DAMAGE TOLERANCE OF COMPRESSED GASEOUS HYDROGEN COMPOSITES VESSELS

M. Weber[a*], C. Devilliers[a], N. Guillaud[b], F. Dau[b], B. Gentilleau[c], F. Nony[c], S. Villalonga[c], D. Halm[d]

[a]*Claude Delorme Research Center (CRCD), Material Science Group, Air Liquide, 1, chemin de la Porte des Loges, 78354 Jouy-en-Josas Cedex, France*
[b]*Institute of Mechanical Engineering, Durability department, located at ENSAM, esplanade des arts et métiers, 33405 Talence cedex*
[c]*CEA, DAM, Le Ripault, F-37260 MONTS, France*
[d]*Institute Pprime, CNRS, ISAE-ENSMA, University of Poitiers, BP 40109, F-86961 Futuroscope Chasseneuil Cedex, France*
**mathilde.weber@airliquide.com*

Keywords: wound carbon fiber, pressure vessels, damage, impact.

Abstract
The present study is part of a project funded by the French National Research Agency, called Toledo. It is dedicated to the study of the effect of mechanical impacts on wound carbon fiber composite pressure vessels performance. A composite pressure vessel with a polymer liner has been designed and subjected to mechanical impacts. The damage in the composite due to the impact has been characterized by X Ray tomography. The residual performance (burst pressure) is assessed after the impact and after subjecting the composite pressure vessel to static or cyclic pressure load prior to burst test. The purpose is to clarify the effect of additional mechanical loads encountered in service by composite pressure vessels on the tolerance to the damage associated with mechanical impacts and thus on residual lifetime.

1. Introduction

The development of hydrogen as a reliable energy vector is strongly connected to the performance and the level of safety of the components of the supply chain. In this respect, achieving an efficient and reliable storage is crucial to address transition markets - such as power supply to off-grid area and hydrogen powered forklifts - and transportation markets. Among storage technologies (cryo, compressed, hydrides, adsorbent), compressed hydrogen storage is the most promising technology. Currently for industrial applications, hydrogen is stored at 200 bar in metallic cylinders which have poor mass storage efficiency (about 1 wt% of Hydrogen stored) but present high impact resistance. To achieve the required performance for hydrogen energy applications in terms of autonomy and weight efficiency, hydrogen must be stored at pressure up to 700 bar in carbon fiber composite pressure vessels (designated hereafter by COPV). As an example, the European target weight efficiency for on-board storage systems in vehicles is set at 4.8 wt% of stored hydrogen [1]. In the scope of hydrogen energy markets, COPV can be subjected to a broad range of impacts either usual or accidental (car accident, fall or impact during handling and transportation of transportable COPV). For carbon fiber composites pressure vessels, damage resulting from a mechanical impact or a fall, its evolution under typical in-service loadings (monotonic pressurization, filling/emptying cycles,…) and the corresponding loss of performance are not well described as only a few studies tackle the consequence of impact on the residual life time of composite materials obtained by filament winding [2][3][4]. As a consequence, COPV may be overdesigned and minor but visible damage resulting from impacts at the surface results in the withdrawal of the cylinder from the supply chain, without knowing if the damage is critical for the cylinder integrity or not. It is thus critical to assess impact resistance of composite pressure vessels and to determine which impact causes a cylinder to burst immediately, after the next fill or after some time in service. In consequence, a scientific knowledge on the behavior of carbon fiber composite cylinders subjected to impacts must be developed. For this purpose, the project Toledo funded by the French National Research Agency was launched in 2011 (duration 42 months). This project coordinated by Air Liquide (Gas Company) gathers several research academics partners: PPRIME, I2M laboratories and the French Institute CEA. The project combines experimental works on specimens and COPV, but also numerical approaches. In the course of the project, a significant number of high pressure COPV have been subjected to impacts tests representative of normal and accidental in usual situations. Different techniques have been used to characterize the resulting damage in the composite structure (micrography, X-ray tomography, acoustic emission, etc). After impact, COPV are then subjected to mechanical load (static or cyclic load) to assess the effect of further use on damage evolution and loss of performance (hydraulic burst test). The present paper addresses this part of the project.

2. Description of the composite cylinders and experimental set-up

2.1. Carbon Fiber Pressure Vessels

The COPV studied is a 2.4L cylinder with a thermoplastic liner and a steel boss (25E thread). The composite wrapping is based on a high resistance carbon fiber and an epoxy resin. The composite pattern was determined for the purpose of the project to reach a burst pressure close to 2100 bar (corresponding to 700 bar Working Pressure according to the regulation for the Transport of Dangerous Goods [5]). The test pressure is 1050 bar (1.5 times the working pressure). The external surface of the COPV is protected by a 1 mm thick layer of glass fiber composite wrapping (same resin than the carbon fiber composite). A picture of a finished COPV is displayed in Figure 1. Mean burst pressure was assessed by hydraulic tests on 3 reference specimens and established at 1963 ± 25 bar. Burst occurs on the cylindrical part of the COPV.

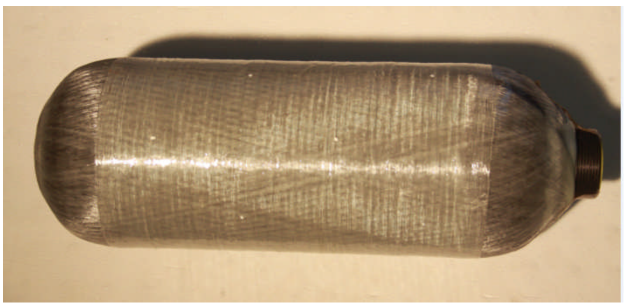


Figure 1. Picture of a 2.4L COPV designed for the project

2.2. Impact set-up

The mechanical impact tests are carried out by dropping a metallic impactor on a cylinder at atmospheric pressure maintained by two stripes on each side of the cylinder. The metallic impactor chosen for this study has a 120° angle and a length of 100 mm. The dropped impactor makes an angle of 45° with the axis of the COPV in the middle of the cylindrical part. Impacts with a mass and a height of fall ranging from 2 to 25 kg and 0 to 2.8 m, respectively, can be carried out with this experimental set-up. Figure 2 displays an example of an impact test monitored with a high speed digital camera (10 000 images/s), a laser displacement sensor and a load sensor (100 Hz).

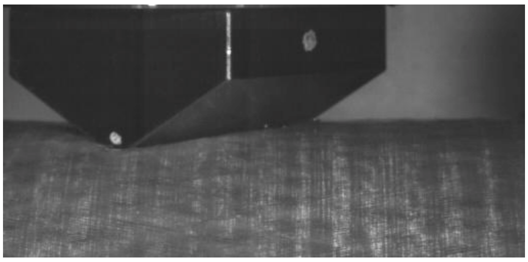


Figure 2. Picture of a 2.4L COPV during the impact (high speed camera)

The speed of impactor at the time of impact is evaluated theoretically and also deduced from the image analysis from the cameras on the one hand and from the laser displacement sensor on the other hand. This value is then used to calculate the impact energy (incident energy).

2.3. Damage analysis

After the impact, the COPV were subjected to X Ray Tomography with a resolution of 100 µm. Delaminations have been especially hightlighted by this way.

2.4. Hydraulic tests

The hydraulic burst test installation has a maximum pressure capacity of 3200 bar. The test chamber is equipped with windows to monitor the burst test with a high speed digital camera (HSDC, image frequency: 20 upto 80kfps). The COPV was mounted to have a visual view of the impacted area (distance between COPV and HSDC<1m). During the pressure ramp, acoustic emission signal has been recorded. The signal analysis is not presented here.

Cyclic pressure tests have been carried out with water at 2 cycles per minutes with a 16 bar minimum pressure and a maximum pressure of 1050 and 1330 bar, depending on the test.

3. Results & discussion

3.1. Effect of the impact energy on the COPV residual burst pressure

One impact is carried out per COPV. After performing X-Ray Tomography, the COPV was then subjected to a hydraulic burst test. Impact test matrix and residual burst pressure are listed in Table 1. Note that the lower impact energy is in the same range as a fall from 1.2 m (representative of a fall from a truck). For impact energies ranging from 52 J (4.6 kg, 1.07m) to 348 J (12.8 kg, 2.8 m), the burst pressure of the COPV remains in the range of the one of a virgin COPV. Results suggest that the dispersion is higher than the one established on the 3 reference specimens, namely ± 25 bar. Only the impact at 491 J (21 kg, 2.22 m) leads to a significant decrease of burst pressure. It is however worth mentioning that the burst is initiated on and grows from the damaged area for all COPV impacted with energy of 68 J (12.8 kg, 1.65 m) and more. Figure 3 gives an example of the COPV burst after an impact with a 120° angular impactor.

Test	Impactor mass (kg)	Height (m)	Impact energy (J)	Burst pressure (bar)
1	12,8	0.5	230	2032
2	12,8	1.65	68	2008
3	12,8	2.8	348	1966
4	21	2.22	491	1721
5	4,6	1,07	52	2047
6	4.6	2,22	106	1982
7	21	1,07	236	1937

Table 1. Characteristics of the impact test matrix carried out on the 2.4L COPV with an angular impactor 120°

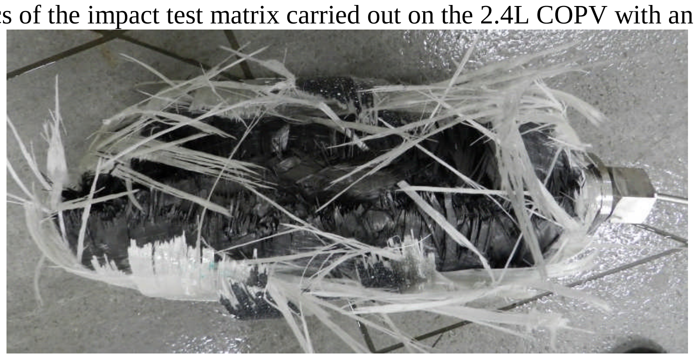
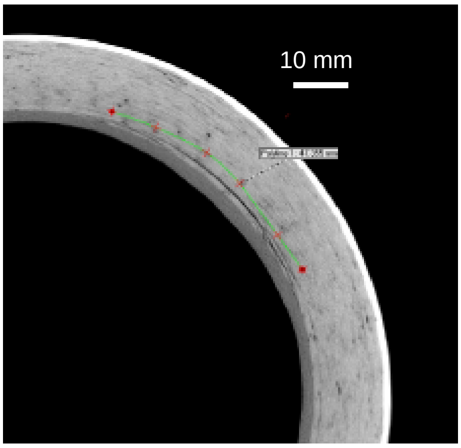


Figure 3. Picture of a 2.4L COPV after impact after the burst test

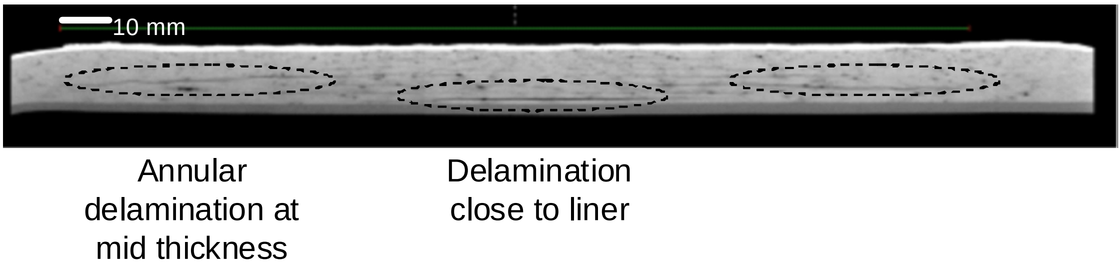
3.2. Damage characterization in the composite after an impact

Impact creates several interlaminate damages and matrix cracking in the composite. The first delamination occurs in the glass fiber layer (more precisely between the glass fiber composite and the outer carbon fiber composite layer). It gives a whitening of the cylinder in the impact area matching with an ellipsoidal shape as illustrated in figure 4A for impact test n° 4. Delaminations in the carbon fiber composite wrapping were observed by X-Ray tomography. For the two lowest impact energies, one main delamination is observed at mid thickness of the carbon fiber composite. It presents an annular "donut" shape (see Figure 4C). For the 106J impact energy and more, an additional large delamination is observed close to the liner (not in a donut shape, plain delamination) (see Figures 4B and 4C). Smaller delaminations are also observed between the delaminations zone at mid thickness and close to liner in those cases. Delaminations have ellipsoidal shapes with long radius in the axis of the COPV and much broader size than the superficial delamination in the glass fiber composite. The size of the delaminations increases with the energy of the impact.

(A)



(B)



(C)

Figure 4. (A) picture of COPV after impact n°4 (491 J), (B) & (C) X Ray Tomography

The Figure 5 shows the long radius of the ellipsoidal delamination in the glass fiber composite over the one of the carbon fiber composite at mid-thickness for the different impacts. It seems to be a linear correlation between the two parameters. The same trend was observed for the delamination area. This analysis could be a basis to define criteria for visual inspection of such COPV before filling and during periodic inspection.

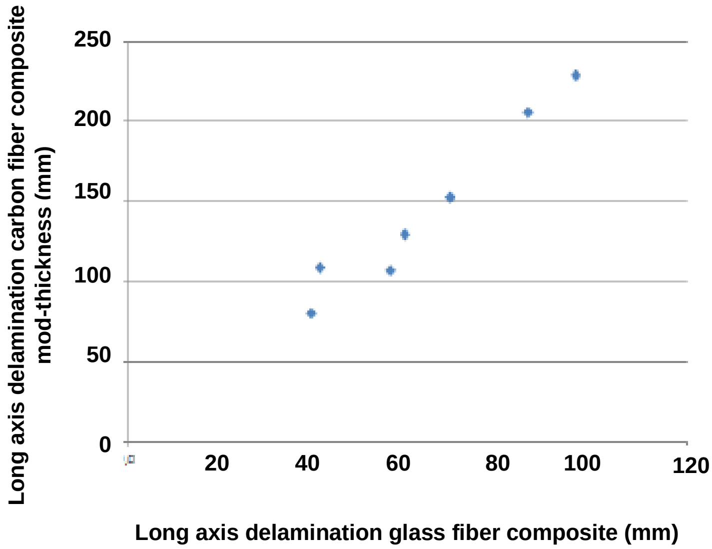


Figure 5. Correlation between the delamination in the glass fiber composite layers and in the carbon fiber composite at mid thickness (long axis of the ellipse)

3.3. Effect of cyclic mechanical load on a COPV after impact

Inspection of COPV consists of a visual inspection (internal and external) and a hydraulic proof test. Periodic inspection frequency is fixed every 3 to 10 years, depending on the application. Furthermore, for transportable cylinders, a visual inspection is performed before each filling. Therefore, it is of high interest to assess the evolution of damage and cylinder performance (burst mainly) after further mechanical loading as encountered in service (i.e. static and cyclic pressure load).

For this purpose, three virgin COPV were thus impacted with the 120° angular impactor at the highest impact energy of the previous test matrix (impact n°4 from table 1, 21 kg, 2.22 m, 491 J) and then subjected to cyclic loads. The cyclic pressure load parameters are described in Table 2. Note that static tests at the same maximum pressure and equivalent duration (90h) are planned on three additional COPV.

Test	Pressure amplitude (bar)	Number of cycles	State of cylinder
1	1050 (1.5xWP)	10800	No leak, no significant evolution of surface whitening
2	1330 (1.9xWP)	674	Burst
3	1330 (1.9xWP)	1912	Leak

Table 2. Characteristics of the cyclic pressure load on the COPV after the 491 J impact (21 kg, 2.22 m/s)

The impacted COPV sustained 10 800 pressure cycles at test pressure (1.5 times the Working Pressure, corresponding to the maximum allowable pressure in service) with no leak and no significant evolution of the surface whitening. Its residual burst pressure will be assessed after

X Ray tomography analysis (on-going). Cycling at higher pressure (1330 bar) leads however to a leak or burst of the COPV as a consequence of the impact. The damage evolution is under assessment for those COPV.

4. Conclusion

High pressure COPV were designed for this study with a burst pressure of 1960 ± 25 bar and a test pressure set at 1050 bar. For the impactor investigated in this study (angular 120°), it has been possible to build a correlation between external damage visible on the surface and the damage within the carbon fiber composite wrapping. Only the impact characterized by the highest energy (491 J, corresponding to a mass of 21 kg and a height of fall of 2.22 m) leads to a noticeable though not so important decrease of burst pressure (from 1963 ± 25 to 1700 ± 25 bar). Such impact does not lead to leak or burst during a cycling test at 1050 bar test pressure though a higher cycling pressure of 1330 bar leaded to premature failure of the COPV. This is a first preliminary result that will help to the definition of visual criteria for the removal of COPV from service.

Further work is necessary to establish a robust criterion for visual inspection that takes into account the loss of COPV performance induced by mechanical impacts. This criterion is important to carry out efficient pre-filling and periodic inspection controls of COPV in service. Further impact test on the 2.4L design and a larger COPV (volume of about 100°C) are on-going in the course of this project. A list of acceptable/unacceptable damage is thus expected from Toledo ANR project in order to optimize service life of composite pressure vessels. This study will lead to recommendations for the industry and normative committees on the design of composite cylinders taking into account a quantitative analysis of COPV damage tolerance, by providing knowledge to define a withdrawal threshold.

Acknowledgements

We greatly acknowledge the French national research agency ANR for the financial support provided to this study. The authors would like to warmly thank all Toledo partners for the amount of work carried out in this project.

References

[1] Fuel Cell and Hydrogen Technologies in Europe: Financial and technology outlook on the European sector ambition 2014-2020, 2011.
[2] S. Wakayama, S. Kobayashi, T. Imai and T. Matsumoto. Evaluation of burst strength of FW-FRP composite pipes after impact using pitch-based low-modulus carbon fiber. Composites Part A: Applied Science and Manufacturing, Volume 37(11), 2002-2010, 2006
[3] S. Kobayashi, T. Imai and S. Wakayama. Burst strength evaluation of the FW-CFRP hybrid composite pipes considering plastic deformation of the liner. Composites Part A: Applied Science and Manufacturing, Volume 38(5), 1344-1353, 2007
[4] L. Ballère, P. Viot, J.L. Lataillade, L. Guillaumat and S. Cloutet, Damage Tolerance of impacted Curved Panels, Int. J. of impact engineering, Volume 36(2), pages 243-253.
[5] Transportable gas cylinders – Fully wrapped composite cylinders, EN 12245, 2009.