

ON BOARD 70 MPA HYDROGEN COMPOSITE PRESSURE VESSEL SAFETY FACTOR

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ABSTRACT

The safety factor of a composite structure in relation to its mechanical rupture is an important criterion for the safety of a 70 MPa composite pressure vessel for hydrogen storage, particularly for on-board applications (car, bus, truck, train...). After an introduction of Type IV technology, the contribution of carbon fibre, composite material, structure, manufacturing process of pressure vessels and environmental effects on the safety factor are commented. Thanks to an experimental-based evaluation on composite material and H₂ composite pressure vessel, the safety margins are addressed.

1 INTRODUCTION

Today, to overcome against global warming and contribute to the increase of the temperature value of the earth's surface to a few degrees, Society is facing energy transition to consider other sources than fossil fuels leading to generate CO₂ greenhouse gas, with a goal of carbon neutrality by 2050.

H₂, di-hydrogen molecule, is called commonly hydrogen. Hydrogen generation associated with a very low carbon footprint is growing fast. In addition, hydrogen could be considered to store energy.

Hydrogen storage appears to be a key issue for the extensive use of H₂ and for the deployment of the whole hydrogen value chain. Hydrogen could be stored in Fuel Cells Electric Vehicles (FCEV like cars, forklifts, trucks, trains, buses, etc.) thanks to 70 MPa Composite Pressure Vessels (CPV). These vehicles are in a commercial deployment phase since 2015. Other application for transportable storage (cylinders, bundles and tube trailers) or stationary storage (HP buffers for Hydrogen Refueling Station, Storage of H₂ produced from intermittent sources) are using also H₂ CPVs.

Beyond the improvement of gravimetric and volumetric performance of CPVs, there is a strong need to guarantee that CPVs are reliable, safe, and cost competitive. It is therefore possible to optimize the design of the CPV by reducing the safety factor and consequently reducing the composite thickness of the exterior shell. Such an objective requires a reduction of the involved carbon fibre, *i.e.* the cost of the composite cylinder.

One can include the safety issues. 70 MPa Nominal Working Pressure (NWP) CPVs mechanical burst is a significant hazard.

An important safety factor is the "burst ratio" which is equal to the value of the minimum burst pressure value of the CPV at its initial life divided by the value of the NWP during its all life. This minimum burst pressure ratio shall not be less than a value given by a table in the regulations like EC79 or RM134. Today, for CPV using carbon fibre, at initial life, the value is equal to 2.25.

In this document, we introduce the corresponding papers that detail the CPV technology, and try to give a well-definition of the safety factor against CPV burst event. The composite material commonly used for CPV is taken into account. Experimental results on composite wrapping are reported to provide a better understanding of the damage accumulation mechanisms and kinetics under typical loadings under service. The safety gaps evaluation can be compared to the safety factor.

2 ON-BOARD 700 BAR HYDROGEN PRESSURE VESSEL

2.1 Context

Fuel-cell electric vehicles are progressively replacing gasoline internal combustion engine vehicles (Gasoline ICE Vehicles) with an equivalent range and filling time. Transportation sector represent today 14% of worldwide CO₂ emissions,

However, FCEV shall be economically accessible while having a level of safety equal to or greater than Gasoline ICE Vehicles. For reasons of maturity and compactness (table 1), the technology of on-board storage of hydrogen gas at 70 MPa is deployed through CPV considering composite materials.

Table 1. FCEV H₂ storage systems global comparison

FCEV Compressed H ₂ Storage System	Gaseous	Cryo-compressed hydrogen	Liquid	"Solid"
Storage temperature (°C)	15°C	-230°C	-250°C	15°C
Storage pressure (MPa)	70 MPa	35 MPa	0.1 MPa	0.1 MPa
Hydrogen Volumic mass (g/L)	40	80	63	-
Volumetric density (g H ₂ / L system)	25	60	45	20
Gravimetric density (kg H ₂ for 100 kg system)	5% to 6%	4%	10%	< 2%
TRL (Technology Readiness Level)	type III 35MPa: Commercial type IV 70MPa: Commercial type V: 3 to 4	5 to 6	3-4	Commercial*
Cost (€/kg H ₂ for 10000 units per year)	700	>>500	>>500	>>500

* example of submarine (hydrides)

In order to reduce the cost of the storage system, the amount of carbon fibre used for the composite laminate required for sustaining high pressure as external envelope, can be minimised while respecting performance and safety constraints through numerous approval tests [1][2].

Carbon fibre is the first cost driver constituting today about 33% of the cost of the vessel (estimated value for reservoirs 700 bar of 62L produced at 8000 units per year [3][4]),

Indeed, it seems possible to minimize this mass by a better use of the carbon fibre that is used at about 20% to 30% (1) (this dimensionless number will be the subject of another article being redacted) of its overall capacity during a burst [3]. For example, we can imagine a reduction of the composite mass by 11% gives, in first order, a reduction of the cost by 3.7%.

$$Efficiency_{Type\ IV\&\ V\ CPV\ laminate} = \frac{\overline{P_{burst}} \cdot V_{fluid}}{\sigma_{UD\ ply}^L \cdot V_{laminate}}, \quad (1)$$

where $Efficiency_{CPV\ laminate}$ - dimensionless number; $\overline{P_{burst}}$ - mean burst pressure, Pa; V_{fluid} - internal vessel volume, m³; $\sigma_{UD\ ply}^L$ - axial tensile strength of the composite, Pa; $V_{laminate}$ - composite vessel volume, m³.

The current performance of the Compressed Hydrogen Storage System and improvement objectives are shown in the following table. It would be necessary to reach by 2030 a gravimetric rate of 6% (table 2) with a cost of 300 €/kg H₂ stored.

Table 2. European FCH-JU target for Compressed H₂ Storage System (CHSS) [5]

Key Performance Indicators for On-board Compressed H ₂ Storage System (CHSS)	Unit	2012	2017	2020 (old)	2020 (revised)	2024	2030
Cost	€/ kg H ₂	>3000	800	600	500	400	300
cost reduction / 2020 revised cost	%	<-500%	-60%	-20%	-	20%	40%
Volumetric capacity	kg H ₂ / L of CHSS	0.02	0.022	0.023	0.03	0.033	0.035
Gravimetric capacity	kg H ₂ / kg of CHSS	<4	4	5	5.3	5.7	6

According to the more or less optimistic forecasts, Figure 1, [6], there will be in public space millions of vehicles with several 70 MPa tanks.

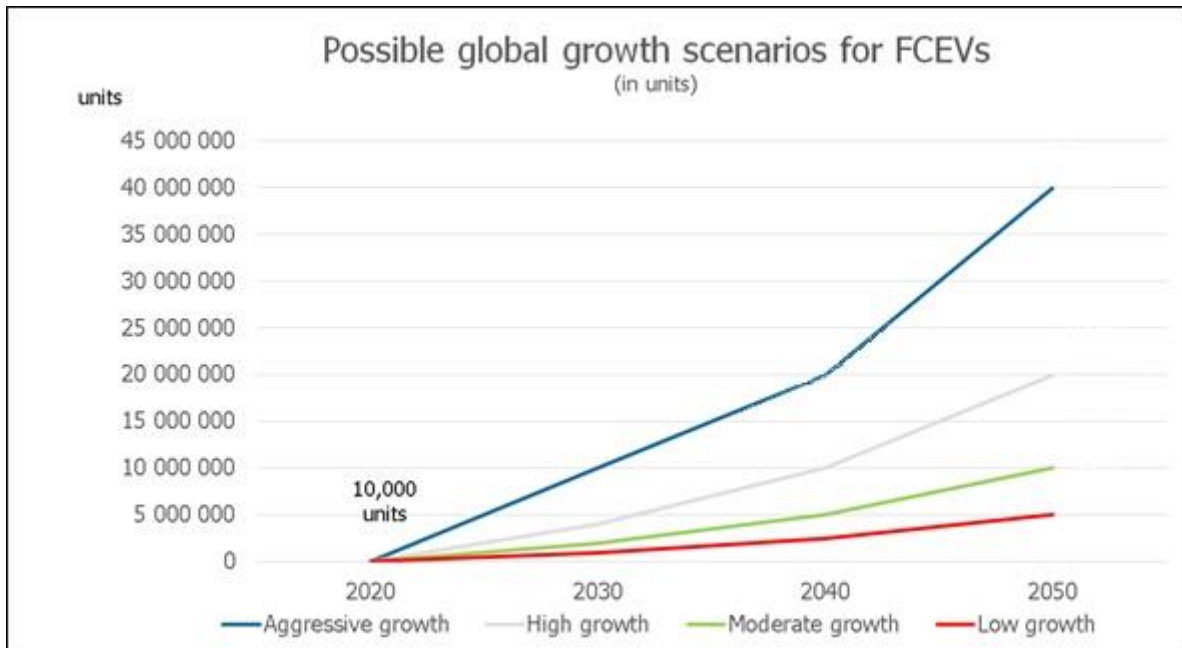


Figure 1. Possible global growth scenario for FCEVs

2.2 Composite pressure vessel technology

The most mature technology for storing hydrogen is in compressed form in type III (35 MPa) or type IV (35 & 70 MPa) composite pressure cylinders, Figure 2. For 70 MPa application, type IV vessels are used thanks to good resistance to fatigue behavior. The 70 MPa type IV mandrel is a bladder made of polymer material called a liner whose function is to contain hydrogen. At its ends, one or two metallic parts called bosses are used. The bosses ensure the connection with the storage system. A composite laminate is wound around the liner to procure a high mechanical strength to the vessel.

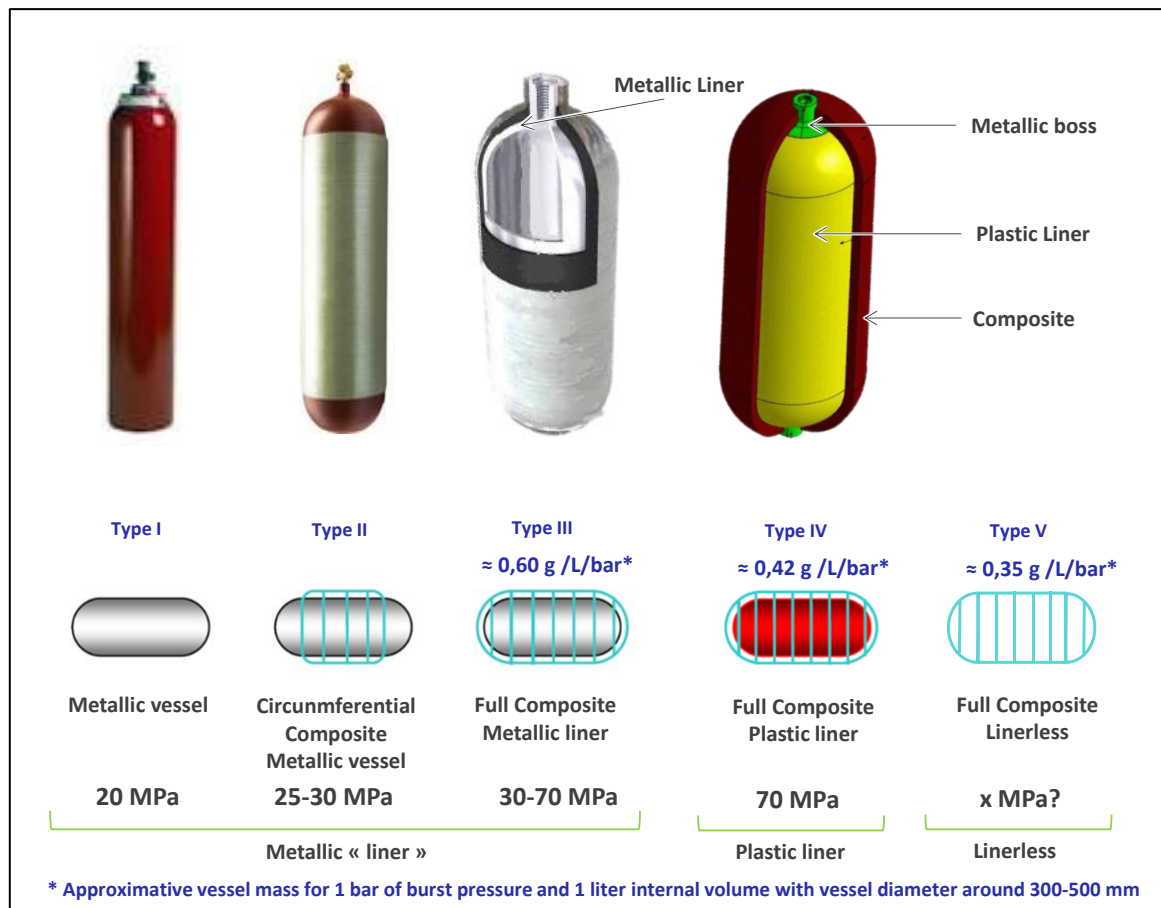


Figure 2. Pressure Vessel types from I to V

Composite laminate is manufactured by filament winding process, Figure 3. During the process, continuous reinforcements (filament, wire, yarn, tape, or other) impregnated of resin are wound around a rotating plastic liner in a prescribed way. When the required number of layers is applied, the resin of the wound form is cured following a temperature transient. The materials used commonly for 70 MPa type IV pressure vessel are:

- For the composite
 - Toray T700 carbon fibre
 - Epoxy resin
- For the liner
 - Poly-ethylene
 - Poly-amide
- For the boss
 - Aluminium 6061



Figure 3. CEA Robotic Filament Winding Machine (CEA concept)

2.3 Material and Manufacturing Process key parameters

This section introduces the main parameters that influence pressure vessel performance, i.e. processing parameters of the vessel itself. The properties and variability of each material, the composite process manufacturing, Figure 4, and the variability of each process step (resin mixing, impregnation, winding, curing) and the variability and the sensitivity to external condition explain the main variability on vessel thermo-mechanical performance.

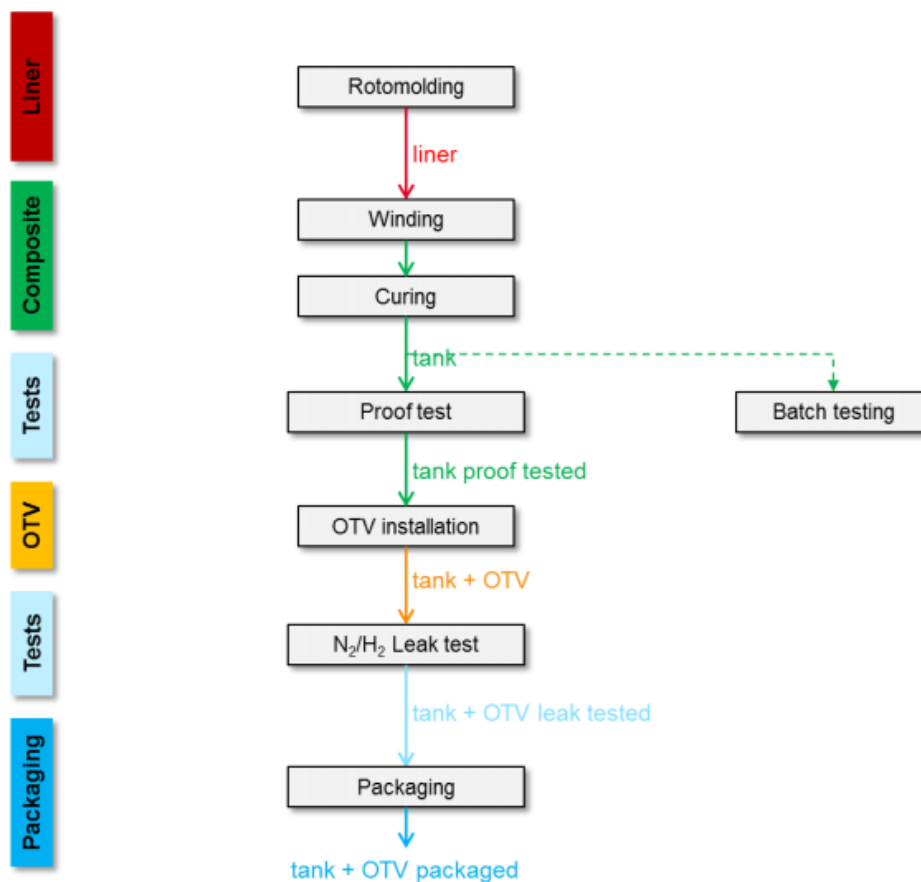


Figure 4. On-board 70MPa H₂ CPV manufacturing steps with Winding and Curing steps for composite laminate manufacturing [1]

This variability inherent in the carbon-epoxy composite materials and the process necessarily affects that of the CPV. Not all, parameters have the same importance in the behaviour and service life of the tank. To rank them, it is important to remember that the breakdown of the structure is the consequence of degradation mechanisms. The accumulation of damage means that either an opening of the CPV cylinder zone is found or a rupture of one of the two domes occurs. Mechanical analyses suggest that combining material properties with each of these modes is in balance. More precisely, the rupture of the cylinder zone is driven by the rupture of the fibres and that of the domes uses a more complex mode combining the degradation of the matrix by shear and fibre rupture. The consequence is that any variability on the resistance of the fibres or on the resistance of the matrix will have an impact on the overall resistance. While some parameters are fairly well controlled (fibre resistance and variability), others are subject to thermal and water aging couplings with alternating loads. More concretely, knowing the evolution of the shear strength of the composite over a 20 years lifetime is strategic to understand the long-term performance and refine the value of the “burst ratio”.

The parameters of variability are discussed in the following sections.

Carbon fibre: Fibre strength and variability

For the 70 MPa storage application, the reinforcement consists of carbon fibre mono-filaments of 7 μm diameter joined in tows of 24,000 filaments. The carbon fibre, which is almost isotropic transverse, largely determines the mechanical properties of the future composite material. A statistical analysis on the variation of rupture strength of T700 carbon fibre 24,000 dry filament tows was performed [7]. Figure 5 shows the statistical distribution (following Weibull distribution) of fibre tensile strength property of the T700 carbon fibres. It is well known that fibre strength property can significantly affect the tensile strength of composite laminates according to the fact that the fibre properties play an important role on the strength and ultimate behavior of the composite. At the first order, this carbon fibre distribution explain the experimental scatter of initial 70 MPa CPV burst pressure value at initial life that can reach ± 17.5 MPa ($\pm 10\%$ of the nominal working pressure) or more. For this reason, the minimum burst pressure value shall be greater than 157.5 MPa (70 MPa nominal working pressure value multiplied by 2.25 burst ratio for type IV CPV with carbon fibre). Other parameters are described in the following sub-sections.

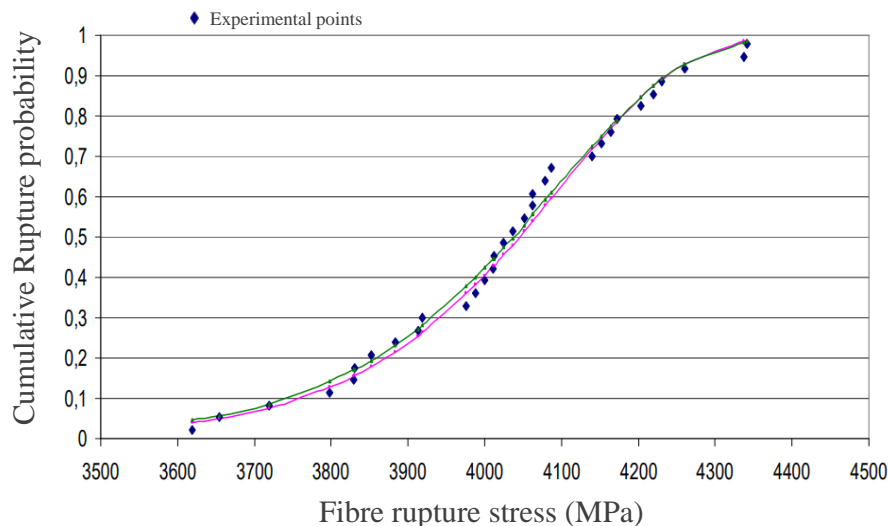


Figure 5: Statistical distribution (following Weibull distribution) of 24000 T700 dry filaments [7]

Several Abaqus Finite element simulation using Weibull distribution of the fibre on 3D 70MPa 2L vessel model [8] show the same scattering between experimental burst results and calculation burst results, Figure 6.

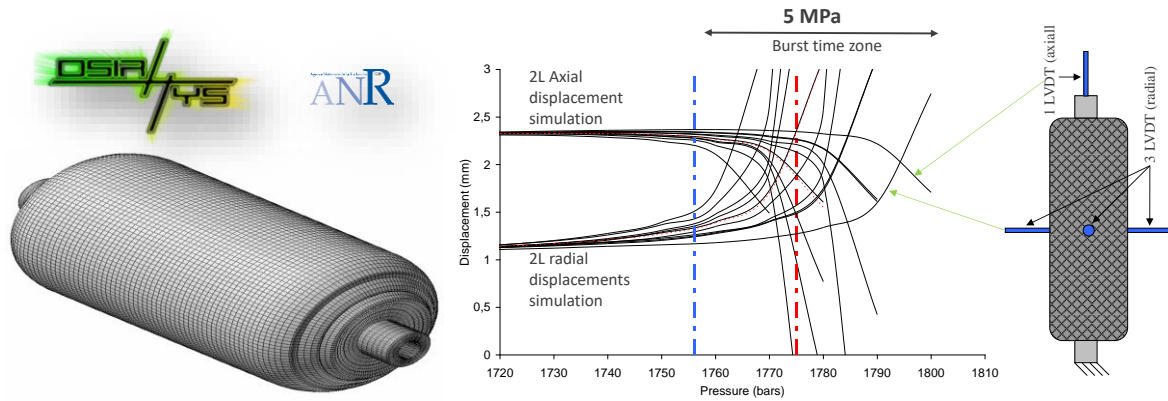


Figure 6: Simulation of the distribution of the burst pressure for a 2L 70MPa vessel [8]

Matrix: Resin of the composite: Modulus and sensitivity to temperature and humidity

The material used for impregnation are commonly thermosetting resins. Examples of suitable matrix materials are epoxies or thermoplastic-modified epoxies. The mechanical properties behaviour of such epoxy networks are sensitive to temperature especially close to the glass transition temperature, Figure 7. In the glass transition region, the stiffness of the polymer is decreasing drastically as the temperature increases. For a given crosslinked epoxy polymer, the observed T_g value is defined by the crosslink density, i.e. the molar mass between crosslinks (and also by the chemical architecture of the chains between crosslinks). After curing of a part prepared from an epoxy resin, the crosslink density is influenced by the potential occurrence of the vitrification which limits the continuation of the 3D polymerization. This limitation of reaction which could occur if the curing temperature is lower than the T_g for the fully cured network leaves reactive groups unreacted. This could happen in different positions in the composite for example across the composite wall thickness if the temperature is not well controlled.

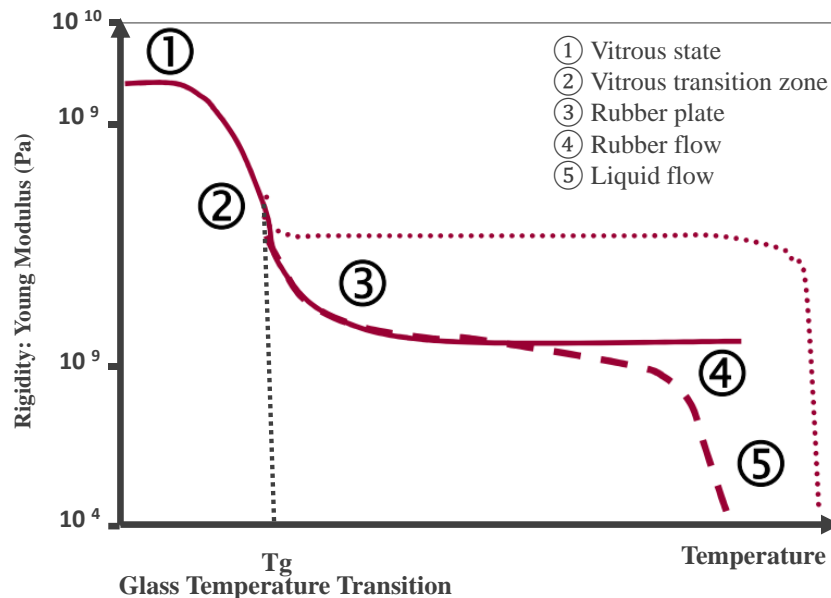


Figure 7: Stiffness (modulus) decrease of viscoelastic polymers in the glass temperature transition (T_g): (—) polymer network; (----) amorphous thermoplastic polymer; (....) semi-crystalline polymer

Resin & temperature: For new ISO 19881 standard [9], 2018, feedback from European Pre-normative HYCOMP project [10], has been taken into account. For the first time, a new specification on resin system materials that shall have a glass transition temperature of at least 20°C above the maximum CPV temperature, *i.e.* $\geq 105^\circ\text{C}$ as the working temperature range of on-board H_2 CPV is between -40°C and $+85^\circ\text{C}$. The glass transition temperature of resin materials shall be determined in accordance with ASTM D3418 standard. By this way, there is safe container operation experience at T_g at least 20°C above the maximum CPV temperature. At temperature becoming greater than “ $T_g - 20^\circ\text{C}$ ”, viscous flow phenomena can have an effect, resulting in stress concentration and damage accumulation in the laminate. For type IV CPV [1], the curing temperature for thermosetting resins shall be at least 10 °C below the softening temperature of the plastic liner. The softening temperature shall be $\geq 100^\circ\text{C}$ [1]. For polyethylene liner, the softening temperature is around 115°C so the maximum temperature of curing shall be lower than 105°C . This low curing temperature is a strong limitation for high composite resin T_g .

Resin & water: The cured resin can be also sensitive to water uptake leading to plasticization effect, *i.e.* decrease of T_g . For that, ISO standard [9] and EC79 regulation [2] introduce a specific material test in accordance with ASTM D2344 standard (24-hour water boil, the composite shall have a minimum shear strength of 13,8 MPa)”. The water intake diffusing into the HYCOMP [10] composites could cause a reduction to the glass transition temperature of the material. The modified T_g for the hygro-saturated composites reduced to 95°C , which is 20°C lower than the T_g for the virgin composites. This means that the damage process can be accelerated in the water-saturated composite when subjected to a lower temperature. In fact, the plasticization exacerbates the viscoelastic character of the epoxy matrix, *i.e.* the mechanical behaviour becomes more sensitive to the temperature and strain rate.

In conclusion, two temperature margins shall be taken into account. 20°C for T_g reduction due to water uptake and 20°C to limit the decrease of the matrix stiffness close to T_g , *i.e.* the concerned temperature range is 40°C. If maximum operational temperature of the H_2 CPV is 85°C , the T_g shall be higher than 125°C to avoid the damage accumulation process of the composite. Taking into account this condition, the lifetime of the composites couldn't be significantly affected. For controlling the curing achievement, *i.e.* the conversion of reactive species of the thermoset resin, and the polymer network architecture which controls its mechanical behaviour, the temperature should be followed at the surface of the material rather than the temperature of the oven.

For 70 MPa H_2 vessels, it should be interesting to choose an epoxy matrix with the highest resistance to stress relaxation (creep) process and the highest glass transition. Another route could be the decrease of the maximum operational temperature that is actually $+85^\circ\text{C}$. Nevertheless, this way seems to be not compatible with the reduction of the filling time at the HRS.

Good impregnation: fibre volume fraction and variability

In general, there is variability of the impregnation during filament winding process. In general, the fibre volume fraction is close to 60% but, depending on the operating conditions, the level of local compaction, the fibre volume fraction value is not constant as reported in Figure 8. In consequence, locally in the composite wall along the vessel, there is a scattering on the ultimate strength of the composite.

The quality of the process is a key parameter to minimise this scattering. For that reason, to minimize the scatter of fibre volume fraction, towpreg winding is the best solution rather than wet winding.

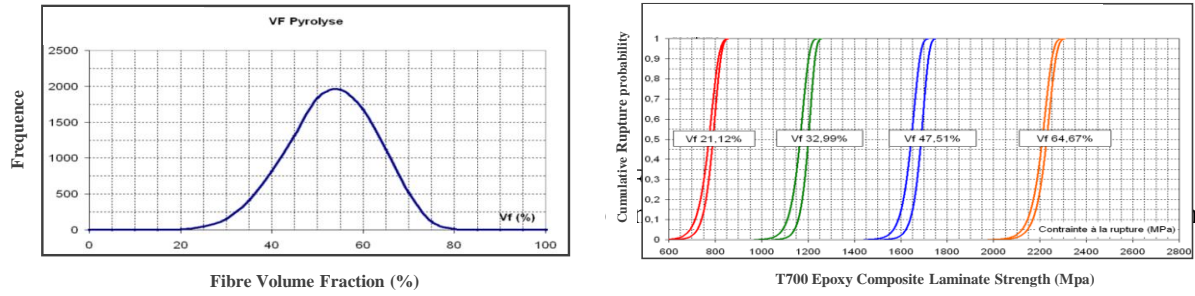


Figure 8: Fibre volume fraction variability and consequence on T700 – epoxy laminate performance distribution [8]

Good compaction: Porosity and variability

In general, the composite laminates of 70 MPa H₂ CPV are manufactured by wet filament winding. The level of porosity of composite laminate of CPVs is around 3% to 6% - 8%, Figures 9 & 10. This level of porosity is not acceptable in aeronautic application where the safety factor is equal to 1.8 with level of porosity <1%. One of the consequences of the porosity is a decrease of shear strength of the laminate and also initiation point for damage mechanism. The level of porosity can be directly linked to the linear speed of filament winding and also the quality of the impregnation bath for wet winding. Under 1% or 0.5%, there is very low consequence of the porosity on laminate performance. Over 1%, the mechanical properties of the composite part are significantly affected [11] and in particular the interlaminar shear. In fact, the resistance to Inter Laminar Shear Stress (noted ILSS), is very sensitive to the presence of these porosities (gas inclusions). The average decrease in ILSS was estimated to be an average of 6% per unit of porosity rate for carbon/epoxy laminates, Figure 11. The CPV dome region is very sensitive to ILSS. In general, the burst initiation of optimized 70 MPa CPV is localized in this area. The porosity in this area should be very low, under 1% for example.

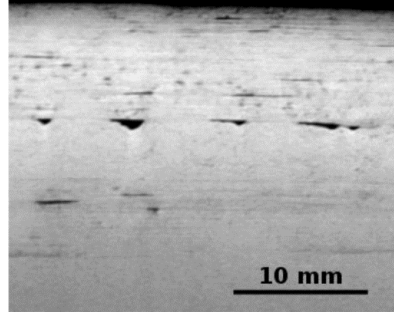


Figure 9: Tomography scan: region of interest scan (44 μ m voxel size) [12].

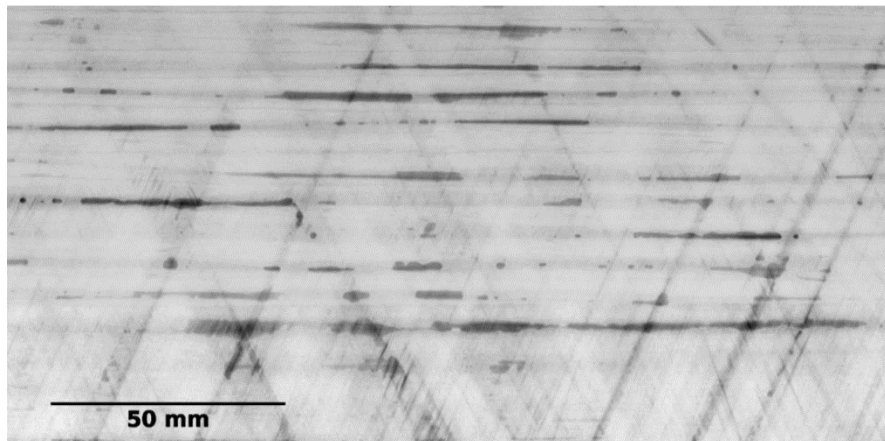


Figure 10: Large voids located at an interface between two plies. Sum projection across 30 slices (approximately 3 mm) [12].

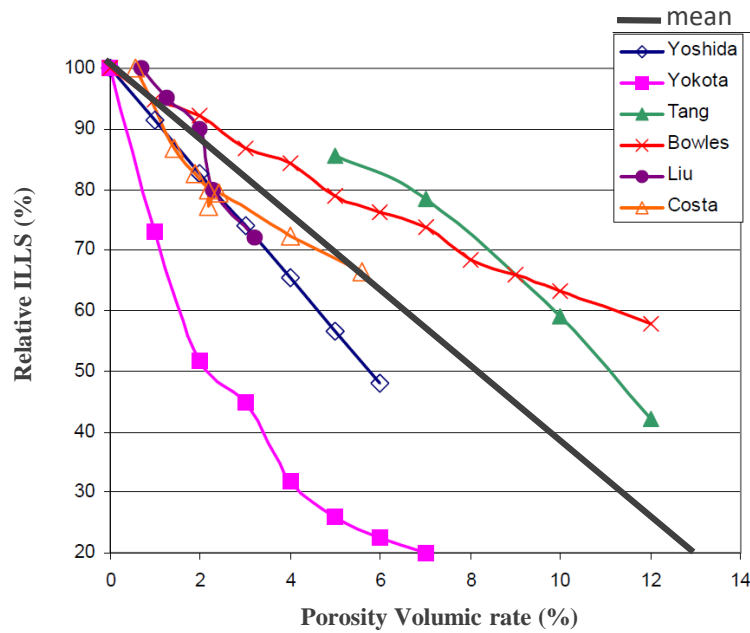


Figure 11: ILSS relative decrease with level of carbon-epoxy laminate porosity [11]

3. SAFETY FACTOR OF A 70 MPa H₂ CPV FOR ON-BOARD APPLICATION

Today, the value of the burst pressure ratio is equal to 2.25, Figure 12, for on-board carbon composite H₂ pressure vessels at initial life [1][2][9]. For a 70 MPa pressure vessel, the minimum burst pressure at initial life is 157.5 MPa.

The pressure operational range is from 2 MPa to 87.5 MPa. 87.5 MPa is the value of the maximum pressure during the filling at the HRS.

The value of the safety factor (SF) can be defined as the value of the minimal burst pressure (157.5 MPa) divided by the value of the maximum pressure allowed during operational life of the vessel (87.5). The value of SF is 1.8, Figure 12.

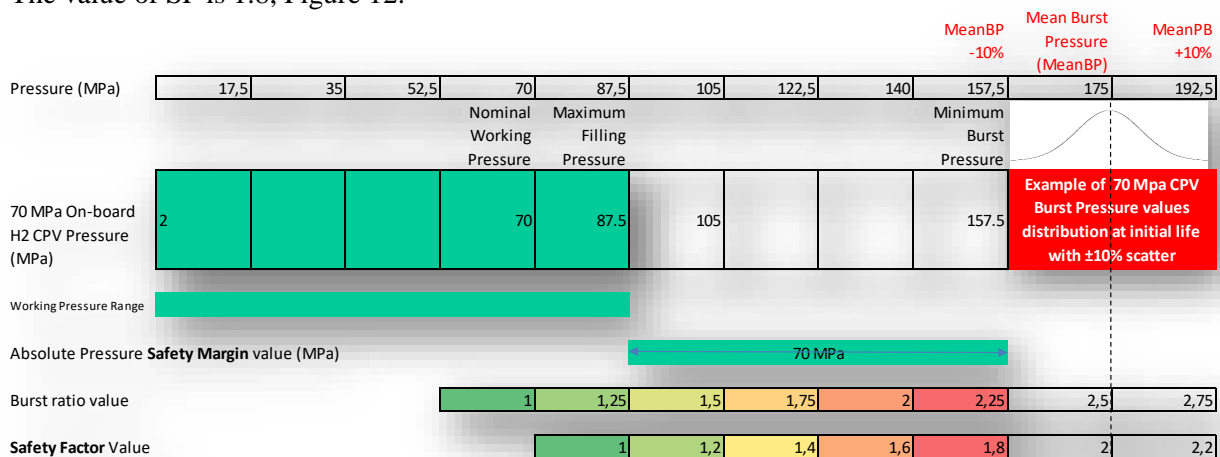


Figure 12: 70 MPa safety margin of composite material for 70 MPa on-board H₂ carbon CPV

Few test of the CPV certification [1] take into account a constant temperature and a constant humidity effect at sample or CPV scales.

- Resin shear strength test shall be performed [1] on a “representative” composite material. After boiling in water for 24 hours the minimum shear strength of 3 samples of composite shall be 13,8 MPa.
- During the CPV certification [1], an accelerated stress rupture test is required on one vessel. The vessel is pressurised to 1.25 times nominal working pressure (87.5 MPa for a 70 MPa vessel) for 1 000 hours at 85 °C. The CPV shall achieve a burst pressure greater than 133.9 MPa (Burst ratio = 1.91, Safety Factor=1.53).
- One another test, on one vessel also, is required at extreme temperature pressure cycle test at constant temperatures (injected liquid temperature, external surface temperature: -40°C and +85°C) and relative humidity $\geq 95\%$ for +85°C cycling test. After, a burst test is performed and the CPV burst pressure shall be greater than 133.9 MPa (Burst ratio = 1.91, Safety Factor=1.53).

During the pre-normative HYCOMP project [10], the determination of the safety factor of the carbon-epoxy composite material itself (UD: Uni Directional composite), called intrinsic-safety factor. The value of this safety factor taking into account the variability of the carbon fibre and the damage mechanism is 1.6 for the material itself, Figure 13, without consideration of temperature, humidity, porosity, fibre volumic ratio. This value is very high.

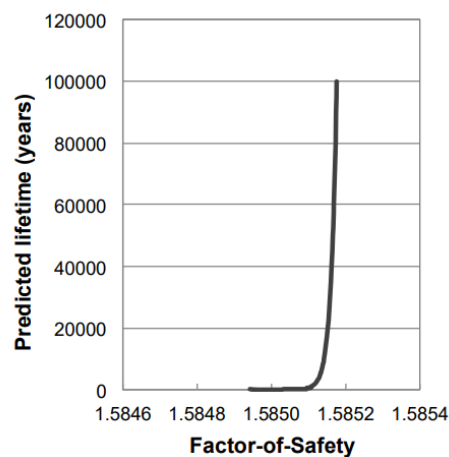


Figure 13: Hycomp Predicted lifetime versus factor-of-safety for UD carbon-epoxy laminates.

It is observed, during the Hycomp experiments but also with finite element calculations, a rapid increase in lifetime when the applied stress level on the composite decrease. The predicted lifetime curve of carbon-epoxy material itself is shown on Figure 14. Accounting only for the effect of variable material strength on its lifetime, taking into account the shape of the curve, for 70 MPa H₂ on-board CPV subjected to the long-term steady load-to-rupture, a safety factor limit value of 1.6 has been determined for a lifetime greater than 20 years.

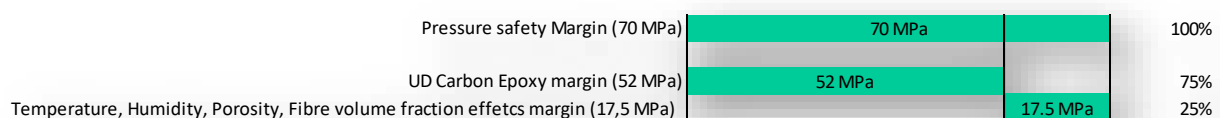


Figure 14: Detailed safety margins of composite material for 70 MPa on-board H₂ carbon CPV

Other aspects such as environmental damage should be taken into account for evaluating the overall safety factor value. On 1.8 safety factor 70MPa H₂ CPV credit, 1.6 is used by the UD carbon composite material itself. 0.2 is remaining to take into account effects due to the translation from Unidirectional Material to composite structure, temperature effect, humidity effect, porosity effect, oxydation or hydrolyse, Figure 15.

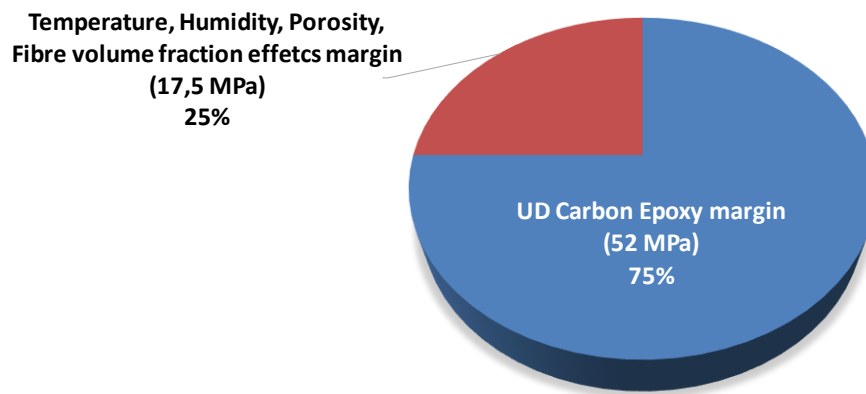


Figure 15: Hycomp safety margin decomposition for 70 MPa on-board H₂ carbon CPV

It should be welcome to introduce in the standard and the regulation special prescriptions on a minimum T_g (like ISO 19881) for temperature and humidity effects but also consideration on the level of porosity and fibre volume ratio of the laminate.

It should be necessary to justify more experimental statistical performance of pressure vessel to confirm or not 1.6 factor due to the carbon-epoxy material itself. For a lifetime of 15 or 20 years, it should be interesting to evaluate the safety margin linked with temperature, humidity and porosity.

For each type of vessel manufactured and certified, it should be necessary to evaluate the contribution of each previous parameters on the composite structure safety factor.

4.0 CONCLUSION

More and more 70 MPa H₂ composite pressure vessel will be used in public area during 15 or 20 years lifetime, around ten millions in 2030 for on-board applications (car, truck, train, bus...).

In this document, details on the on-board 70 MPa H₂ CPV technology and project experimental and computational results on composite wrapping have been reported to provide a better understanding of 2.25 burst pressure ratio equivalent to a safety factor equal to 1.8.

Considering Hycomp project experimental-based results on the common composite material used for this type of vessel, carbon-epoxy, the value of safety factor taken into account to prevent from mechanical burst hazard is equal to 1.8. On this 1.8 credit, 1.6 is due to intrinsic-safety factor of the composite material itself, at the uni-directional scale. A safety factor of 0.2 is remaining to take into account during the lifetime of the vessel the effects structure compare to UD, the effects of temperature, humidity and composite quality (porosity, fibre volume ratio).

Faced with this situation, it seems more than necessary to increase the knowledge on the behaviour of the material under conditions close to the use. More specifically, it would be interesting to carry out tests of thermo-mechanical or hydro-thermo-mechanical fatigue by combining water, thermal and

mechanical oscillations making it possible to apprehend, statistically, damage kinetics on materials such as tanks (with or without defects). It will then be possible to verify that this material contribution does not increase the present value of the safety factor for a life of 15 or 20 years. Results from Maxime Bertin [13] show that this cross cycling can generate accelerations of degradation. The question is open.

The other question concerns the residual resistance at the end of life. Since ageing at 20 years has reduced the safety factor, what will be the resistance to an exceptional mechanical stress of the impact type, for example? This last point should be addressed using aging acceleration techniques to validate tanks in service during 20 years lifetime.

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