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Deliverable Report

WP6
Final WP6 report to be included as appendix of WP7 report – Recommendations for Industry and RCS

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TABLE OF CONTENT

1. Executive summary

1.1 Summary of deliverable content and initial objectives

The purpose of this deliverable is to summarize the work performed in WP6, and the main results obtained. The first task was to review published and draft Regulations, Codes and Standards (RCS) for all applications covered by HyCOMP (stationary, transportable and on-board). Based on this review, requirements were analyzed and compared in order to identify specific information that HyCOMP has to address.
Then, based on the results obtained in WP2 to WP5, recommendations have been proposed. They cover different topics, such as design requirements and testing procedures for type approval, manufacturing assurance quality and in-service inspection. A list of 8 recommendations is proposed in this deliverable with the corresponding explanations coming from experimental results.

1.2 Partners involved

All partners were involved in this Work Package, especially: Air Liquide, CCS, Hexagon, Faber, CAQ and academic partners.

1.3 Relation with others WPs / Tasks

This deliverable makes the link between all WPs of the project: from WP2 to WP5. Indeed, WP6 is the work package where we gather all the results coming from each WP in order to propose new design requirements and new testing procedures.

2. Synthesis of existing requirements for cylinders and identification of gaps and possible improvements

The objective of the first task in WP6 was to identify the existing requirements for composite cylinders for all applications (transportable, on-board and stationary). Based on this comparison, gaps and/or improvement chances for some of the specific tests were pointed out, and a list of questions addressed to each work packages was established.

2.1 List of standards / regulations

For each application, following standards and regulations have been analyzed and compared:

- Transportable:
    - **ISO 11119-1** Gas cylinders - Refillable composite gas cylinders and tubes - Design, construction and testing - Edition 2. Part 1: Hoop wrapping fibre reinforced composite gas cylinders and tubes up to 450 L
    - **ISO 11119-2** Gas cylinders - Refillable composite gas cylinders and tubes - Design, construction and testing - Edition 2. Part 2: Fully wrapped fibre reinforced composite gas cylinders and tubes up to 450 L with load-sharing metal liners
    - **ISO 11119-3** Gas cylinders - Refillable composite gas cylinders and tubes - Design, construction and testing - Edition 2. Part 3: Fully wrapped fibre reinforced composite

gas cylinders and tubes up to 450 L with non-load-sharing metallic or non-metallic liners

- **ISO 11515** Gas cylinders – Refillable composite reinforced tubes of water capacity between 450 L and 3000 L – Design, construction and testing
- **ISO/CD 17519** Gas cylinders – Permanently mounted composite tubes for transportation

- **EN 12245**:2009+A1 Transportable gas cylinders – Fully wrapped composite cylinders
- **EN 12257**:2002 Transportable gas cylinders – Seamless hoop wrapped composite cylinders

- European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR)

- On-board:
  - **ISO/TS 15869** Gaseous hydrogen and hydrogen blends – Land vehicle fuel tanks. Edition 1
    - Note: This document is not applicable as standard, but as a Technical Specification (TS)
  - **Global Technical Regulation No. 13**, EC/TRANS/180/Add.13, Global technical regulation on hydrogen and fuel cell vehicles, United Nations, July 2013
  - **79/2009/EC** and **EU 406/2010** of 26 April 2010 implementing Regulation (EC) No 79/2009 of the European Parliament and of the Council on type-approval of hydrogen-powered motor vehicles
    - Note: In the absence of approved standards, the regulation contains more technical requirements than it normally would if there had been an approved standard to be used as a reference document in the regulation.

- Stationary:
  - **ISO/CD 19884** Gaseous Hydrogen – Cylinders and tubes for stationary storage Edition 1 (changed from ISO/CD 15399)
    - Note: The scope of this standard is under discussion between experts. In the absence of regulations and approved standards, there is no international standard supporting the European pressure equipment directive (PED) regulating the design of stationary storage.

2.2 Discussion and conclusions

In general, the same questions apply for all applications whether the composite cylinders are designed for onboard, stationary or transportable applications. Below are listed several questions that point out the lack of rationale in standards.

2.2.1 Service conditions

In all cases, there is a need to properly assess the service conditions that apply for the specific applications. It is therefore important to define all the conditions that each type of cylinders is likely to experience during its service life such as number of pressure cycles per year, amplitude of pressure cycles, temperature extremes, environmental conditions, and expected service life. Since these service conditions differ whether the cylinder is designed for onboard, stationary and transportable applications, it is important to really establish the service conditions that are representative for the application.

2.2.2 Qualification tests & design requirements

Once the service conditions are known, it is important to define a series of qualification tests that will ensure that all cylinders that are manufactured as per the reference design will remain safe during their whole service life. These series of qualification tests will differ based on the applications. Currently, the approved standards covering cylinders used in transportable applications do not include requirements for stress ratio, plastic material requirements and metallic boss requirements that are included in the standards for onboard and stationary applications.

As part of the exercise of defining the series of qualification tests for each application, there is a need to determine the strength properties of the cylinders and their scatter in order to determine the safety margin that should be considered as well as the number of samples that should be tested to have a proper evaluation of the whole population of a specific design.

Since the rupture of the storage container is the most important failure that has to be avoided, it is important to define a series of qualification tests that will ensure that the cylinder does not fail due to stress rupture, fatigue or environmental loads. As part of these requirements, there is a need to determine the stress ratio that will prevent stress rupture under representative loading for the expected lifetime and a test method that will demonstrate the ability to withstand elevated loads for whole service life. There is also a need to define the pressure cycle tests that will reliably ensure absence of rupture due to pressure cycling.

Since we are dealing with cylinders that will be filled with hydrogen, there is a need to determine how material compatibility is ensured. In particular, is a hydrogen gas cycling test justified and relevant for type 4 cylinders in the qualification test program? And should a hydrogen gas cycling test be performed on all types of cylinders as opposed to only type 4 as it is the case in all standards right now? This question has been raised in WP3. For both Type 3 and Type 4 cylinders, 10 cylinders were cycled hydraulically, 5 at room temperature and 5 at elevated temperature. Then 4 Type 3 cylinders and 3 Type 4 cylinders were gas cycled on the Gastef facility at JRC for comparison with hydraulic cycling. Test results give no significant indication that gaseous cycles are more critical than hydraulic ones – in the way they have been performed. A possible influence of gaseous versus hydraulic cycling has been indicated for Type 3 cylinders. This is a preliminary result, and more tests are needed to confirm the conclusion.

2.2.3 Batch tests

The batch tests that are performed to confirm conformance with the reference design (qualified in the type approval process) should be re-assessed for their suitability. More specifically, the key process and materials parameters that have a non-negligible effect on cylinder performance (on the short-term and long-term) must be properly monitored. It must be demonstrated in batch tests that these parameters do not deviate too much from qualification tests.
In WP2 and WP4, parameters having a strong influence on cylinder performance have been identified. From the results obtained, recommendations are given for batch tests.

2.2.4 In-service inspection

Finally, there is a need to determine what kind of in-service inspection should be performed on the composite cylinders to assess potential degradation of the cylinder in use, the pass/fail criteria for the cylinder to remain in service and the required frequency for these in-service inspections. In this case as well, it is important to determine if the requirements should differ for onboard, stationary and transportable applications.

NOTE: A special focus on on-board storage requirements has been proposed by JRC-IET. The comparison includes not only ISO standard and the EU 406/2010 regulation, but also the SAE work and to a certain extent the related Global Technical Regulation 13 of the UN-ECE. The objective was to map more exhaustively the present international standardization and regulation frame.

2.3 Questions addressed

Based on the analysis of existing requirements, specific questions were expressed and addressed to each work package to orient testing program and expected outcomes.
Here are the questions addressed to WP6:

Questions	State of the Art – Current standards
Stress ratio What is the right stress ratio to reliably ensure absence of burst, assuming adequate material and manufacturing?	Stress ratio requirements are based on stress rupture studies conducted in the 1970s and 1980s, with general confirmation provided by field service statistics. It has been suggested that such stress rupture studies should be conducted again with a broader scope of materials.
Design validation What is the most appropriate test protocol (effectiveness, safety, efficiency, reproducibility) for demonstrating fitness for the anticipated service? • What are the test conditions and criteria to be applied for ensuring absence of failure (specify failure mode) for anticipated service, considering scatter of performance? • What is the criterion on measurable state of damage determining fitness for service? Another approach is needed for demonstrating reliability (scatter)	Scatter of performance, which may affect reliability, is not considered directly. The manufacture sets the mean at a level above the minimum to ensure the mean, less deviation, stays above the minimum. Lack of knowledge about the relationship between test conditions and service conditions. Fitness for service is not defined. State of cylinder after a period in service is often evaluated by hydraulic proof test. Some fuel container standards recommend visual inspection. Some standards and regulatory officials are giving consideration to NDE methods, such as ultrasounds, acoustic emission, or holography for inspections.
Control of manufactured products What is the most appropriate test protocol for checking that manufactured cylinders will behave as observed under type approval? According to what sampling rules? With what pass/fail criteria? (cf. WP4)	Hydraulic proof test and burst of sampled cylinders; will not necessarily allow detecting vessels unfit for service as a result of most likely manufacturing deviations.
Periodic inspection testing in service	Hydraulic proof test and visual

What is the most appropriate test protocol for inspection in service? With what pass/fail criteria? At what frequency?	inspection - Not designed to detect potential abnormal damage accumulation making cylinder unfit for service. Ultrasounds and acoustic emission are sometimes used for periodic inspection.

3. Analysis of existing design requirements & Terminology

For the following parts, notations mentioned below are used.

Notations:

NWP: Nominal Working Pressure is the pressure at 15°C. Sometimes, NWP is also called service pressure (P_s).

SF: Safety Factor, defined as the ratio between the burst pressure and the Design Pressure.

GSF: Global Safety Factor, defined as the ratio between the burst pressure and the Nominal Working Pressure.

Note: Global Safety Factor is also called Burst Pressure Ratio (BPR) for on-board storage.

DP: Design pressure: Pressure for which cylinder is designed, defined as DP = 1.5*NWP

Note: the Design pressure is also known as test pressure, hydraulic proof test pressure and maximum allowable working pressure (MAWP).

T_g: Glass transition temperature of the epoxy resin. This is the is the temperature range where a thermosetting polymer changes from glassy state to a “rubbery” state

Note: Resin properties can vastly differ depending on the curing process, curing temperature, and time cured. A good curing is essential to reach the highest thermal performance characteristics of the resin.

Figure 1 presents in a schematic way design requirements of existing standards, in terms of Burst Pressure Ratio, for transportable and on-board storage.

For transportable storage (ISO 11119 parts 1, 2 and 3), reference pressure is the Design Pressure (DP), whatever the type of gas transported (general service) and test pressure (P_h) is defined to be equal to Design Pressure. It corresponds as well to the Maximum Allowable Working Pressure (MAWP). This means that a cylinder can be filled up to P_h, if the maximum temperature in service defined by the manufacturer is not exceeded.
Nominal Working Pressure (NWP) is defined as 2/3 of P_h for general purpose cylinders at 15°C. NWP is used as reference point for calculation of storage capacity of the cylinder
A safety factor (SF) is specified as the ratio of minimum burst pressure over Design Pressure. The value of this safety factor depends on the type of cylinder:

- For metallic cylinders (Type 1), SF = 1,6.
  - This value has been defined based on field experience and compromises between by ISO experts.
- For hoop wrapped cylinders (Type 2), SF = 1,67.
  - This value has been increased compared to metallic cylinders, because at that time behavior of composite materials was unknown. In type 2 cylinders, composite wrapping is used in low quantity as reinforcement in the cylindrical part only. Most of the pressure load is ensured by the thick metallic liner.
- For fully wrapped cylinders (Type 3 and 4), SF = 2.

- o In this case, SF value has been significantly increased compared to Type 2 cylinders, mainly because the whole pressure load is ensured by the composite wrapping, and degradation effects over the lifetime of the cylinder was not known.
- o It must be mentioned that no distinction is made between the different types of fibers.

Combination of both “sub-factors” gives the Global Safety Factor related to the Nominal Working Pressure. This is the minimum burst pressure expected for a burst test.

For on-board storage (EU 406/2010), pressure reference is not the Design Pressure, but the Nominal Working Pressure.
The Maximum Allowable Working Pressure is defined as 1,25*NWP, with a maximum temperature in service of 85°C.
In this case the definition of Burst Pressure Ratio is equivalent to the definition of the Global Safety Factor for transportable storage, and specified for carbon fibers as 2,25. By deduction, the equivalent safety factor of on-board storage as defined by EU 406/2010 is 1,8 (to be compared to the value of 2 for transportable storage).

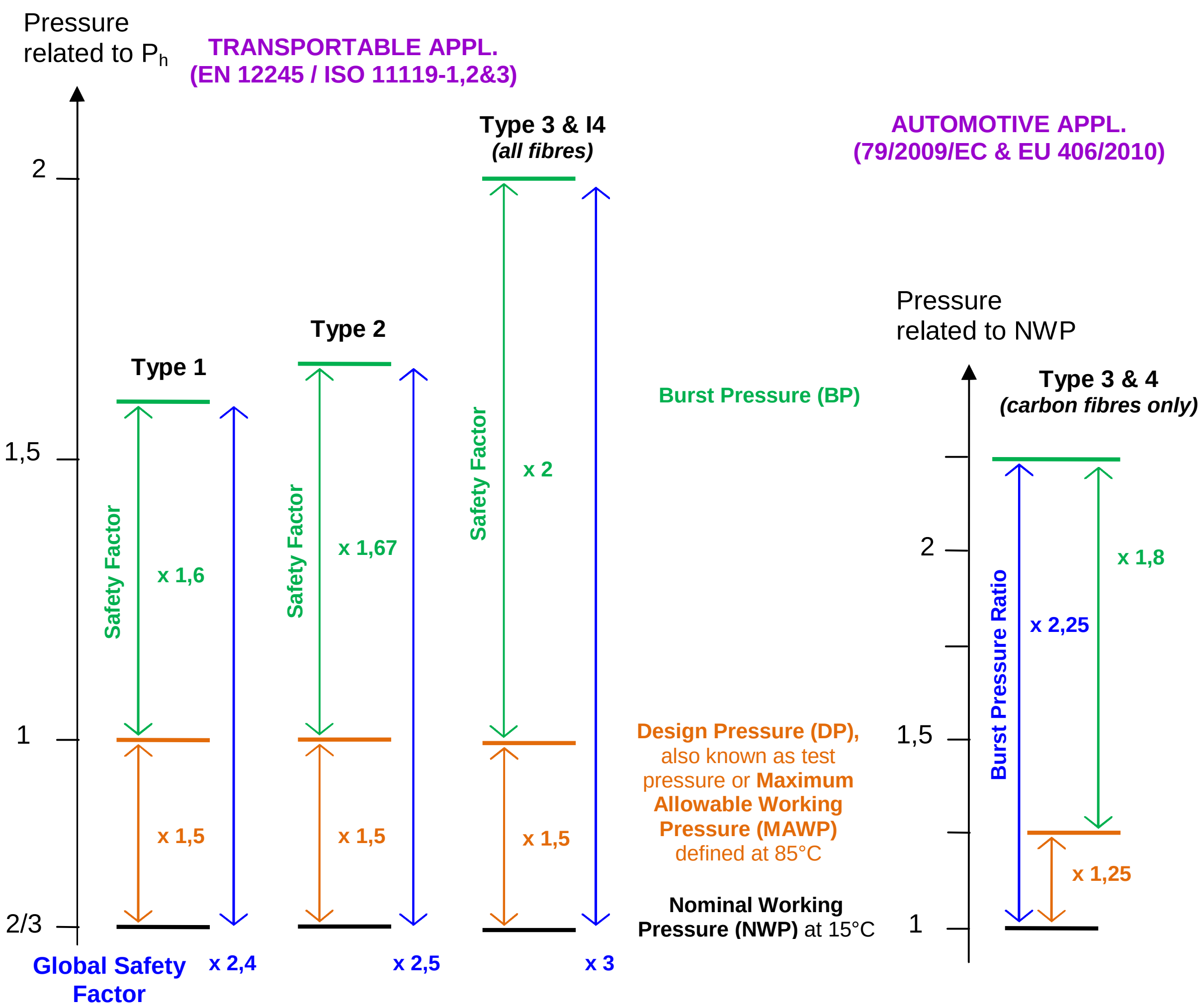

Figure 1 : Schematic graph of definition of different pressures and safety factors in current standards for transportable and on-board application.

4. List of recommendations based on HyCOMP results

4.1 General recommendations

Before proposing recommendations extracted from HyCOMP results, some general recommendations related to existing ISO standards have been unanimously raised by partners:

- Clearly define the scope of each standard with respect to service conditions
    - Adapt the standard to a specific application (stationary, transportable, on-board). In particular expected number of filling cycles and maximum developed pressure must be specified.
- For each standard, need to clearly define the purpose of each test. The rationale behind each test should be explained in an informative annex.

If added into existing standards, these explanations would allow knowing how current standards have been built and how test conditions and criteria have been chosen. For a targeted application, for which service conditions are well defined, testing conditions could be adapted to fit with the purpose of cylinders in service: more cycles in a cycling test, reduced test pressure, higher maximum temperature in service, etc…

4.2 Design requirements

Design requirement, based on Safety Factor or Burst Pressure Ratio, shall ensure the structural integrity of composite pressure vessels throughout their service life and specified service conditions.

As exposed in Figure 1, the Global Safety Factor or Burst Pressure Ratio can be divided into two different factors: one defining the Design Pressure (related to the NWP), and one which is “pure” margins applied to the Design Pressure defining the minimum burst pressure required. This last factor is called Safety Factor in this document.
The following parts propose recommendations for both “sub-factors” of the Global Safety Factor.

4.2.1 Recommendation 1

Proposal:
Use the Maximum Developed Pressure as the Design Pressure (instead of Test Pressure, see Figure 2).

Consequence: Pressure Receptacles will be designed for dedicated service (specific gas). Type of gas has to be specified in type approval certificate and on the cylinder label.

Rationale:
For certain gases, the maximum developed pressure at the maximum temperature is much lower than the pressure for which the cylinder has been designed (P_h). Hydrogen shows a very low expansion compared to other gases. At 65°C (resp. 85°C), the developed pressure is 1.18*NWP (resp. 1.25*NWP), much lower than the actual Design Pressure (1.5*NWP).
For those specific gases, it should be possible to lower the Design pressure to the Maximum Developed Pressure at the maximum temperature, in order to avoid unnecessary margins.

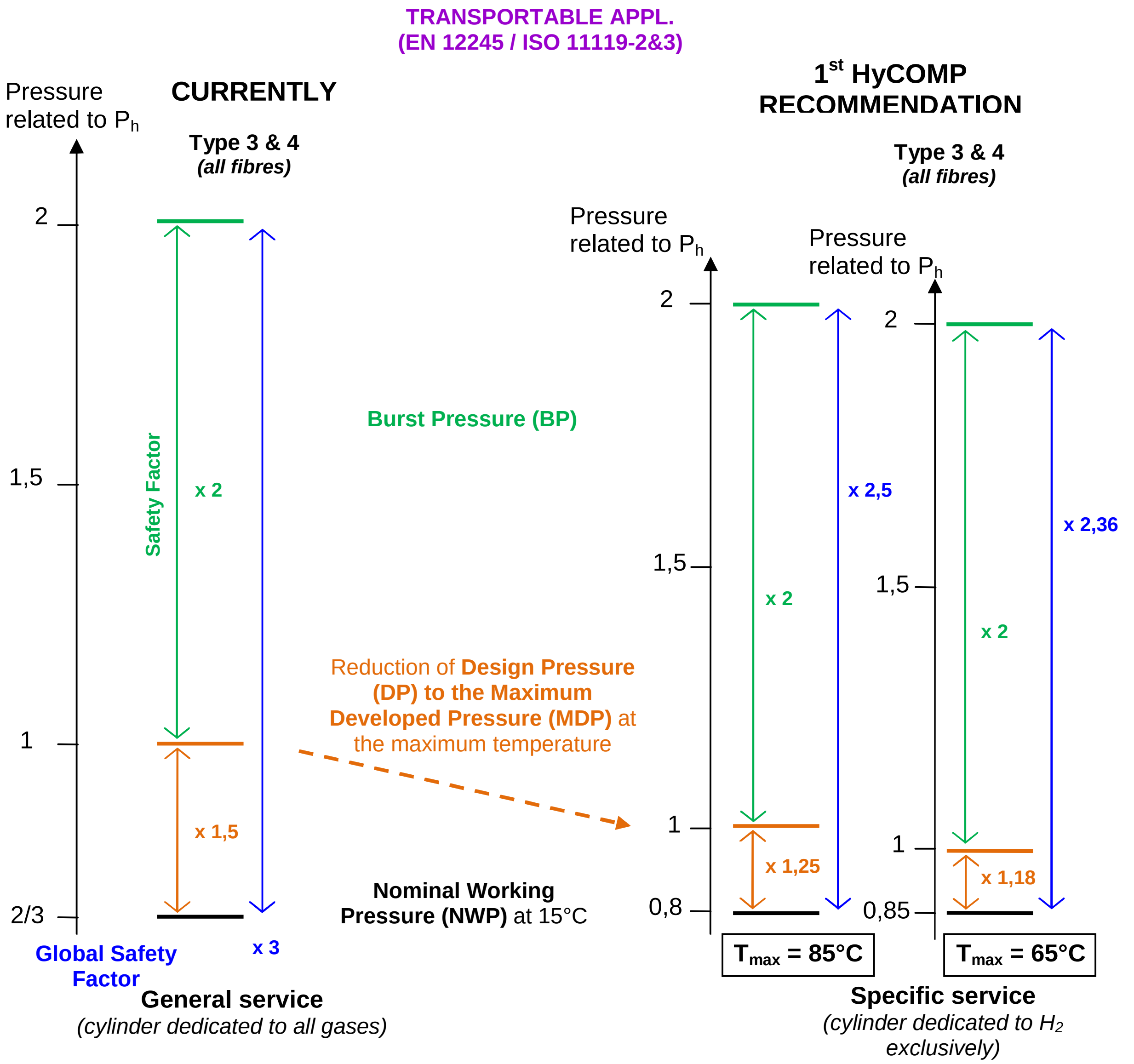

Figure 2 : Schematic graph illustrating recommendation n°1, related to the change of Design pressure to the Maximum Developed Pressure at the maximum temperature

The purpose of HyCOMP was to demonstrate a possible reduction of the Safety Factor while ensuring the structural integrity of composite pressure vessels throughout their service life.
For this purpose, the objective was to better understand damage accumulation rate at a micro level. Prediction of damage accumulation under loads encountered in service (static, cycling) and effect of environmental conditions (temperature humidity) will allow the optimization of cylinder design through performance-based design requirements.

> _Note_: A different approach has been studied in HyCOMP, proposed by BAM and supported by HEX. This approach is based on a Probabilistic Assessment of test results. This approach has been largely discussed together with the DeliverHy project, especially with NTNU in Norway who developed the same kind of approach. In few words, it recommends to have a safety factor depending on the coefficient of variation (also known as scatter) and taking into account degradation during cylinder lifetime.
> This approach seems promising but needs further research on the issue of kind of distribution, influence if sample sizes and the legal requirement for acceptable unavoidable failure rates.

In the following recommendation, we will focus on the initial approach followed in HyCOMP, as described in the Description of Work.

4.2.2 Recommendation 2

Statement:
HyCOMP has performed theoretical studies on flat panels and specific materials (carbon fibers) demonstrating that SF covering intrinsic material properties can be down to 1.4 for sustained loading, given an unlimited lifetime. This value covers intrinsic material properties only, and is called intrinsic Safety Factor (iSF). For composite cylinders, others factors shall be taken into account to cover other aspects.

For transportable applications, **there could be a potential to reduce the minimum SF from 2**, if justified by further test results such as type approval tests. Design criteria might depend on the lifetime of the cylinder. For other applications, 1.8 is already used (refer to EU 406/2010, JARI S001, GTR 13).

Additionally, to get the type approval, cylinders must **successfully pass all the other tests** as defined in standards for the different applications

Consequence:
If a cylinder designed with the minimal required Safety Factor fails to pass a test, due to a failure of the composite structure, it is then necessary to increase the Safety Factor to pass this test. Therefore, the final Safety Factor corresponds to the maximum value required to pass all the tests of the qualification test program.
This final Safety Factor characterizes cylinder design and must be kept for manufactured batches.

Rationale:
The value of 1.4 must be seen as the minimum theoretical value covering intrinsic material properties (variability of carbon fibre properties).
It was determined on plate specimens manufactured by filament winding with the specific materials studied in HyCOMP:
- Resin: Tg = 115°C (measured by DSC),
- Carbon fiber: E = 230 GPa, ε = 2,1%, UTS = 4,9 GPa

The methodology to determine this value has been described by Armines in deliverable D2.4. It is based on the measurement of damage accumulation (by acoustic emission) during static tensile loading tests of unidirectional composite specimen. The results show that a specimen can be loaded continuously up to 71% of its ultimate tensile strength (meaning a SF of 1.4), without any rupture for an infinite lifetime. A probability of failure of 10^{-6} is considered. A value of 1.6 is found for a probability of failure of 10^{-9}.
This value covers the **maximum degradation rate of tested CFRP-material under sustained loading and ambient conditions on flat panels**.

It still needs to be proven whether this result can be extended to composite cylinders or not, and whether filament wound structures behave locally like unidirectional composites when they are pressurized (due to the geodesic paths of carbon fibres). Concerns have been raised that unidirectional/flat panels do not have the same 3-dimensional loading and response and therefore can't model the effect of curvature in a cylinder, i.e. thick wall effects. Hence, additional margins taking into account structural effect (from micro to macro scale) must be considered but are difficult to quantify.

> *Note*: In the absence of rationale for other fibres, it must not be **applied to other fibres than carbon fibres**.

Parameters like temperature and humidity have been studied on specimens in WP2 (see Deliverable D2.4). It has been demonstrated that temperature has an important effect on damage accumulation rate. Unfortunately, safety factor assessment at high temperature would require much more tests on specimens. It has not been possible in the frame of the project to perform this complementary study.
As temperature influence is assessed by a performance-based test at extreme temperatures, it would be redundant to include temperature effects in the safety factor value. By consequence, safety factor value is defined at ambient conditions, and cylinder performance at elevated temperature is assessed by the qualification test program (see recommendation n°5).

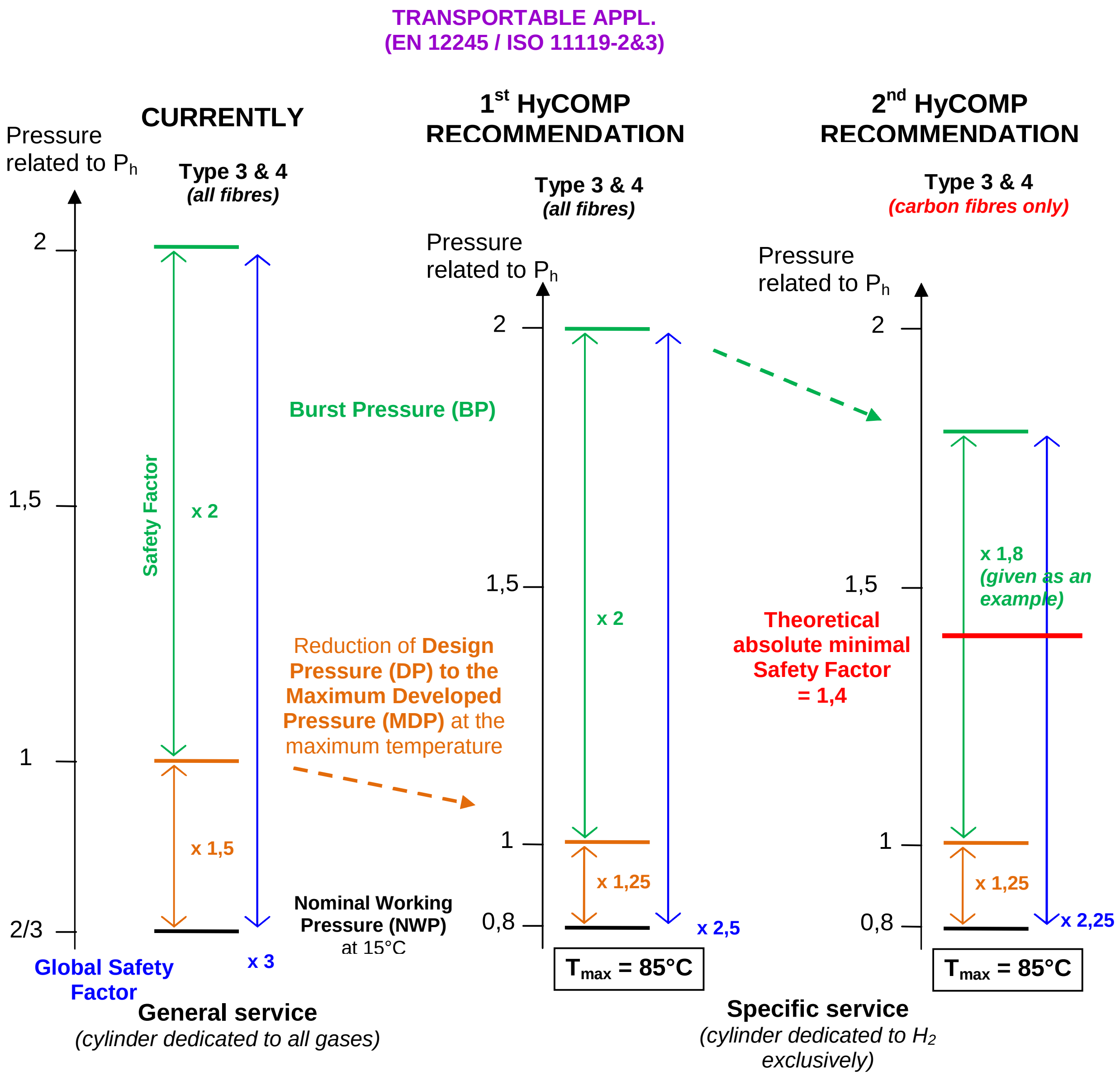

Figure 3 : Schematic graph illustrating recommendation n°2, related to the potential reduction of Safety Factor

Remark: The value of 1.4 covers degradation of fibre strength variability, but not manufacturing variability of cylinders. The value of 1.8 is an exemplification value figuring out the intended tendency. This value is currently valid for fixed mounted, well protected automotive cylinders with a limited life time of 20 years,

4.2.3 Recommendation 3

Proposal:
Add specifications on glass transition temperature of the composite (to be verified during type approval and controlled during quality assurance)

Example: T_g must be higher than T_{max} + 30°C (based on composite specimens tested in HyCOMP)

Remark: If T_g has to be measured, the type of test to determine T_g must be specified, as the value is highly dependent on the method.

Rationale:
WP2 demonstrate that temperature is an important parameter influencing damage accumulation (fibre breaks) in the composite wrapping. As we can't change operational conditions (maximum temperature in service), it is important to ensure that difference between T_{max} and T_g is sufficient so that damage does not accumulate too quickly when temperature is getting close to T_{max}.

4.3 Testing procedures for type approval

4.3.1 Recommendation 4

Proposal:
Make a statistical assessment of the key property(ies) of the composite, respectively cylinder.

Rationale:
Experimental results on cylinders have shown that degradation can get assessed or reliably demonstrated, only when mean value and scatter of properties are taken into account.

4.3.2 Recommendation 5

Proposal:
Tests at elevated temperature should be performed **at a temperature not less than T_{max}** defined for the application

Rationale:
It has been demonstrated in D2.4 that temperature has an important effect on damage accumulation rate, as viscoelastic properties of the resin, influencing fibre breaks, are directly affected. The glass transition temperature (T_g) of the resin is a key parameter. When temperature reaches 25°C below T_g, damage accumulation starts to increase drastically. It is necessary to ensure that difference between T_{max} and T_g is enough so that damage does not accumulate too quickly when temperature is getting close to T_{max}.
Tests at elevated temperature in the qualification test program are set to demonstrate the proper choice / curing of the resin (relative to T_g). By consequence test temperature conditions must be properly chosen to ensure that resin will withstand elevated temperatures in service, up to T_{max}.

4.3.3 Recommendation 6

Proposal:
Perform all relevant tests at the maximum developed pressure at T_{max} (as defined in recommendation 1).

Note: This recommendation is **valid for transportable application**.

Consequence:
Change test pressure to the maximum developed pressure, instead of P_h = 1,5 * NWP, or only NWP.
In ISO 11119-3, this list includes:
- Ambient temperature cycle test
- Environmental cycling test (hot cycle test phase only)
- Cycling test on flawed cylinder: currently performed at NWP for 5 000 cycles
- Cycling after a drop test: currently performed at NWP for 12 000 cycles

Rationale:
As cylinders will never be pressurized beyond the maximum developed pressure (defined at the maximum temperature in service), it is sufficient to demonstrate that cylinders will withstand the most severe loads in service (not more, not less). However, there is no reason that a flawed cylinder or a cylinder exposed for an external impact cannot be pressurized beyond the NWP.

Note: Hydraulic proof test must still be performed at 50% above the nominal working pressure.

4.4 Batch testing procedures for Manufacturing Quality Assurance (MQA)

4.4.1 Recommendation 7

Proposal:
Control the resin mixture and curing process (by monitoring temperature, time…) and/or consider tests in standard to verify the good curing of the resin.

Example: Perform a Barcol hardness test on each cylinder after curing.

Barcol test: Often used for composite materials to determine how much resin has cured. Inexpensive and quick.

Rationale:
It has been demonstrated in D4.4 that an improper curing of the cylinder causes an important effect on cyclic performance on Type 3 cylinders and on burst pressure scattering on Type 4 cylinders. This confirms that curing quality is of high importance to ensure good performance of the composite wrapping. Therefore, there is a need of introducing a further control of this characteristic in the regulations, codes and standards, by specifying additional tests to verify the proper curing of the resin mix.

4.5 Methods and procedures for inspection in service

4.5.1 Recommendation 8

Statement:
Continue to develop Non-Destructive Techniques to carry out periodic inspection of composites pressure vessels.
Acoustic Emission (AE) seems to be a promising technique for in-service inspection of composite pressure vessels. Nevertheless **further research is needed** to have a technique fully operational, with universal pass/fail criteria. AE technique needs to be developed in close relationship with

mechanical impact study. Indeed removal criteria from service are mainly dependant of the severity of impacts that a cylinder may undergo in service. This was not the initial purpose of HyCOMP.

Rationale:
Actual standards and regulations prescribe for periodic inspection of a container to perform a visual inspection (external and internal) and a hydraulic proof test, whatever the type of cylinder. Hydraulic proof test consists of hydraulically pressurize the cylinder at 1,5*NWP during at least 30 seconds. No leakage must be observed. If a cylinder successfully passes this test, it is declared as appropriate for service.

Acoustic Emission technique is a recognized Non Destructive Technique (NDT) widely used for the identification of damage sources in composites materials (see Figure 5). It allows the discrimination of different types of damage leading to the rupture of a structure. It consists of hearing acoustic emissions coming from the material through piezoelectric sensors. Acoustic emissions are produced when the structure is mechanically loaded. Depending on acoustic characteristics (hit number, energy, frequency, amplitude, etc…), it is possible to characterize the type and level of damage. Furthermore a localization of damage is possible by a combined analysis of several sensors.

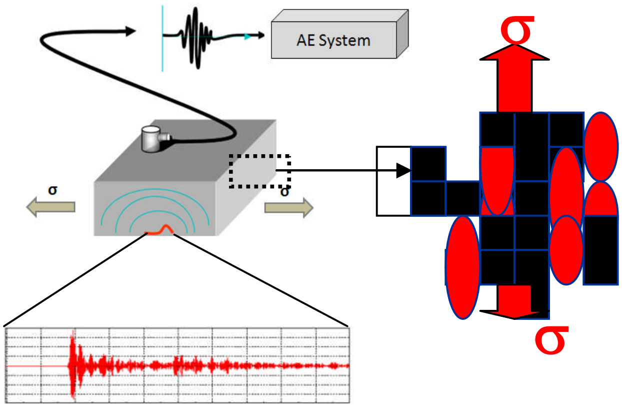

Figure 4 : Principle of Acoustic Emission testing

Acoustic emission has been largely used in HyCOMP by different partners, in different WPs and tasks. Nevertheless, it has been noticed that Acoustic Emission seems to be highly operator-dependant. This means that the way to use acoustic emission and to analyze results is specific to each operator.

In HyCOMP three different partners have used Acoustic Emission:
- Armines in WP2 on specimens
- WUT in WP3 (T3.4) and WP4 (T4.2) on cylinders
- BAM in WP4 (T4.3)

The initial intention was to apply the “master curve” approach to quantify the level of damage after preconditioning (cyclic or static test). This has been performed in WP2 by Armines, WP3 and WP4 by WUT. This approach is described in Annex 1.

Unfortunately, we faced experimental difficulties that did not allow us to propose a clear methodology for in-service inspection. Indeed, it is necessary to accelerate damage accumulation process to get a “master curve”. The question is how to correlate the master curve obtained in accelerated conditions to the real master curve in operating conditions. Furthermore, the difficulty to position a specimen on a master curve has been pointed out.

Another issue encountered was related to the equipment. Indeed, AE equipment differs from one supplier to another one. This can lead to significant discrepancies in AE results. A harmonization between the different suppliers would be useful, in terms of signal processing for example.

BAM used Acoustic Emission in another way. The purpose here was not to quantify level of damage in composite pressure vessels, but to detect any deviation from a reference batch. Here acoustic emission can be seen as a manufacturing monitoring tool for product inspection

Other technique investigated: Optical Fiber

The incorporation of Optical Fibre (OF) in Type 3 cylinders was successfully accomplished. The OF measurement during autofrettage showed the restrictions of the system. However the crack causing the failure of the cylinder was detected by this method. No investigation on how surface damages like an impact or scratches influence the measurements have been accomplished for this project but seem to be promising fields for future projects.
The test program did show a reduction of cyclic strength for the cylinders that were pre conditioned. OF measurements showed a change in the state of deformation and were interpreted as a loss of pre tension introduced during autofrettage. This is a service life determine influence for the type III cylinder.
The strain monitoring during the load cycle tests showed a continuously rise of the global deformation over LC. It seems to be important to evaluate this typical **behaviour of damage evolution in viscous** materials further in future projects in order to better interpret and understand the results of the OF measurements. So far it is not possible to derive a global strain criterion from this investigation for the surveyed cylinder design. But **for future investigations the global strain over LC appears to be promising for a life time assessment of type 3 cylinders as a monitoring system and a promising indicator for the inspection in service**.
A further result was obtained by the comparison of the location of the final failure at burst and cycling. Both showed no agreement. This is a strong hint that burst tests are not a good indicator for service life or reliability assessment of the type 3 design. The failure mechanism in service differs and therefore a safety factor for burst test is meaningless regarding a failure caused by service loads.

5. Conclusions

Existing standards and regulations for composite pressure vessels are many and varied depending on the targeted application (transportable, on-board and stationary). All of these standards and regulations are based on cylinder performance. Discrepancies between standards have been noticed. Furthermore, often no particular rationale is provided in standards to justify test conditions and pass-fail criteria.

A possibility to reduce over dimensioning is to **use the maximum developed pressure (MAWP) as the design pressure of cylinders with dedicated gas service (as e. g. exclusive hydrogen)**. This is justified by a relatively low expansion of hydrogen at elevated temperature. By consequence, cylinder becomes gas-dependant. All tests required by standards must be performed at this pressure: **Maximum Developed Pressure becomes the new design pressure**. This leads to a reduction of the minimum required burst pressure without reducing safety factor.

One possibility is to reduce margins also called Safety Factor in relation to the Maximum Developed Pressure. For transportable applications, **there could be a potential to reduce the minimum SF from 2**, if justified by further test results such as type approval tests. Lifetime of the

cylinder may influence the value used. For other applications, 1.8 is already used (refer to EU 406/2010, JARI S001, GTR 13).
An intrinsic safety factor of 1.4 has been demonstrated at a material level (plate specimens) to ensure an unlimited lifetime **under sustained loading and ambient conditions**. This value is an absolute minimum accounting for intrinsic material properties and does not cover scatter of initial strength.
For composite cylinders, others geometrical factors must be taken into account to cover other aspects

Additionally, cylinders must successfully pass all tests of the qualification test program. If a cylinder must be reinforced to pass a test by adding carbon fiber layers, this new value of safety factor is kept as the minimum requirement in serial production.

Furthermore it has been demonstrated that temperature has an important effect on damage accumulation rate, especially due to matrix relaxation effects resulting in a lower load transfer. This parameter is not taken into account in the intrinsic SF value, but is assessed by tests at elevated temperature (sustained tests and cyclic tests). To ensure the proper choice of the resin (relative to T_g), it has been proposed to **perform tests at elevated temperature at a temperature not less than T_{max}** defined for the application. To ensure cylinder quality related to this parameter, it is proposed to **add specifications on glass transition temperature of the composite to verify the suitability of the resin**.

Linked to the influence of temperature, it has been confirmed as well the importance of a proper matrix curing. Therefore, it is proposed to **control the resin mixture and curing process and/or to consider tests in standard** to verify the good curing of the resin, like a Barcol test for example.

Furthermore, as scatter can be important for the same design, one proposition is to **make a statistical assessment of the key performance property(ies) of the composite, respectively cylinder**.

The last topic covered by HyCOMP was in-service inspection of composite pressure vessels. Acoustic Emission was used to follow damage accumulation for almost all tests on specimens and cylinders. Unfortunately, we faced scientific and experimental difficulties to use AE as an inspection method for the evaluation of cylinder damage. **AE seems promising** because well adapted to composite materials (possibility to discriminate types of damage and to quantify each of them), **but further research is needed** to propose a universal methodology with well defined pass/fail criteria, complying with industrial constraints. Furthermore, the development of NDT techniques for in-service inspection must necessarily be coupled with a study on mechanical impact. Impact severity will determine AE parameters threshold to remove a cylinder from service.

6. Technical annexes

6.1 Annex 1: Master Curve and Damage Thresholds

The failure of a structure occurs when the damage that it has accumulated reaches a critical threshold above which it can no longer support the applied stresses. In the case of a carbon fibre filament wound pressure vessel the loads are almost completely supported by the fibres and they must break in a burst test for complete failure to occur. Burst tests allow the damage threshold level in a simple burst test to be experimentally determined and quantified using the acoustic emission technique. Under steady or constant amplitude cyclic loads, lower than the burst strength, fibre breaks accumulate, after initial loading, due to relaxation of the matrix and subsequent additional overloading of fibres. The rate of fibre breaks under these conditions can be described analytically or in greater detail by the multi-scale model of damage accumulation which has been developed. In this way a master curve relating damage accumulation to time under load can be drawn and extrapolated to the point where it reaches a designated damage threshold level, as shown in Figure 7. As, in service, the pressure vessel will be subjected to loads less than the burst pressure the damage threshold level for complete failure is, as a consequence, raised, as illustrated in Figure 8. This and means that the burst threshold level can be used as a conservative critical damage level and the master curve used as a means of determining minimum lifetimes of the structures.

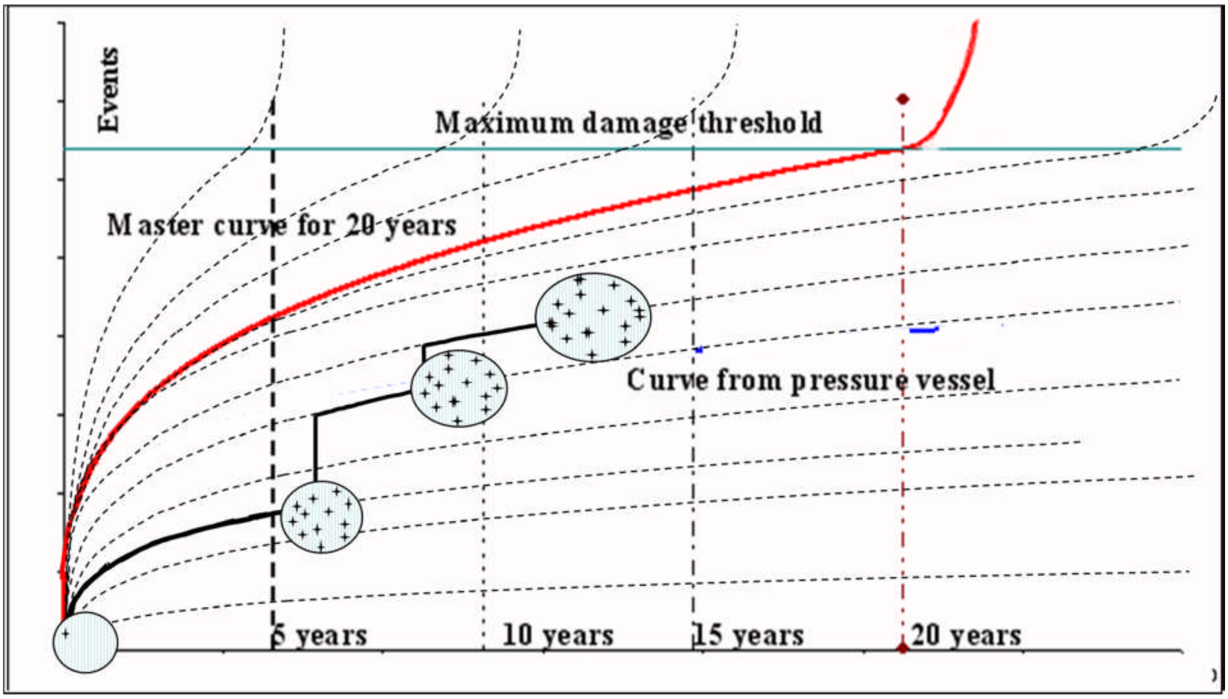

Figure 5 : Effects of overloading during the life of a pressure vessel

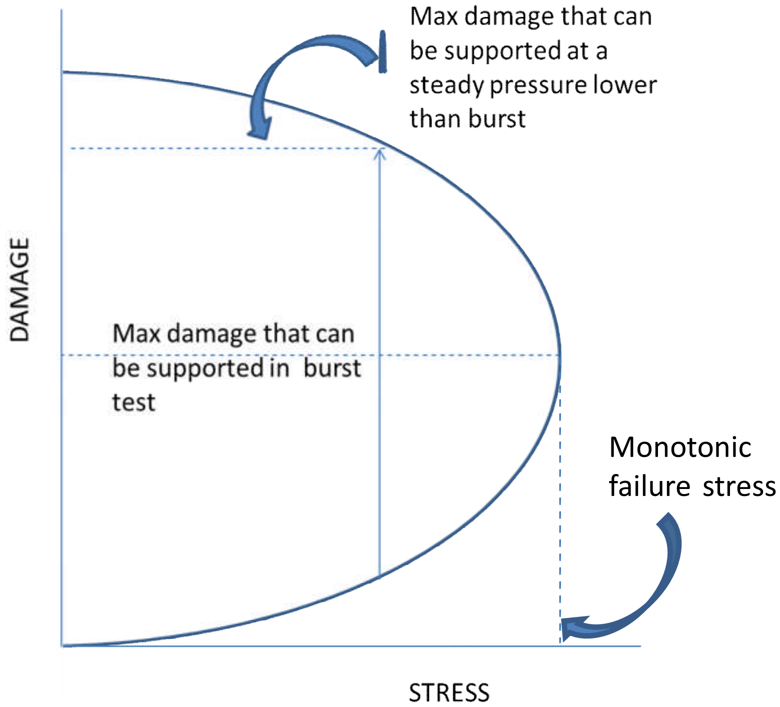

Figure 6: At stresses lower than the monotonic failure stress the composite can withstand higher levels of damage.

There are some practical difficulties in experimentally determining the threshold levels as the mechanism which dominated failure is the breakage of the fibres. Acoustic emission is the obvious technique for monitoring fibre failure but requires interpretation as other failure processes, such as matrix cracking and interfacial debonding, also produce emissions. This can be exacerbated, during periodic in-service testing to identify where a pressure vessel is placed on the master curve, as the length of time necessary to determine the failure rate under a load equal to the maximum in-service load may be unduly long as the composite must first be allowed to relax after reaching the test pressure. Nevertheless the approach is of practical use as these processes only increase the apparent measured damage activity. A comparison with the damage threshold level at burst using the master curve technique can only overestimate of the state of damage in the pressure vessel which allows a conservative evaluation for future reliable use to be made.