

In order to inspect the chemical changes in the E glass-reinforced VE 411 composite tube with respect to temperature, differential scanning calorimetry DSC (heat generation rate), and thermo-gravimetric analyses TGA (mass losses) were conducted. The specimen weight loss was measured using TGA instrument type TA. Regarding the heat generation rates, they were performed by means of DSC tests equipment type TA instrument Q100. For both tests, the specimens were heated from 0°C to 450°C at 5°C in Nitrogenous (N_2) environment.

2.5.2. TMA test

To investigate the dimensional stability of the E glass/VE 411 composite with respect to temperature, thermo-mechanical analysis (TMA) tests were carried-out using TMA 402 F³ Netzsch equipment. In fact, during the test a constant force of 0.2 N was applied to the 5 mm thick specimen. The temperature was uniformly increased from 22 °C to 430 °C at 5 °C/min. The measurements were conducted in a Nitrogen (N₂) environment.

2.5.3. DMA test

In order to study the evolution of mechanical properties of the E glass/VE 411 tubes in function of the temperature, a dynamic mechanical analysis (DMA) was conducted on non-exposed to fire and burnt specimens using a DMA equipment of type Metravib. The tube was cut into specimens of 30 mm in length, 10 mm in width and 5 mm in thickness. The measurements were performed at temperatures ranging from 25 °C to 400 °C with a rise rate of 5 °C/min and at a frequency of 0.1 Hz in a Nitrogenous (N₂) environment. The tensile test was carried-out with a dynamic displacement of about 5.10^{-6} m.

3. Results and discussion

3.1. Fire endurance and internal pressure until failure tests

3.1.1. Single tube

The temperature/time profiles of the flame, tube, and water recorded by the different thermocouples along the fire endurance test are illustrated in Fig. 4. From the measurement recorded by the thermocouple 1 (cf. figure 4-a), it can be noticed that the flame temperature increases abruptly after 100s to reach 1000 °C. This temperature was steadily maintained during the test as recommended by the IMO resolution A 753. Moreover, it can be mentioned that, the temperatures measured by the thermocouples 2 and 3, which represent the temperature of the tube's outer surface exposed directly to fire, are similar and the order of level is slightly lower to the source temperature (cf. figure 4 b). Based on the water temperature measurement recorded by the thermocouple 4, two phases can be distinguished (cf. figure 4-c). The first phase is characterized by a slight variation of the water temperature (from 26 °C to 30 °C). However, the second phase is illustrated by an abrupt temperature augmentation from 30 °C to 78 °C. This augmentation may be related to the reduction in tube thickness as a result of the resin degradation during the test. As the temperature rises as function of time, the resin was degraded leading to the tube thickness decrease. Consequently, the thermal resistance of the tube increases and a higher water

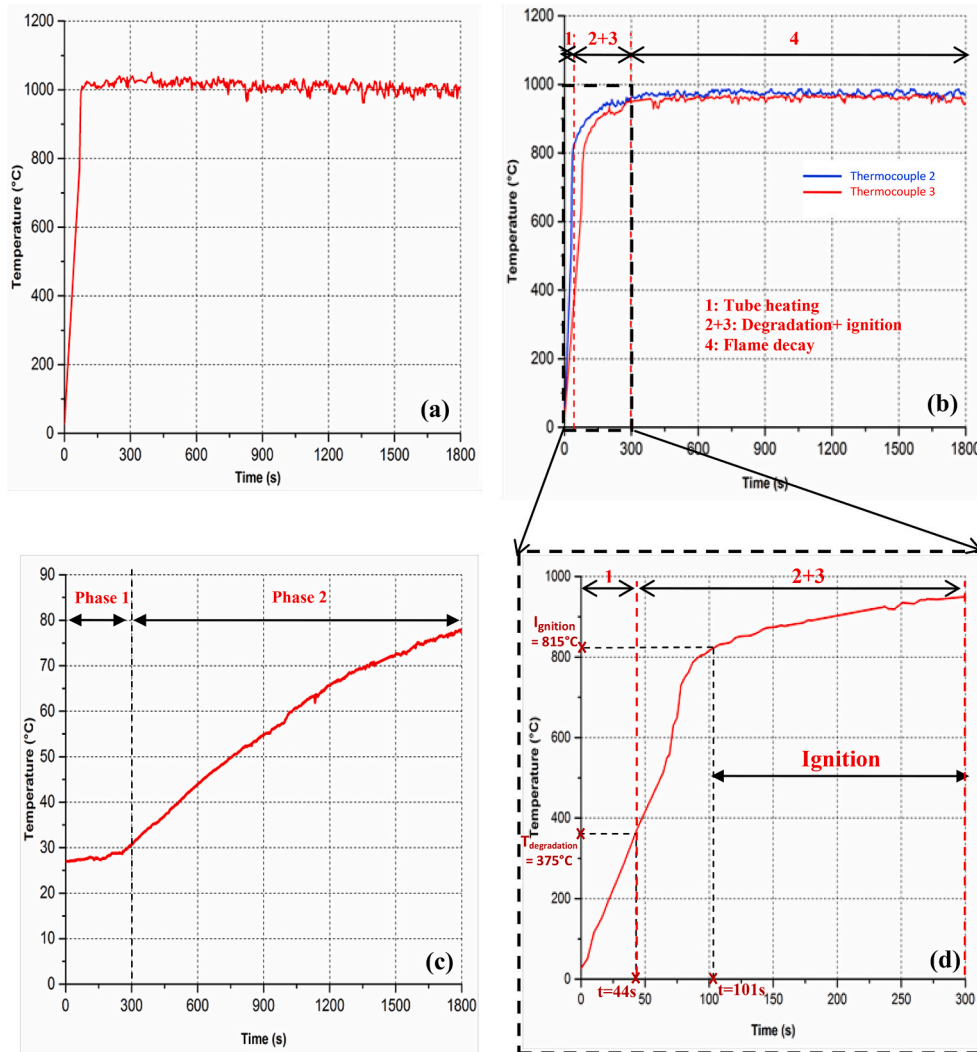


Fig. 4. The temperature-time profiles along the fire endurance test. With (a) The flame, (b) The tube outer surface, (c) The water inside the tube and (d) Zoom of the highlighted zone on figure (b).

temperature is recorded.

Based on visual observations, during the fire endurance tests, it was found that the Vinylester 411 resin on the tube surface which was exposed directly to the fire source was thermally degraded after 44s. This thermal degradation was observed by the color change of the tube's outer surface in contact with the flame, resulting from the apparition of a black layer named char (Fig. 5-b). At this time, the temperature recorded with thermocouples 2 and 3 was around 375 °C (Fig. 4-d). This can be explained by the fact that the resin decomposition temperature was reached. This phenomenon was accompanied by volatile gases and smoke generation.

Subsequently, after 101s, the fire grew leading to the tube ignition (Fig. 5-c). This is due to the gas released from Vinylester 411 resin. The gas release rate became sufficient that the air and the gas were flammable. The measured ignition temperature is of about 815 °C. The ignition process was maintained for 200s as illustrated in Fig. 4-d. It is worth noting that as long as the energy amount transferred to the material is sufficient, the ignition phenomenon is maintained, leading to the release of combustibles gases. Referring to results reported in Ref. [26], it has been confirmed that the ignition time of the Vinylester 411 resin ($t_{\text{ignition}} = 101\text{s}$) is shorter than the Phenolic one ($t_{\text{ignition}} = 460\text{s}$) and longer the one characterizing the Polyester ($t_{\text{ignition}} = 30\text{s}$).

After about 300s, it was found that the flame began to go down and weaken as shown in Fig. 5-d. This is basically due to the char formation. Indeed, the char acts as a thermal barrier to delay the heat transfer between the tube surface exposed directly to fire and the virgin material. Consequently, the pyrolysis rate decreases. Fig. 5-e reveals that the glass fibers remain intact following the fire test as their decomposition temperature was not reached, whereas the VE411 resin degraded. This visual inspection can confirm the fact that the water temperature increases

due to resin degradation as mentioned above.

In order to identify the fire-induced damage of the E glass/VE 411 composite tube after being exposed to a heat source during 30 min, optical observations were firstly carried-out.

Fig. 6 illustrates the optical images of the burnt composite tube. The postmortem observations, performed on zone B which is highlighted in Fig. 5-d, reveal that the resin (of the RM layer) on the surface exposed directly to fire was thermally degraded to finally form a solid carbonaceous char which appears as a black layer (Fig. 6-a). This optical investigation confirms the increase of the water temperature inside the tube as witnessed in Fig. 4-c. Nevertheless, it is noticed that the E glass fibers remained intact as the temperature reached is not enough to engender their melting which occurs at approximately 1100 °C [41]. Furthermore, the optical observations confirm that the AC layer remains unaffected by fire (Fig. 6-b). This result can be explained by the fact that the composite material has a low thermal conductivity. Fig. 6-b illustrates the delamination phenomenon between whether the RM layers or, AC and RM layers. This phenomenon is obviously related to the difference in thermal expansion between the hot and the cold surfaces.

For a better understanding of the damage mechanisms resulting from the fire endurance tests, SEM observations were conducted. The post-mortem observations reveal that the VE411 resin close to the heat-exposed surface was completely consumed after 30 min (Fig. 7-a), leaving a material composed only of glass fibers coated with a thin layer of resin as witnessed in Fig. 7-b. Moreover, the SEM images depict the presence of char resulting from the resin decomposition process taking place at a temperature of 375 °C (Fig. 7-c). Below the charred region, the delamination phenomenon between the tube plies was observed (Fig. 7-d). Chang [44] shows that the composite material has a low thermal conductivity. Consequently, a very steep temperature gradient was

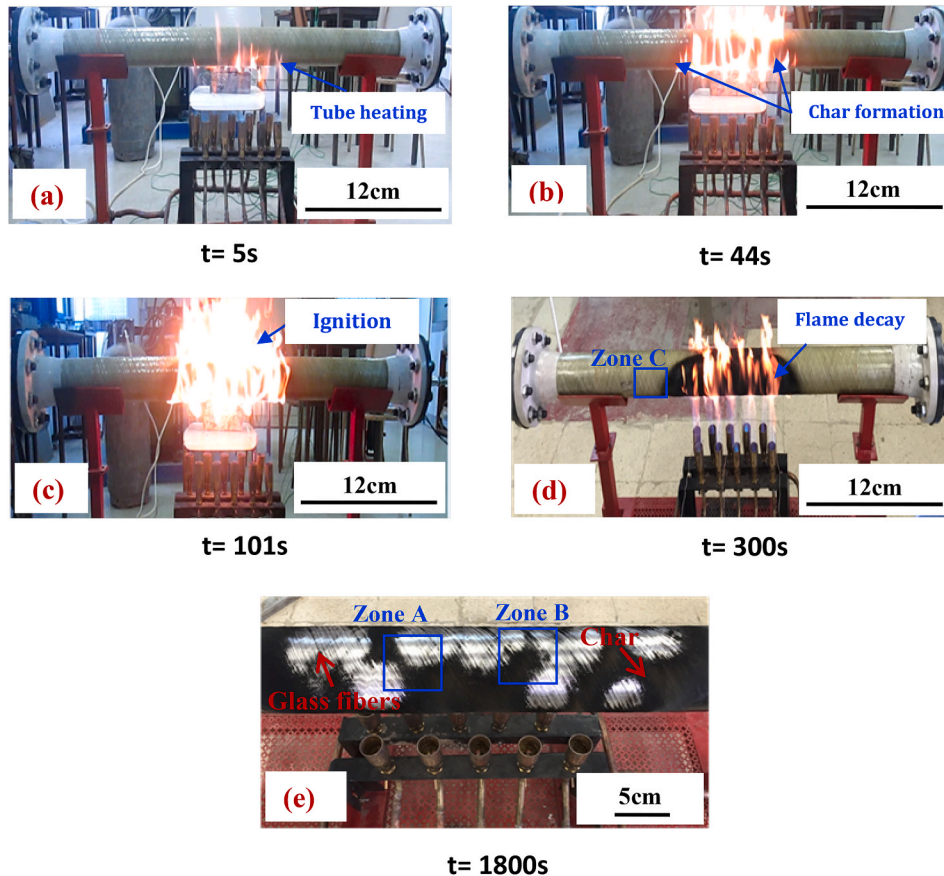


Fig. 5. The combustion process of E glass/Vinylester 411 composite tube exposed to fire during 30 min. With (a) Tube heating, (b) Degradation, (c) Ignition, (d) Flame decay, (e) The tube surface exposed directly to fire.

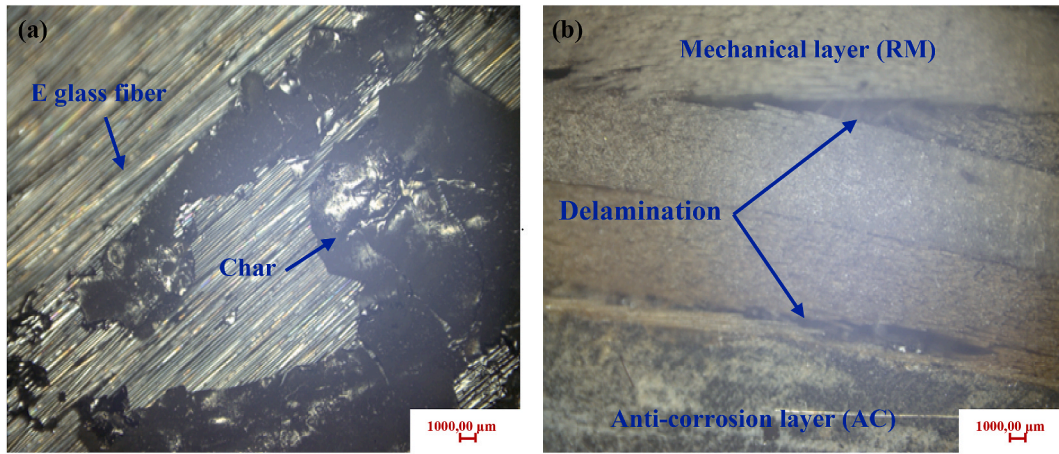


Fig. 6. Optical observations of burnt E glass/Vinylester 411 composite tube (zone B highlighted in Fig. 5-d). With (a) Char formation in the tube surface exposed directly to fire, and (b) Delamination (cross section).

established at the front surface. The author suggests that the delamination phenomenon is associated with the difference in thermal expansion between the fire exposed surface and the underlying virgin material which is much cooler. The zone close to the cold surface remains unaffected by fire owing to the fact that the temperature is too low to cause damage. Regarding the unexposed surface (zone c highlighted in Fig. 5-c), it was noticed that matrix cracking has been the dominant damage mechanism as depicted in Fig. 7-e. This can be explained by the fact that the surface temperature is not enough to induce the VE411 resin decomposition process.

To further understand the fire-induced damage through the composite tube thickness, a non-destructive x-ray tomography technique was carried-out. Fig. 8 illustrates the X-ray tomographic images of the E glass/VE411 composite tube subsequent to the fire test. These post-mortem observations clearly show the resin amount variation through the thickness direction. From Fig. 8-b, it can be observed that the tube surface exposed directly to fire is the first zone to decompose to char at a temperature of 375 °C as illustrated in Fig. 4-d. Albeit, it was found that below this zone, the matrix is partially degraded. Moreover, the delamination phenomenon either between the tube plies (Fig. 8-c) or the anti-corrosion and the mechanical layer (Fig. 8-e) was detected. It is important to mention that compared to other failure mechanisms such as char formation and matrix cracking, the delamination phenomenon is dominating. Referring to Fig. 8-d, the presence of the pores in the resin layer was noticed. This can be explained by the loss of mass of the thermoset resin resulting from the pyrolysis phenomenon taking place after 44s as it was differed from visual observations (Fig. 5-b). This result is in accordance with TGA outcomes.

After being cooled to room temperature, the burnt tubes were subjected to internal pressure tests until failure in order to quantify their functional (leakage) and structural (burst) failure pressures following NF EN ISO 14692-3 standard [23]. Indeed, functional failure takes place when the tube can't contain the fluid anymore. For the structural failure, it is defined as the loss of strength subsequent to the tube burst [45]. Furthermore, hydrostatic tests were performed to assess their ability to contain the fluid after being exposed to fire for 30 min.

Fig. 9-a illustrates the pressure evolution of the E glass/VE411 composite tube during the internal pressure test until failure. From this figure, it can be witnessed that the pressure increases gradually until reaching a value of 79 bars. At this level, water leakage on the burnt tube outer wall was observed (cf. figure 9-b). It can be inferred that the leakage occurrence is mainly related to the growth of cracks caused by fire. These cracks appear mainly due to the delamination taking place between the different layers of the tube. These cracks propagate as the internal pressure rises inside the tube. Consequently, the leakage takes

place on the tube surface exposed directly to fire. The continuous increase of the pressure leads to the tube burst. The burnt tube lost its strength for an applied pressure of about 92 bars as witnessed in Fig. 9-c. This burst results from the matrix and fiber failure (cf. figure 9-c). Beyond the maximum reached pressure (92 bars), it can be clearly seen that the tube pressure decay.

Regarding the hydrostatic test, the burnt composite tube was subjected to its nominal pressure of about 10 bars. Following the IMO resolution and NF EN ISO 14692-2 standard instructions, this pressure should be held for 15 min as illustrated in Fig. 10. During these 15 min, no leakage was detected on the burnt tube outer surface even with the various types of localized fire damage. This result is in accordance with the internal pressure tests until leakage results presented above ($P_{leakage} = 79$ bars). From this experimental inspection, it can be inferred that the E glass/Vinylester 411 composites' tubes are able to contain the liquid in the fire situation for 30 min. Moreover, they are classified under the EA category for the NF EN ISO 14692-2 standard. The EA category confirms that the tubes are working in the safe zone ensuring protection for the offshore staff in extreme accident such as fire, and allowing enough time for their evacuation (referring to IMO standard).

In order to better understand the pressure behaviour of the burnt tube, a comparison with Ben Khalifa's work [46] is conducted. Indeed, Ben khalifa and al [46] have investigated the pressure behaviour until failure of filament wound single tube. It is important to mention that tubes used in this work and the ones used in Ref. [46] were manufactured within the same resin and reinforcement ratio. Furthermore, they are characterized with the same thickness of RM (2.5 mm) and Ac (2.5 mm) layers as well as the same mechanical and physical properties.

The pressure behaviour until leakage of the non-exposed to fire and the burnt single tube subjected to internal pressure are depicted in Fig. 11. A significant difference between their functional pressures is perceived. It was found that the leakage pressure of the non-exposed tube is estimated at about 348 bars. Obviously, the functional pressure of the non-exposed tube is around 441% higher beside the burnt one. Hence, it can be inferred that the mechanical properties of E glass/VE411 tubes have been decaying after being exposed to fire.

3.1.2. Adhesively bonded tube

The fire behaviour of the E glass/VE411 adhesively bonded tube was also investigated under the same experimental conditions of the single tube. It should be mentioned that the combustion process of the adhesively bonded tube is similar to the single one as described in section 3.1.1.

Fig. 12 illustrates the adhesively bonded tube subsequent to the fire endurance test. From this figure, it was observed that the VE411 resin at

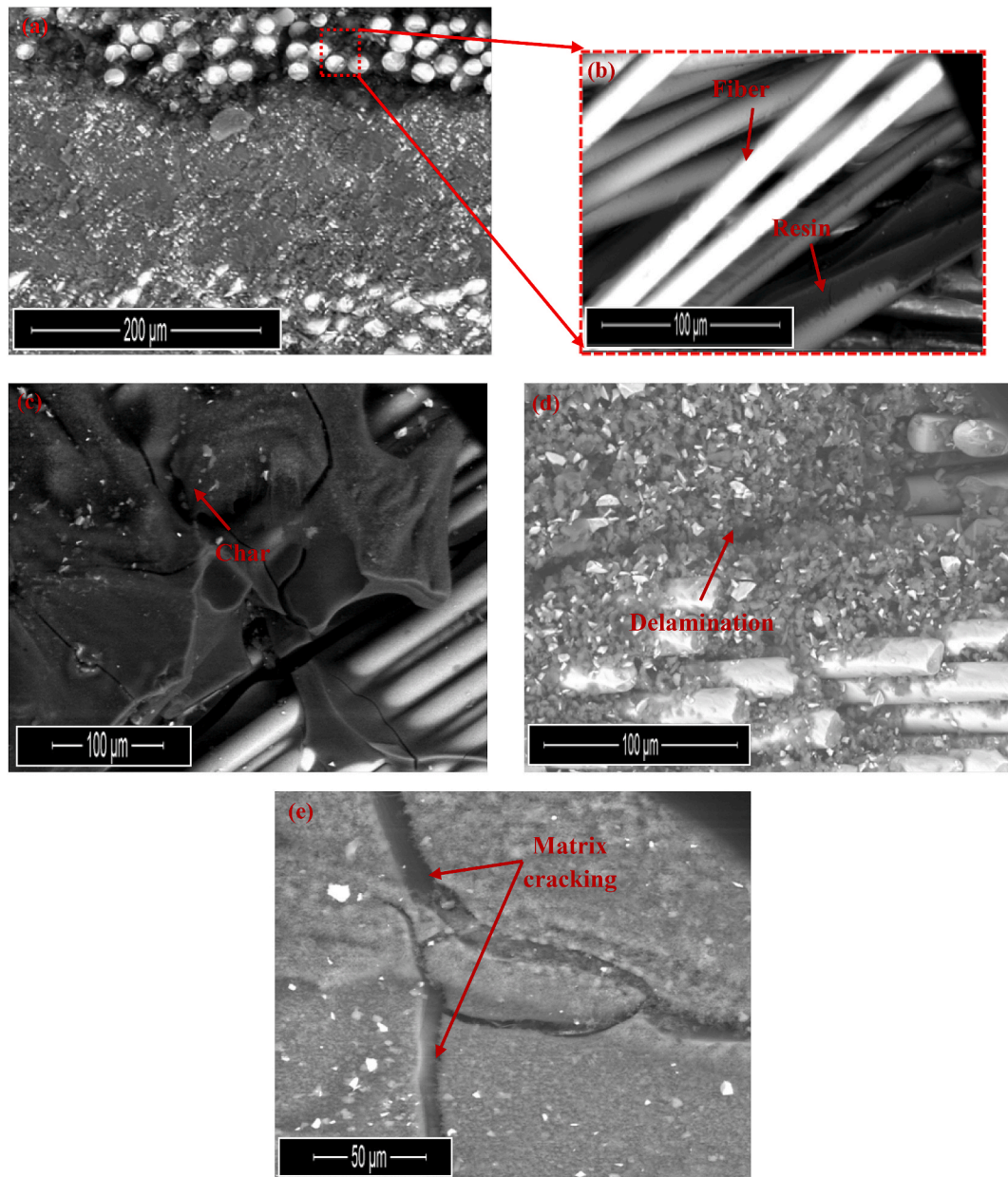


Fig. 7. SEM observations of E glass/VE 411 resin tube after being exposed to fire. With (a) Resin decomposition (b) Zoom of the highlighted zone (c) Char formation (d) Delamination, and (e) Matrix cracking (zone c highlighted in Fig. 5-c).

the surface exposed directly to fire was degraded with char formation, in addition to the apparition of cracks in the Araldite adhesive. It is important to mention that no leakage was observed during the fire test. It can be then inferred that the E glass-reinforced VE411 joined tube can also contain the liquid in fire scenarios.

The pressure evolution of the burnt single and burnt joined tubes made of E glass-reinforced VE411 resin, during the internal pressure test until failure is shown in Fig. 13. The comparison between the two curves shows that the burnt single tube is more resistant under internal pressure loading than the joined burnt tube. This result can be mainly attributed to the fact that the fire-induced cracks in the bonding zone start to propagate before in the tube plies. The authors in Ref. [9] proved, through the X-ray tomographic technique, that the leakage produced in the non-exposed to fire joined E glass/VE 411 tube was the result of the bonding interface failure.

In order to better understand the pressure behaviour of the burnt joined tube, a comparison with a non-exposed to fire adhesively bonded

tube [47] was conducted. The experimental results of both tubes are illustrated in Fig. 14-a. From this figure, it can be noticed that a sharp contrast in their functional pressures is observed. The non-exposed tube has a functional pressure of around 995% higher compared to the burnt one. Furthermore, it is important to mention that the leakage in the burnt joined tube was detected in the bonding zone (cf. figure 14-b). This leakage was the result of the propagation of fire-induced cracks in the adhesive layer. Nevertheless, the authors in the previous work [47] suggest that the leakage formation on the non-exposed to fire joined tube is caused by the initiation and propagation of cracks on the adhesive layer, which confirms this difference in their functional pressures.

3.2. Thermal analyses

3.2.1. DSC and TGA

The chemical changes of the E glass-reinforced Vinylester 411 composite with respect to temperature were inspected from the heat

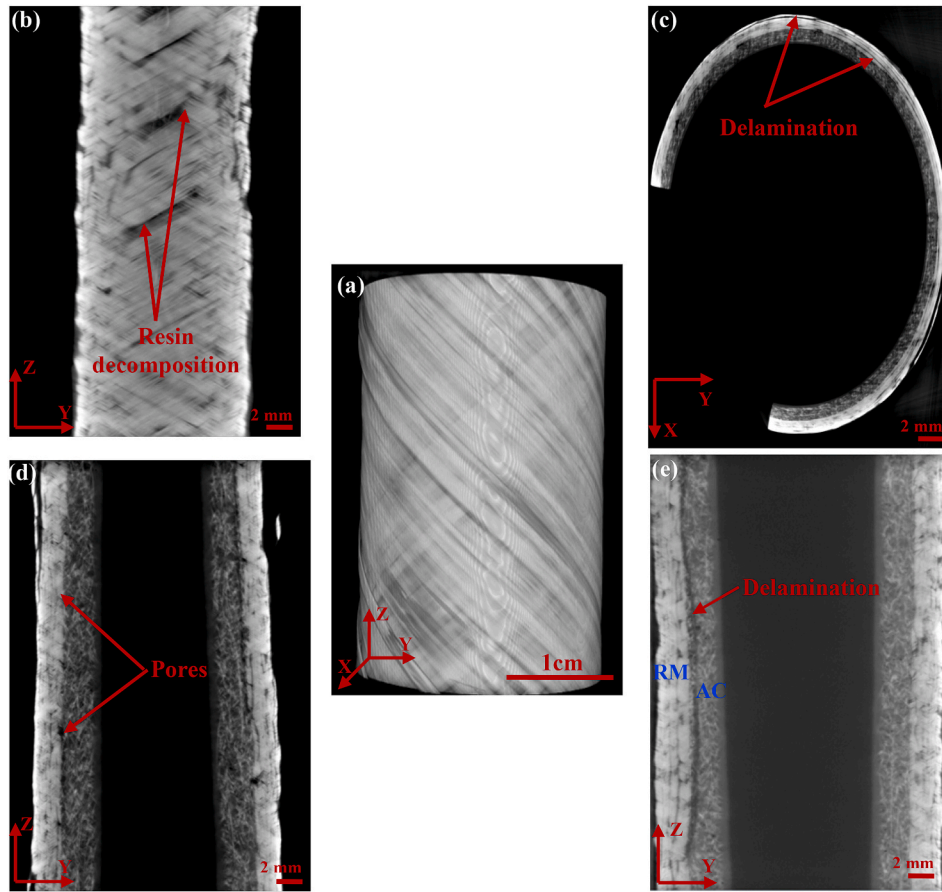


Fig. 8. X-ray tomographic images of burnt E glass/Vinylester 411 composite tube showing the fire induced damage. With (a) 3D image, (b) Resin decomposition, (c) Delamination between the tube plies, (d) Pores, and (e) Delamination between AC and RC plies.

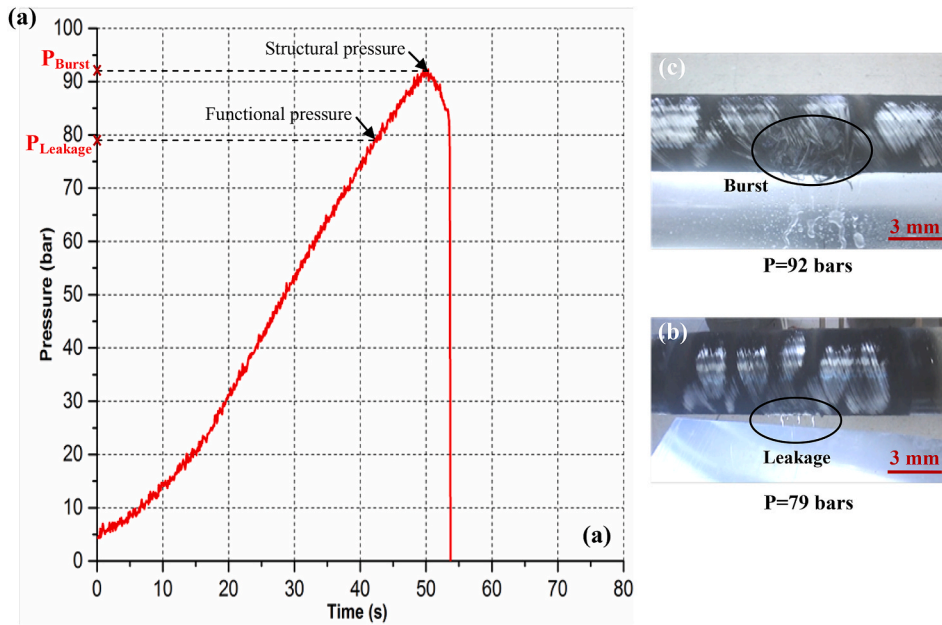


Fig. 9. Internal pressure test until failure. With (a) Pressure-time response of the burnt composite tube under pressure loading, (b) Tube leakage and (c) Tube burst.

generation rate as well as the weight losses. The results of DSC (heat flow) and TGA (weight losses) are depicted in Fig. 15.

It has been proved through experimental outcomes that the chemical changes of GFRP are present in two zones namely volatilization

[78°C–367 °C] and decomposition [367°C–430 °C] as illustrated in Fig. 15. It has been found that the VE 411 glass transition temperature is equal to 78 °C. Beyond this value, it is worth noting a small weight loss of the specimen. This mass decrease can mainly be attributed to the

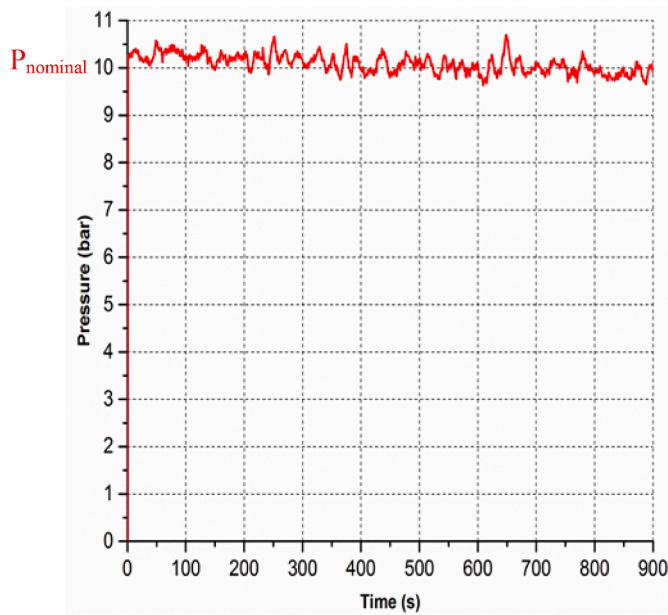


Fig. 10. Hydrostatic test conducted on E glass/Vinylester composite tube after the fire endurance test.

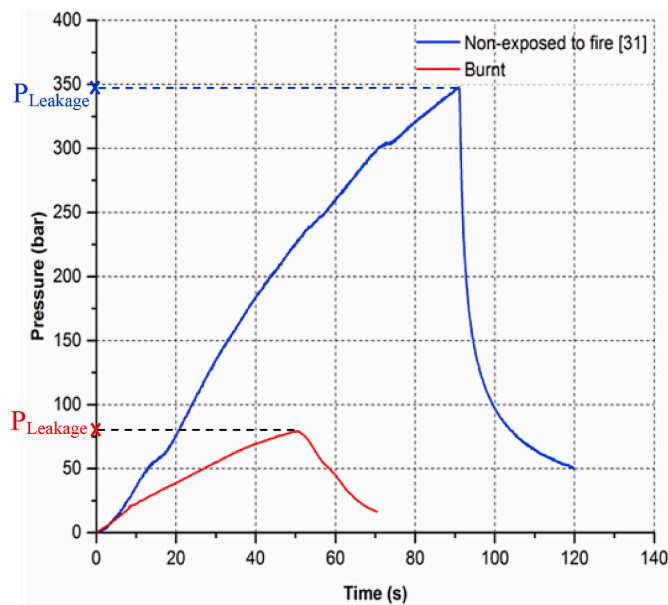


Fig. 11. Comparison between the pressure evolution of the non-exposed to fire and the burnt single composite tube made of E glass fiber reinforced VE411 resin.

volatilization of moisture. Moreover, it can be concluded that the Vinylester 411 resin has a higher glass transition temperature compared to the Polyester ($T_g = 61^\circ\text{C}$) [49] and lower than the Phenolic one ($T_g = 100^\circ\text{C}$) [48].

At a temperature of 367°C , the thermal gravimetric analysis (TGA) has outlined a significant mass loss of the tested sample. This temperature corresponds to the VE411 resin degradation temperature (T_d). This result corroborates well with the resin degradation temperature detected with visual observations during the fire endurance tests. Compared to Ref. [26] outcomes, it can be inferred that the Vinylester 411 resin degradation temperature ($T_d = 367^\circ\text{C}$) is slightly higher than the Polyester one ($T_d = 350^\circ\text{C}$) and lower than the Phenolic degradation temperature ($T_d = 450^\circ\text{C}$).

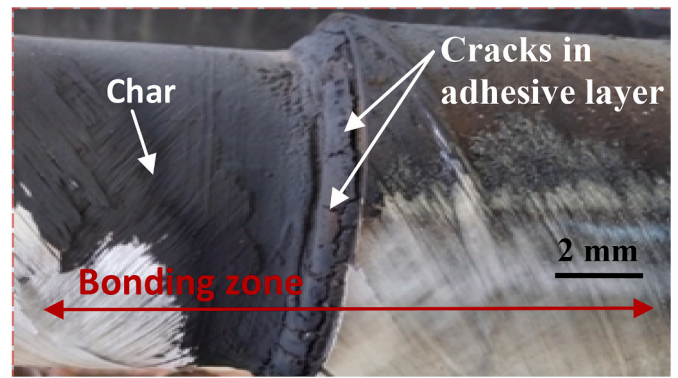


Fig. 12. The adhesively bonded composite tube subsequent to the fire endurance test.

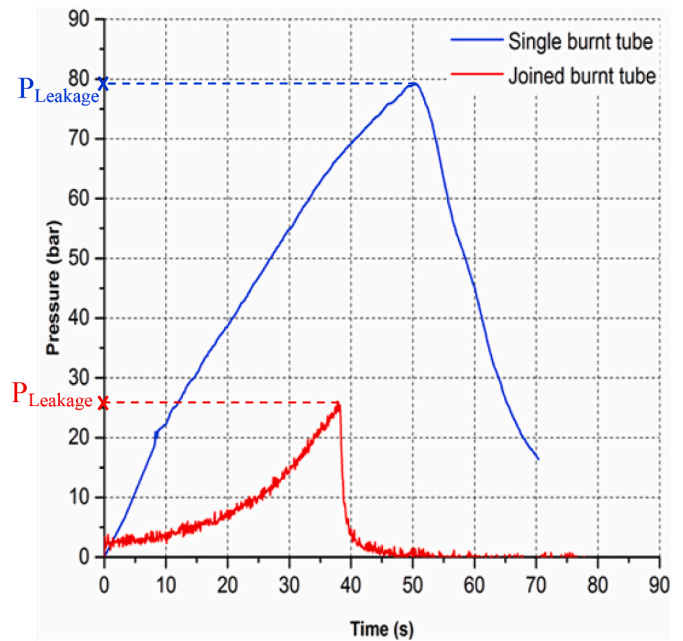


Fig. 13. Comparison between the pressure evolution of the single burnt tube and the joined burnt tube made of E glass/VE411 resin.

A pronounced endothermic peak was recorded at 410°C . In fact, additional heat is needed in order to break the bonds within molecular chains [48].

3.2.2. TMA and DMA tests results

Fig. 16 illustrates the TMA result of the E glass/VE411 composite. This figure reveals that the curve slope increases as the temperature varies from 22°C to 430°C . It is indeed essential to mention that the curve slope presents the thermal expansion coefficient (CTE) of the composite. The $\frac{dl}{l_0}$ versus temperature curve denotes an abrupt change in the curve slope around the glass transition temperature (T_g). It was found that the CTE was $3.810^{-5}^\circ\text{C}^{-1}$ below the T_g and increases to reach $23.410^{-5}^\circ\text{C}^{-1}$ above this temperature.

The increase in the curve slope can be explained by the thermal expansion of the specimen. When the composites are exposed to fire, the pyrolysis phenomenon takes place. It follows that gases are released from the VE411 resin decomposition. As the temperature rises, the generation rate of the gases increase. These gases are mainly absorbed by the composite. Consequently, the internal pressure build-up in the sample is witnessed. Afterwards, the specimen begins to contract at $T = 357^\circ\text{C}$. This phenomenon is attributed to char formation which reacts as

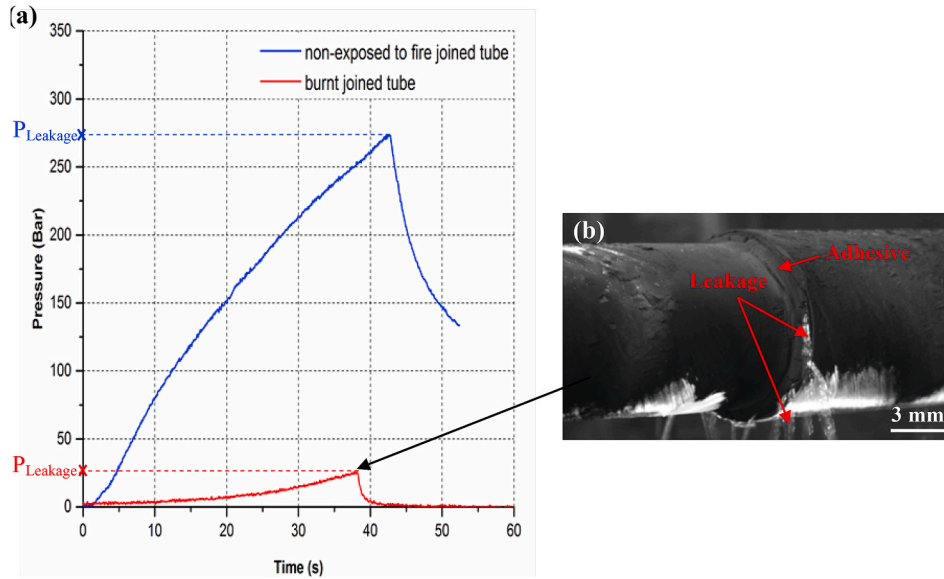


Fig. 14. Internal pressure until leakage tests. With (a) Comparison between the pressure evolution of non-exposed to fire and the burnt joined composite tubes, and (b) The burnt joined tube leakage on the bonding zone.

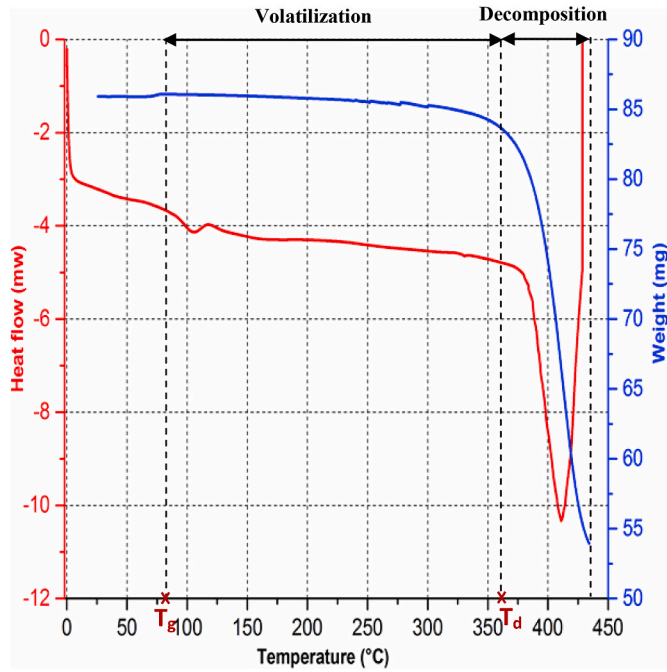


Fig. 15. DSC and TGA test results of E glass reinforced Vinylester 411 composite.

a thermal barrier to delay the heat transfer [49]. Therefore, pyrolysis reduces. This result is in perfect agreement with visual observations during the fire endurance tests, which explains the flame decay phenomenon.

The degradation of the mechanical properties of both non-exposed to fire and burnt composite tube has also been studied by means of DMA tests. Fig. 17 shows the comparison of stiffness evolution with respect to temperature in both non-exposed to fire and the burnt specimens when the temperature increases from 25°C to 400°C. The comparison between the two curves reveals that the stiffness of the non-exposed to fire specimen presents a higher value compared to the burnt tube. The analysis of this curve shows a steep decrease in the stiffness values of both non-exposed to fire and burnt composite samples above the glass

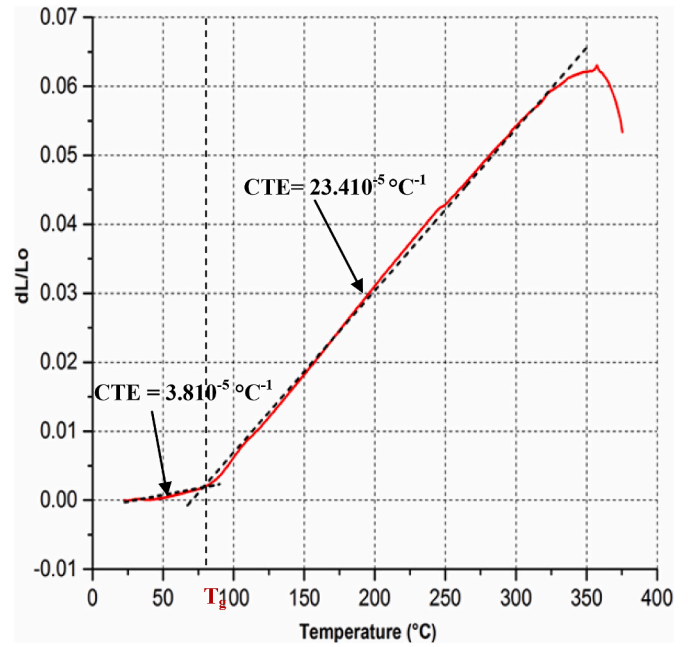


Fig. 16. TMA test result of E glass/Vinylester 411 composite.

transition temperature (T_g). Subsequently, it was noticed that the stiffness is almost constant in the temperature range of 150°C–367°C. Above the degradation temperature ($T = 367^\circ\text{C}$), a rapid decrease in stiffness was observed. The degradation of the mechanical properties with respect to increase in temperature was mainly related to the resin degradation. This result is in accordance with the internal pressure tests until failure, presented in section 3.1.1, which proved that the non-exposed to fire tube can withstand higher pressure than the burnt one.

In summary, it can be inferred, through thermal analyses, that tremendous chemical and physical processes are involved during the fire endurance test of the E glass/VE 411 composite tube as witnessed in Fig. 18. Indeed, when the tube is exposed to fire its temperature rises by conduction until reaching the glass transition temperature ($T_g = 78^\circ\text{C}$). At this stage, moisture volatilization is also noticed. As the temperature

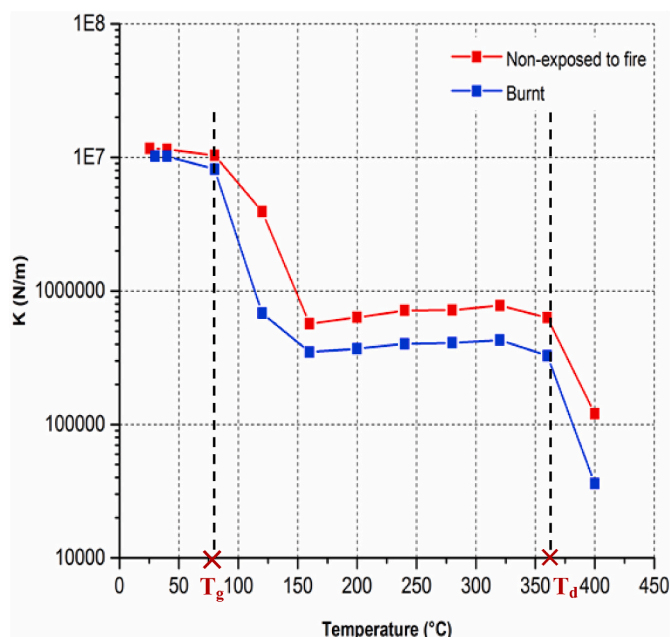


Fig. 17. DMA result of E glass/Vinylester 411 resin composite.

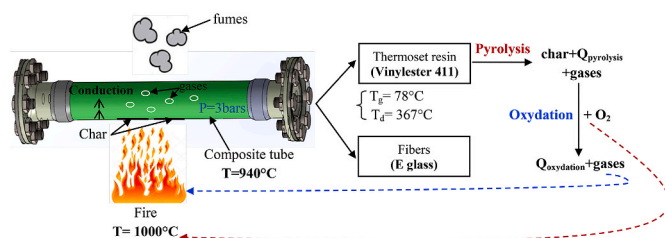


Fig. 18. Chemical processes of E glass/Vinylester 411 tube under fire.

increases, the Vinylester 411 resin begins to decompose at $T_d = 367^\circ\text{C}$ with char formation as well as gases and fumes are released. Consequently, a resin mass loss was recorded. A part of the released gas was absorbed by the material leading to the tube expansion. Moreover, it was proved through DMA tests that the mechanical properties of the tube are degraded.

4. Conclusions

The fire behaviour of the water-filled single and joined hybrid filament-wound composite tubes under static pressure was experimentally investigated. Based on this experimental work the following conclusions can be drawn:

- It was noticed through fire endurance tests that the combustion process of both single and joined E glass-reinforced thermoset resin tube, exposed to fire for 30 min, can be described by the following four steps: tube heating, resin degradation, ignition and flame decay. Optical, SEM and X-ray tomographic observations conducted on specimens, subjected to heat flux during 30 min, denote several fire-induced damages like char formation, matrix cracking and delamination between the tube plies and the anti-corrosion/mechanical layer. These observations have also shown that the E glass fibers remained intact at a temperature of 1000°C .
- The internal pressure tests performed until failure on the single burnt tubes (after 30 min of fire exposure) showed that their leakage pressure is 790% higher beside their nominal pressure recommended by the company. Hence, it can be inferred that the burnt E glass/

Vinylester 411 filament-wound composites tubes are able to contain the fluid during a fire situation.

- The comparison between the pressure behaviour of the non-exposed to fire and the burnt single tubes, reveals that the non-exposed structure can withstand a higher pressure than the burnt one. This comparison outlined that the mechanical properties of the composite tube are degraded during the fire tests. This result was confirmed by DMA tests which show that the mechanical properties of the composite tube were degraded when the temperature increases from 25°C to 400°C . Moreover, the DSC and TGA tests proved that the Vinylester 411 degradation temperature is equal to 367°C . Beyond this temperature, a significant mass loss was recorded resulting from the Vinylester 411 resin degradation.
- The fire endurance tests conducted on E glass/Vinylester 411 adhesively bonded tube assert that no leakage was detected during the whole duration of the fire test. Moreover, the internal pressure tests until failure show that the leakage appears on the bonding zone at a pressure of about 25 bars.
- The comparison between the pressure behaviour of the single burnt and the joined burnt tube show that the single tube is much resistant under internal pressure loading than the adhesively bonded tube because the bonding interface fails before the cracks initiation in the tube plies. This explains the fact that the leakage pressure of the single burnt tube is 292% higher than the joined burnt tube

CRedit authorship contribution statement

Sonia Braiek: Formal analysis, Experimental tests, results analysis. **Ated Ben Khalifa:** Experimental test (fire test). **Redouane Zitoun:** Formal analysis, Writing - review & editing, Contribution to the results analysis, experimental tests (thermal analysis), Reviewing. **Mondher Zidi:** Experimental test (fire test), Supervision. **Mehdi Salem:** reduction, Optical and Microscopic observations.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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