Thermo-mechanical properties of filament wound CFRP vessel under hydraulic and atmospheric fatigue cycling

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ABSTRACT

Mechanical properties of composite pressure vessels under thermo-mechanical conditions were performed by a hydraulic and atmospheric pressure test, respectively. During atmospheric fatigue test, the temperature of the gas and vessel varied remarkably with the pressure, indicating that the vessel was under thermo-mechanical cyclic loadings. Besides, the mechanical properties of the filament wound resin matrix and composition-dependent composites varied significantly throughout the temperature changing range, which could stimulate more damages to the vessel during atmospheric fatigue test than that during hydraulic test characterized by acoustic emission. These damages might lead to a reduction in the final burst pressure of the vessel by 9.6%.

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1. Introduction

Filament wound carbon fiber reinforced polymer composite (CFRP) pressure vessel has been one of the most effective solutions for high-pressure storage [1–3]. To meet practical requirements, some composite pressure vessels used in automotive fields should be at the maximum pressure of 70 MPa [4]. Naturally, the complex safety requirements for high-pressure vessels need to be considered: the fatigue lifetime in particular is one of the critical parameters. To predict the fatigue lifetime of CFRP vessels, hydrostatic fatigue or/and burst tests, which only produced pure internal pressure to the vessels, are widely employed [5]. Nevertheless, in numerous regular custom applications, pressure vessels are directly subjected to the cyclic loading of both high pressure and extreme temperature by gas or fuel charging and discharging processes [6]. It has been reported that the charging of high pressure gas can result in a sharp increase of temperature within vessels due to the released heat of compression and the Joule–Thomson heating of the fuel [7,8]. Moreover, in procession of the gas usage routine and exhaustive deflation, pressure reduction in vessels may lead to obvious temperature drop [9]. The great fluctuation of temperature can lead to the substantial increase in internal thermal stresses [10]. During the vessel lifetime, numerous charging and discharging circular procedures are performed, thus leading to thermo-mechanical fatigue.

There are some approaches to improve the thermo-mechanical properties of CFRP vessel in the previous studies. Onder et al. [11] reported that the burst pressure of CFRP vessels can be depressed at high temperature due to the thermal stresses and the reduced mechanical strengths. Messinger group [12] applied cryogenic and mechanical cycling to a CFRP pressure vessel and damage at a portion of the surface was produced by cryogenic cycling. Experimental works dealing with CFRP laminates behavior also show that failure in vessels occurs more easily under thermal cycling than in mechanical fatigue process [13]. Ju and Morgan [14] conducted a thermal cycling test under mechanical loading on composite, and concluded that the interface strength was degraded at high temperature range of the thermal cycle, leading to debonding that promoted transverse crack growth at the subsequent low temperature range of the thermal cycle. However, the thermo-mechanical behavior of vessels is extremely complex in actual cases. For the further deep understand of the integrated performance of CFRP vessels, it is absolutely necessary to investigate the charging/discharging cycling process by the atmospheric pressure test. However, the atmospheric pressure tests are not commonly performed since testing high-pressure vessel under atmospheric pressure loading is a tedious and costly procedure.

Accordingly, the present work aimed to investigate the thermo-mechanical properties of filament wound CFRP vessels under hydraulic and atmospheric fatigue test. CFRP vessels were fabricated by filament winding, and fatigue pressure tests of the vessels under hydraulic and atmospheric conditions were carried out 100 times at the pressure ranging from 0 to 35 MPa. Mechanical properties of the epoxy resin and carbon fiber/epoxy composites were
evaluated at elevated and cryogenic temperatures. The acoustic emission dates were collected during fatigue tests to study the fracture development of the vessels. The final strengths of CFRP vessel were obtained by blasting the vessels in hydraulic test in order to further clarify bursting characteristics of the vessels under thermo-mechanical fatigue cycling.

2. Materials and methods

2.1. Materials

Toray’s Torayca T700-12k carbon fiber tow were used for the filament winding, with the tensile strength and tensile modulus at 4.9 and 230 GPa, respectively. Diglycidyl 4,5-epoxy tetrahydro phthalate (TDE-85) provided by Bluestar New Chemical Materials Co., Ltd. (epoxy value 0.81) and 4,4’-Methyleneedianiline (DDM) provided by Star Chemicals and Catalysts Co., Ltd. were used as matrix and curing agent.

2.2. Preparation of CFRP vessel samples and specimens

Two vessel samples were fabricated using a winding machine (JNW01.3-60-5/2NC, Jiangnan Co., China). The vessel prototype prepared in this work was composed of an axis-symmetric thin 6061 aluminum alloy inner liner (thickness of 2 mm) with two domes fabricated by a rotary extrusion process. The CFRP vessels were wound by T700 carbon fiber impregnated with the TDE-85/DDM epoxy resin system. The structure of the vessel’s composite was composed of 24 helical patterns and 30 hoop wraps as shown in Fig. 1. After the filament wrapping, the vessels were placed in an oven, and the resin was gelled and cured. The wall thickness of the composite was about 10 mm. The vessel has a diameter of 430 mm and its length was less than 1.3 m from opening to opening. The registered capacity was 130 L.

TDE-85/DDM epoxy resin specimens were cast into the test configurations from bulk resin. A standard dog bone-shaped specimen was employed for all epoxy resin tensile testing. Specimens were 200 mm in length and 10 mm in width in the gage section, and 3.5 mm in thickness. The tensile and interlaminar shear strengths of NOL ring specimens, which were used carbon fiber (CF)/epoxy composites, were prepared by the same filament winding machine with a winding tension of 30 N [15]. Seven groups of epoxy resin and CF/epoxy composite samples were prepared for different testing temperatures. One group of the epoxy resin and CF/epoxy composite samples needed a cryogenic/elevated temperature cycling sample that had been cycled 100 times from −95 to −100 °C was tested at 25 °C.

2.3. Characterizations

2.3.1. Fatigue properties and burst characteristics of composite vessels

The autotretage pressure artificially applied with 44 MPa into two composite vessels induced plastic deformation of the metal liner to make a strong adhesion between composite and metal liner [16]. Additionally, one vessel was tested for hydraulic fatigue with a calibrated hydro-proof pump system 100 times from 0 to 35 MPa. The vessel was pressurized at the rate of 10 MPa/min. The other vessel was carried out atmospheric fatigue pressure test. One cycling pressure ranging was also from 0 to 35 MPa. During the charging process, pressure was applied to vessels by compressed N₂ at the rate of 5 MPa/min. Furthermore, during the discharging process, pressure was reduced at the rate of 10 MPa/min. Pressure was controlled by a computer. Three thermocouples were employed to check the temperature changes for the fatigue test, which can measure the temperature of gas inside the vessel (No. 1), the cylindrical body surface (No. 2), and the surface of dome area (No. 3), respectively, as shown in Fig. 2. Finally, the two vessels, which had undergone the fatigue test, were loaded with hydraulic pressure to burst pressure by using a loading rate of 10 MPa/min.

2.3.2. Mechanical properties of epoxy resin and CF/epoxy composites

In this study, an environmental test chamber (Instron 3119-407) was used to set up the cryogenic and elevated temperature measurement, and an Instron 1196 universal test machine was used to evaluate the mechanical properties. Tensile properties of the epoxy resin were measured based on the ASTM D638. The tensile strength of the NOL-ring was tested on the universal testing machine at a rate of 5 mm/min [15]. According to the ASTM D2344, more than six composite specimens with dimensions of 20 mm × 6 mm × 2 mm were selected for each interlaminar shear strength test. All of the mechanical properties of epoxy resin and CF/epoxy composite were tested separately at six different temperatures in the test chamber, which were maintained constantly at −95, −50, 25 (RT), 50, 80, and 100 °C, respectively. The cryogenic/elevated temperature cycling samples that had been cycled 100 times from −100 to 100 °C were tested at 25 °C.

2.3.3. Acoustic emission data collection

Acoustic emission (AE) dates were collected from the two kinds of fatigue tests during the first 15 cycles and last 15 cycles. A multichannel physical acoustic corporation AE analyzer was used to record the acoustic emission flaw growth data from eight AE channels, each representing a transducer at a unique location on the test bottles as shown in Fig. 3. The data sampling threshold was set to record all acoustic emission hits that had an amplitude of 40 dB or greater.

2.3.4. Morphology observation

The fracture surfaces of the epoxy resin were observed by using scanning electron microscopy (SEM) (HITACHI S-4300). The morphologies of the dome section of the vessels after the burst test were also observed using SEM. Prior to examination, gold was vapor-deposited on all specimens to make them electrically conductive.

3. Results and discussion

3.1. Fatigue behavior of the composite vessels

In the present study, the two vessels exhibited no leakage and burst characteristics during the fatigue cycling. During the hydraulic fatigue cycling, water was directly infused into the vessels at
first, and hence, the vessels only bear pure internal pressurization and the temperature did not change obviously. Nevertheless, it was quite different from the process of the atmospheric fatigue test. As shown in Fig. 4, the changes of the temperature were nearly dependent of the internal pressurization. As the pressure raised up to 35 MPa, the temperature of the vessel and gas both slowly reached the maximum, and the temperature-rising rate of the gas was slightly higher than that of the composite, especially in the starting stage. The total temperature enhancement of the gas in charging presses was up to 80 °C, but only 64 °C for the vessels. However, with the decreasing pressure, the temperature declined. Additionally, the temperature reduction of the gas (more than 140 °C) was far greater than that of the vessels (about 80 °C). In addition, the temperature on the surface of the vessels was higher in the location of sensor 2 than in the location of sensor 3, likely due to a larger thickness of the wall in the dome region.

During the atmospheric fatigue test, there was a significant increase or decrease in gas temperature mostly due to the kinetic energy of gas [17]. When the kinetic energy of the gas, produced by higher pressured storage vessels, transforms into internal energy in the process of charging for vessels leads to temperature rise. In contrast, the discharging process is a cooling process for the gas inside the vessel. This kind of situation can cause the violent change of the superficial difference in temperature inside and outside the vessel, which causes the composite to produce thermal stress due to the mismatch in the coefficient of thermal expansion of adjacent plies with different fiber orientations [18]. These thermal stresses, when added to the stresses resulting from internal pressure, can cause significant laminate damages in the form of micro cracks in the resin that further leads to composite failure [19]. Thus, compared with the hydraulic fatigue test, the vessel was bore with both internal pressure and thermo-mechanical loading during the atmospheric fatigue test.

3.2. Mechanical properties of the epoxy resin and CF/epoxy composites

Thermo-mechanical properties of CF/epoxy composites vary significantly with temperature. As the CF/epoxy laminate carries the internal pressure of the vessel, the effect of temperature on
its material properties can decide the final mechanical properties of the vessels. Fig. 5 shows effects of the cryogenic and elevated temperature on the properties of the epoxy resin used in the study. The tensile strength and modulus of epoxy resin increased as the temperature rose from 25 to −95 °C, and decreased as the temperature dropped from 25 to 100 °C. However, an opposite trend was noted for the elongation percentage of the epoxy resin. The elongation percentage of resin decreased when treated at cryogenic temperatures, whereas increased when treated at elevated temperatures. Compared to the epoxy resin at 25 °C, elongation percentage at −95 °C was decreased by 75% and that at 100 °C increased by 66.7%. This suggests that the fracture toughness of the epoxy resin decreases largely at cryogenic temperature. However, the tensile strength, modulus, and elongation percentage of the epoxy resin, which were under cryogenic/elevated temperature cycling from −100 to 100 °C, were equal to the those of the untreated resin. This indicates that the temperature cycling has little effect to the final mechanical properties of the epoxy resin. The microscopic images of the fracture surface of the epoxy resin, which were tested at different temperatures, were compared. As shown in Fig. 6, at cryogenic temperature, the fracture surface of the epoxy resin was smoother than that at RT and elevated temperature. The epoxy resin showed obviously brittle behaviors at cryogenic temperature and the plastic deformation of the matrix was observed at high temperature. However, morphology observation of the resin after cryogenic/elevated temperature cycling did not present obvious difference compared to the epoxy resin directly tested at RT.

Fig. 7 shows the tensile strength and interlaminar shear strength of CF/epoxy composites at different temperatures. No significant change in average tensile strength was noted throughout the temperature range. However, interlaminar shear strength of the composites increased significantly by 36% at −95 °C. When the temperature was up to 100 °C, interlaminar shear strength of the composites decreased by 54.5% compared to the strength at 25 °C. Fiber matrix interfacial adhesion controls the stress transfer between the fibers and the matrix, the stress relaxation and mechanisms of damage accumulation and propagation [20]. Consequently, the fiber–matrix interface strength of the composite may substantially influence damage development [21]. While the thermo-mechanical behavior of vessels during actual usage is
extremely complex, the violent changes of the mechanical properties of the composites and epoxy resin at different temperatures with internal pressure could effectively affect on the mechanical properties of vessels.

3.3. Fracture development measured by acoustic emission

Figs. 8 and 9 presented damaged events measured by AE during the first 15 cycles and last 15 cycles in two kinds of fatigue tests, respectively. The red points in the graphs represented the damage situation of the vessel. Regardless of the applied fatigue types, there were considerable AE activities during the first 15 cycles. But more damaged signals were observed in the atmospheric fatigue test. On the contrary, in the last stage of the hydraulic fatigue test, no damage signals was observed (Fig. 9b). The major reason could be that the damage of the vessel somewhat reached its saturation. However, for the atmospheric fatigue test, AE signals were not reduced (Fig. 8b). As shown in Fig. 10a and b, there were more damage signals during the atmospheric fatigue test than that during the hydraulic fatigue test. In composite material, matrix cracking, fiber failure, delamination, debonding of the matrix from the fibers, fiber pull-out, interfacial debonding and sliding are possible sources of AE [22–24]. This suggests a faster and more damage growth of the vessel in atmospheric fatigue test under thermal-mechanical fatigue than in hydraulic fatigue test under only mechanical fatigue. Moreover, the surfaces of the vessel that underwent the atmospheric fatigue test showed obvious matrix crackles parallel to the vessel axis in resin-rich regions, which was not observed in the vessel under the hydraulic fatigue test as shown in Fig. 11a. It was shown that the surface resin had failed in a brittle manner under internal atmospheric pressure loading.

3.4. Burst characteristics of the vessels

Two types of vessels, following hydraulic and atmospheric fatigue tests respectively, underwent the burst test. The results were shown in Table 1. The burst pressure of the vessel after the atmospheric fatigue test was decreased by 9.6% compared to that of the vessel after the hydraulic fatigue test. The two vessels showed the different failure mode. The vessel after the hydraulic fatigue test failed at the center of the vessel as shown in Fig. 12. In contrast,
the vessel after the atmospheric fatigue test failed at the vicinity of dome tangent line. The dome sections of the vessels were mostly intact after the burst test, because of fiber piled up during filament winding leading to excellent properties.

Samples were fabricated from the dome section of the vessels after the burst test for SEM. For the specimen of the hydraulic test, few micro cracks were observed as shown in Fig. 13. However, as shown in Fig. 14, many micro cracks existed on the vessel after the atmospheric test. In addition, the cracks were along the fibrous direction and ended at the interface between the piles with different orientations, yet no fiber breaks were observed in the layer. Some defects were observed in the layers (Fig. 14), which are probably formed by trapped air bubbles during the composite fabrication. As shown in Fig. 14b, the crackle generally expanded from a defect to another defect or matrix cracks. While the burst test-induced damage and fatigue cycling-induced damage could not be completely separated, it was conceivable that some cracks were created during atmospheric fatigue cycling because AE events were collected as mentioned above. During atmospheric fatigue cycling, the vessel suffered thermal stress with internal pressure. Furthermore, the material mechanical proprieties decreased at extreme temperature. Thus, all of those were most likely to initiate interfacial cracking that leads to composite failure. It is likely that there could be also more damages inside the composites of the vessel, which underwent atmospheric fatigue cycling. As a result, these damages could give rise to the degradation of mechanical properties in the structures [25–27]. Therefore, it could be concluded that atmospheric fatigue cycling showed a great effect to the final mechanical properties of the composite vessel.

### 4. Conclusions

Compared with the vessel under hydraulic fatigue cycling, the vessel under atmospheric fatigue cycling suffered not only internal pressure but also thermal stress caused by the cryogenic/elevated temperature changes accompanied by internal pressure during the charging and discharging processes. The mechanical properties of the epoxy resin and composites as a function of composition also presented violent changes with temperature. The fracture toughness of the epoxy resin matrix decreases largely at cryogenic temperatures and distinctive plastic deformation of the matrix, and dramatically decreased interlaminal shear strength of the composites occur at high temperature. All of these can stimulate more damages to the vessel during the atmospheric fatigue test. AE events and morphology observation show that most of the damages results from the matrix cracks and internal debonding. Damages to the vessel caused by atmospheric fatigue pressure test could lead to degredation of the final properties according to the burst test, resulting in 9.6% reduction of the burst pressure of the vessel after atmospheric fatigue. Therefore, thermo-mechanical

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### Table 1

<table>
<thead>
<tr>
<th>No.</th>
<th>Type of fatigue cycling (0–35 MPa, 100times)</th>
<th>Burst pressure (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Hydraulic fatigue</td>
<td>83</td>
</tr>
<tr>
<td>2</td>
<td>Atmospheric fatigue</td>
<td>75</td>
</tr>
</tbody>
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**Fig. 11.** Optical images of surface cracks of the vessel matrix after 100 times fatigue cycling: (a) atmospheric fatigue process and (b) hydraulic fatigue process.

**Fig. 12.** Optical images of the fractured vessels after burst test: (a) hydraulic fatigue cycling and (b) atmospheric fatigue cycling.

**Fig. 13.** SEM images of the dome section of the vessel after burst test (under hydraulic fatigue cycling).
properties were worth evaluating parameters for composite pressure vessels used in engineering applications.

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References