



# Effects of hydrothermal ageing on the behaviour of composite tubes under multiaxial stress ratios



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## ARTICLE INFO

### Article history:

Received 3 February 2016

Accepted 28 March 2016

Available online 31 March 2016

### Keywords:

Glass reinforced epoxy composite pipes

Hydrothermal ageing

Multiaxial cyclic loading

First ply failure

Moisture

## ABSTRACT

The effects of accelerated hydrothermal ageing on the behaviour of composite tubes under multiaxial stress were experimentally investigated. A set of  $[\pm 55^\circ]_4$  tubes were hydrothermally aged at 80 °C for 1500 h. An indigenous automated test rig was fabricated to accommodate five stress ratios—0H:1A, 1H:1A, 2H:1A, 4H:1A, and 1H:0A. The cyclic test involved, pressurising the tube with 1-min pressure and 1-min no-pressure cycles. The first ply failure points were determined from the axial and hoop stresses. Failure envelopes were constructed at the aforesaid five stress ratios. Fourier transform infrared results show an increase in the intensity of absorbance peaks of the OH stretching bands at the inter-phase. The scanning electron microscopy micrographs of aged samples show clear debonding between the epoxy resin and the glass fibres, which is a cause of failure. Moisture uptake by the epoxy leads to matrix osmotic cracking, resulting in damage.

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## 1. Introduction

Composite manufacturers strive to inspect their products especially because failure in their service can have catastrophic effects. Composites are widely used in the oil and gas industry, where many applications involve treated water. Further, considering the long service life of composites, their performance evaluation is extremely important. Against the background of the wide application of composites in humid environments, hydrothermal ageing research for these materials has gained attention. The qualification of glass-reinforced epoxy (GRE) pipes is governed by the international standard, ISO 14692-1:2002 [1], through ASTM D2992 [2]. The ISO 14692 standard procedure involves a regression-based technique with a long-term test. The test requires at least 18 points to plot the regression line and a point in access of 10,000 h, which is approximately 14 months, to estimate the residual properties at the end of the expected life (20–50 years). This standard predicts the behaviour under pure internal pressure, allowing for axial load effects. Hence, there is a demand for a more effective, yet reliable short-term test. Other combined loading conditions should also be considered to determine the long-term behaviour, which may

provide, more a realistic failure envelope. The action of water on composite GRE pipes is known to degrade the pipes. To assess the effects of ageing on the GRE pipes during operational life, an accelerated ageing setup is necessary.

Nevertheless, the long-term behaviour of composites due to ageing has been investigated only in few studies. An ageing review authored by Maxwell et al. [3] provides a detailed guidebook on the ageing mechanisms, standard ageing test methods, and ISO and ASTM standards. The mechanical properties may degrade because of major environmental ageing factors, such as salt water, chemicals, corrosion, moisture, heat, and ultraviolet light, or because of all of these together [4]. A number of works have focused on the hydrothermal ageing discussed herein.

Boubakri et al. [5] studied the ageing effects of thermoplastic polyurethane by immersing it in distilled water at 25, 70, and 95 °C. Through dynamic mechanical thermal analysis, they observed that the mechanical properties degraded with ageing temperature and immersion duration.

Soykok et al. [6] investigated mechanically fastened joints in glass fibre/epoxy composites subjected to hot water immersion at 50, 70, and 90 °C for one and two weeks. The mechanical properties of the joints were affected by water temperature and immersion time. The degradation of tensile and flexural properties because of hydrothermal ageing was studied by Liao et al. [7]. In their study, the pultruded glass fibre-reinforced plastics specimens

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were aged in salt water at room temperature (RT) or in water at 75 °C for various durations.

Hammiche et al. [8] observed the decrease in Young's modulus due to fibre damage after hydrothermal ageing of Alfa fibre-reinforced polyvinylchloride composites. The specimens were hydrothermally aged in distilled water at 100 °C over a period of 120 h.

Dogan and Atas [9] investigated the effects of hygrothermal ageing on the mechanical properties and impact behaviour of glass fibre epoxy composites. They aged the composite specimens at 95 °C and 70% humidity for 1200 h. They chose the conditioning temperature based on the glass transition temperature ( $T_g$ ) of 78 °C. The researchers found that the tensile strength and modulus of elasticity decreased to some extent with ageing time. The Poisson's ratio decreased up to 500 h and remained constant while the compressive strength remained stable.

Zhou and Lucas [10,11] focused on the hydrothermal effects of epoxy resin and epoxy matrix composites. The materials were aged at 45, 60, 75, and 90 °C for 1530 h. The authors observed that the water molecules bonded with epoxy resins through single and multiple hydrogen bonding. A type I bond, which is a single hydrogen bond observed in water, contains less activation energy and can be removed easily from the resin. Type II bonds are formed as a result of water molecules forming multiple hydrogen bonds with the resin network, and such bonds contain higher activation energy and are harder to remove. The formation of type II bonds depends on the higher immersion time and exposure to water. The authors also studied the effects of variations in the glass transition temperature of the epoxy in a hydrothermal environment. They observed that the change in  $T_g$  did not solely depend on the water content absorbed by the epoxy resins. A higher value of  $T_g$  is a result of longer immersion time and higher exposure temperature.

Becky [12] and his co-workers studied the combined effects of load, moisture, and temperature on the properties of E-glass/epoxy composites. They developed a method to evaluate the combined effects of mechanical and environmental conditioning on fibre reinforced polymer composites. The results emphasised that the reduction in modulus was due to the plasticisation of the absorbed water.

Ray [13] conducted a detailed study on the effects of temperature on the shear strength of carbon/epoxy and glass/epoxy composites during hygrothermal ageing. The moisture intake with respect to change in temperature during the ageing process was examined. The study concluded that during degradation, the composite undergoes changes depending not only on the moisture but also on the conditions under which diffusion occurred in the specimen.

The effect of fibre orientation angle on the phenomenon of humidity diffusion was examined by Boukhoulda [14]. Examination of the ageing degradation of solid and laminate composites, when exposed to hydrothermal conditions, showed that the resins mainly absorb moisture; manufacturing defects contribute to the deterioration, and temperature mainly affects moisture absorption.

Ivo and Rayner [15] evaluated the effects of distilled water absorption on nine-layered composite plates with a fibre fraction of 46%. They performed tests to measure the effect of moisture on the static bending strength and fatigue loading.

Assarar [16] and co-authors experimented the effects of water ageing on mechanical properties and damage of flax-fibres and glass fibres. Tests on the aged composites showed that the flax fibre degraded much faster than the glass fibre composites. The authors concluded the weakening of the matrix fibre interface is the major damage mechanism induced by water ageing.

The effects of seawater and impact loading on the fatigue life of glass/epoxy composite pipes under cyclic internal pressure were

investigated by Deniz et al. [17,18]. The pipes produced by a filament-winding technique were immersed in seawater for periods of 3, 6, and 9 months. Impact tests with three different energy levels (5, 7.5, and 10 J) were conducted on the specimens, and fatigue damage such as perspiration, leakage, and eruption were observed. Hawa et al. [19] studied the burst strength and impact behaviour of hydrothermally aged GRE pipes under impact levels of 5, 7.5, and 10 J. The results revealed that the aged and impacted specimens had low burst strength. Environmental effects on the mechanical properties of GRE tubes were studied by Ellyin et al. [20].

Unlike the works discussed above, this study investigated the effects of ageing on the behaviour of composite tubes subjected to an automated short-term cyclic loading test procedure. A hydrothermal ageing setup was developed to age one set of composite pipes. Virgin and aged pipes were subjected to internal pressure at five different stress ratios. Failure envelopes based on first ply failure (FPF) points were constructed for both virgin and aged tubes, and the results were compared. The effects of ageing and the failure modes under each loading condition were determined. The Fourier transform infrared (FTIR) spectroscopic analysis showed an increase in the intensity of absorbance peaks of the OH stretching due to moisture and water intake during ageing. Field emission scanning electron microscopy (FESEM) analysis was performed for both types of samples; a clear debonding between the epoxy resin and glass fibres, leading to failure, was observed.

## 2. Materials and method

The composite pipes used in this experimental work were fabricated at the Advanced Materials Research Centre (AMREC), Kulim, Kedah, Malaysia, a division of the Standards and Industrial Research Institute of Malaysia (SIRIM). The multiaxial pressure test rig was developed and accelerated hydrothermal ageing and cyclic loading tests under various loading conditions were conducted at our Solid Mechanics and Composite Structures Laboratory at University Malaysia Perlis, Perlis, Malaysia.

Composite pipe specimens with  $[\pm 55^\circ]_4$  winding angle were fabricated through the filament-winding process with an internal diameter and length of 100 and 1050 mm, respectively. A winding angle of  $[\pm 55^\circ]_4$  was chosen as it is the optimum winding angle for 2H:1A pure hydrostatic loading according to netting analysis [21,22]. The glass fibres were reinforced with D.E.R. 331 liquid epoxy resin and hardened using Jointmine 903-5s hardener. The pipes were cured at room temperature and post-cured for 3 h at 80 °C in an oven. The physical and mechanical properties of the composite pipes are tabulated in Tables 1 and 2. The mechanical properties predicted using the classical laminate theory and the experimental results are compared in Table 3.

The automated test rig supported multiple axial to hoop stress ratios, as shown in Fig. 1. A pressurisation mechanism was developed to achieve five multiaxial loading conditions, ranging from pure axial to pure hoop. The schematic of the automated pressure test rig is shown in Fig. 2. The mechanism involved fabrication of end caps, piston-like arrangement and serrated wedges to avoid pressure leaking. The development and procedure of achieving five

**Table 1**  
Physical properties of the GRE pipe.

Average wall thickness	mm	4.34
Density	(kg/m <sup>3</sup> )	1150
Number of plies	-	4
Average length	mm	1052
Average weight	g	2058

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