HyCOMP
Grant Agreement n°256671

Project title: Enhanced Design Requirements and Testing Procedures for Composite Cylinders intended for the Safe Storage of Hydrogen

Funding Scheme: JTI - Collaborative project (FCH)

Date of latest version of Annex I against which assessment will be made: 17th December 2013

Deliverable Report
WP3
D3.4 Summary report for the WP3 with remarks and recommendations

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Deliverable ID:	D3.4
Deliverable Title:	Summary report for the WP3 with remarks and recommendations
Due date:	M33
Responsible partner:	BAM
Contributors:	ALL WP3 Partners

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Document Information

Document Name: HyCOMP_WP3_D3 4_BAM_240314_final.doc
Revision: V5 Final
Revision Date: 17/04/2014
Authors: BAM

Document History

Revision	Date	Modification	Author
V0	22/03/2013	Preliminary Version	C. Gregor (BAM)/ P. Gasior(WUT)
V1	21/10/2013	revised draft	G. Mair,C. Gregor, M. Hoffmann(BAM) /Gasior (WUT)
V2	01/11/2013	revised draft with respect to WP 7 meeting	G. Mair (BAM)
V3	09/12/2013	Further clarifications / editorial changes	C. Gregor(BAM)
V4	11/02/2014	Content/edditorial changes according to discussion during M36-meeting	G. Mair,C. Gregor(BAM)
V5	02/04/2014	Final Version	BAM
VFinal	17/04/2014	Coordinator and Quality check	AL/ALMA

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1. Executive summary

1.1 Summary of deliverable content and initial objectives

The aim of this Work Package was to develop a better understanding of effects of cyclical and hybrid loads on the cylinder structure in order to justify allowable service life. The results are used to determine test protocols and methodologies to ensure with a required level of assurance that the cylinder will not fail in specified service conditions.

Type 3 and type 4 cylinders have specific structural requirements and limitations. These include different failure modes specific to the type of liner used, as well as the influence of manufacturing and environmental conditions on the failure behaviour. The experimental testing in WP3 was designed to cope with these type specific requirements. The focus for type 3 cylinder testing was the influence of the manufacturing (autofrettage) process on the fatigue behaviour and different combinations of static and cyclic loads on the residual cycle strength. The type 4 cylinder testing focused on the influence of different cycle amplitudes and mean pressure and the qualification of the state of damage. For both types the influence of gaseous cycle tests compared to hydraulic cycle tests at elevated temperature, which is comparable to the maximum temperature developing during gaseous H2 cycles and at room temperature on residual strength was studied. Furthermore two NDT methods, Acoustic Emission (AE) and Optical Fibre (OF) strain measurements, were used to evaluate their usability to evaluate the state of damage of composite cylinders.

This report will first give a short summary of the work performed and results of the test campaign in Chapter 2. 1 and then will address questions raised in WP7 based on the work performed in Chapter 2.2. Further remarks out of the testing activities are given in Chapter 2.3.

1.2 Partners involved

BAM, AR, AL, CEA, CAQ, Faber, HEX, WURT, JRC-IE

1.3 Relation with other WPs / Tasks

WP2, WP4, WP6/WP7

2. Main body

2.1 Summary of work done

2.1.1 Literature Review and state of the art on fatigue failure of cylinders T3.1

A review providing an overview of critical factors leading to failure of composite cylinders showed a variety of influencing factors. Furthermore processes leading to different failure modes in type 3 and type 4 cylinders were examined. This includes the influence of the manufacturing process and environmental conditions on the failure behaviour of the common structural shell, and failure modes specific to the type and style of liner used. Additionally, this review gives an overview and discusses aspects of relevant Regulation, Codes and Standards (RCS).
The literature review served as a starting point for the HyCOMP – project and main issues addressed there were explored during the project and are discussed in this paper.

2.1.2 Type 3 specific

The focus for type 3 cylinder testing was the influence of the manufacturing (autofrettage) process on the fatigue behaviour and different combinations of static and cyclic loads on the residual cycle strength. For type 3 cylinders it is not possible to separately evaluate the mechanical behaviour of the Fibre-Reinforced-Plastic wrapping and the metallic inner layer (metallic liner). The two layers interact with each other from the beginning of the manufacturing process [1-4].

The autofrettage procedure is the final manufacturing stage and the main process, where the state of residual stress between the metal liner and composite is generated. This state of residual stress is important to increase the fatigue resistance of this hybrid structure [1-6]. Therefore this process was one important topic of the test activities performed during HyCOMP program. The state of stress and ultimately the residual stress out of the autofrettage process is directly linked to the deformation of the pressure vessel during the autofrettage process. Therefore the volume expansion of the vessels during the autofrettage process was measured for the main part of the type 3 specimens and linked to the cycle fatigue behaviour. The test campaign indicates a clear influence of the state of deformation during the autofrettage process on the cycle strength. There seems to be an optimum deformation during the autofrettage process for achieving optimum cycle strength figure 1.

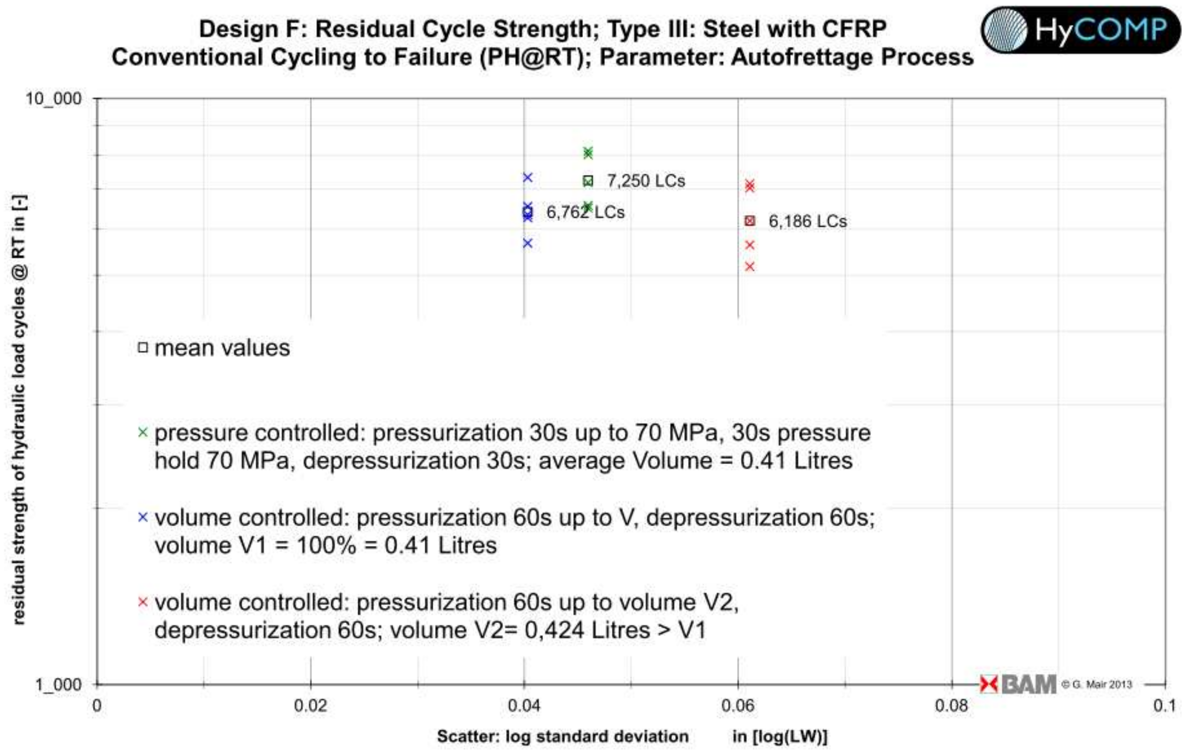

Figure 1: Influence of autofrettage control parameter on sample behaviour indicated by median of LogND and scatter.

The FRP wrapping is a viscous material which naturally shows time and temperature dependent behaviour [10-12]. Both parameters are linked together by a time-temperature superposition principle. One part of the test campaign followed the thesis that the time dependent behaviour can influence the test results out of burst tests. Therefore the pressurization rate is an important process parameter of a burst test. The viscous material behaviour will more and more dominate the processes at slow pressurization rate. This possibly leads to lower burst strength of pressure cylinders with material damages out of service loads and makes it easier to distinguish between damaged and undamaged pressure vessels by burst tests as shown in figures 2 and 3. It was noticed that the pressurization rate has an influence on the strength values and on the scatter of strength. This influence of loading rate on the ultimate strength of composites has been studied and explained by modelling by a multi-scale model approach in WP2 and [19].
For type 4 cylinders most often the ultimate strength and the scatter was decreasing with decreasing pressurization rate. For type 3 cylinders this behaviour could not be clearly observed. The burst strength was slightly increased from cylinders tested at lower pressurization rate and the scatter was not significantly changed. The influence of the metal layer and the state of residual stress of the type 3 model cylinder is most likely the reason for this behaviour.

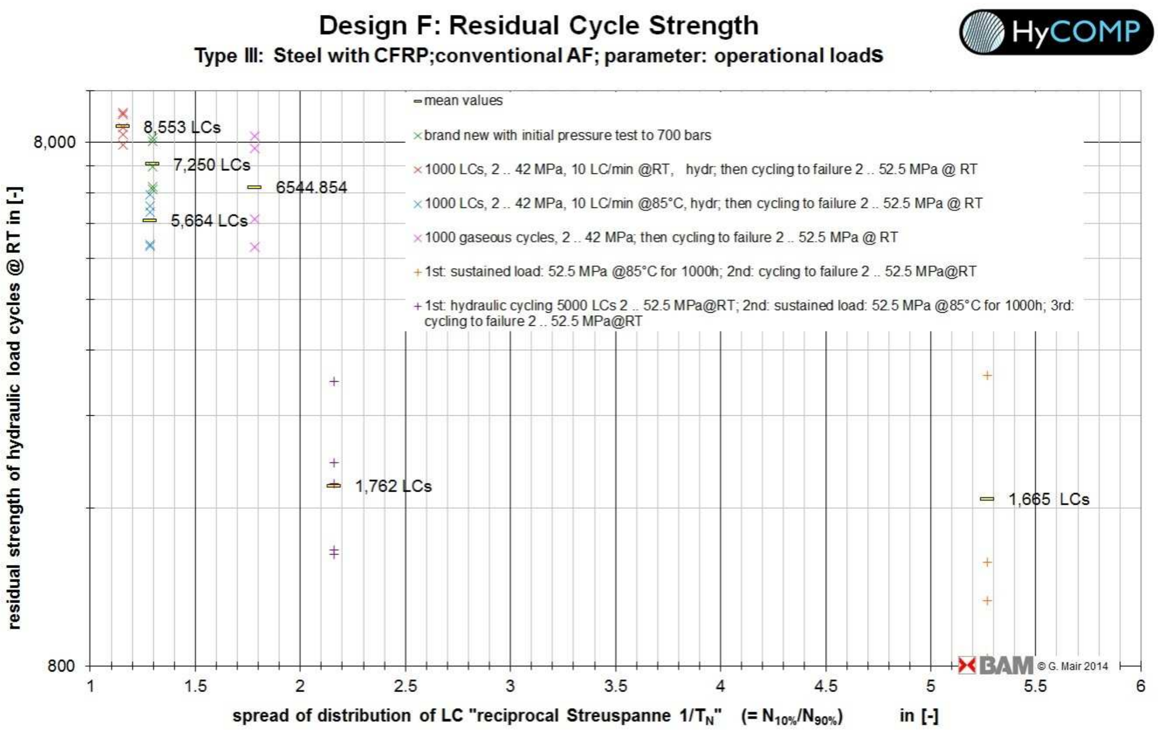

Figure 2: Influence of artificial aging on sample behaviour indicated by residual load cycles (median of LND and scatter).

The influence of hybrid loads (combination of static and cyclic loads) on the cylinders structure and the residual cycle strength is an important aspect in order to determine the reliability of cylinders which have endured service loads. A static load in combination with elevated temperatures, using the time-temperature superposition principle for viscous materials or cyclic loads are often used for testing procedures [12] in order to provoke damage in the FRP wrapping of pressure cylinder. This part of the test campaign in WP3 addressed the influence of a combination of static and cycling loads and the influence of the order of these loads.
The results showed an influence whether a static load was applied at the beginning of a cycle procedure or at the end. The mean residual cycle strength was not varying but the scatter of the results was significantly different see figure 3. This shows the importance of a probabilistic treatment of a material with high scatter of strength and with a change of scatter of residual strength after applying service loads.

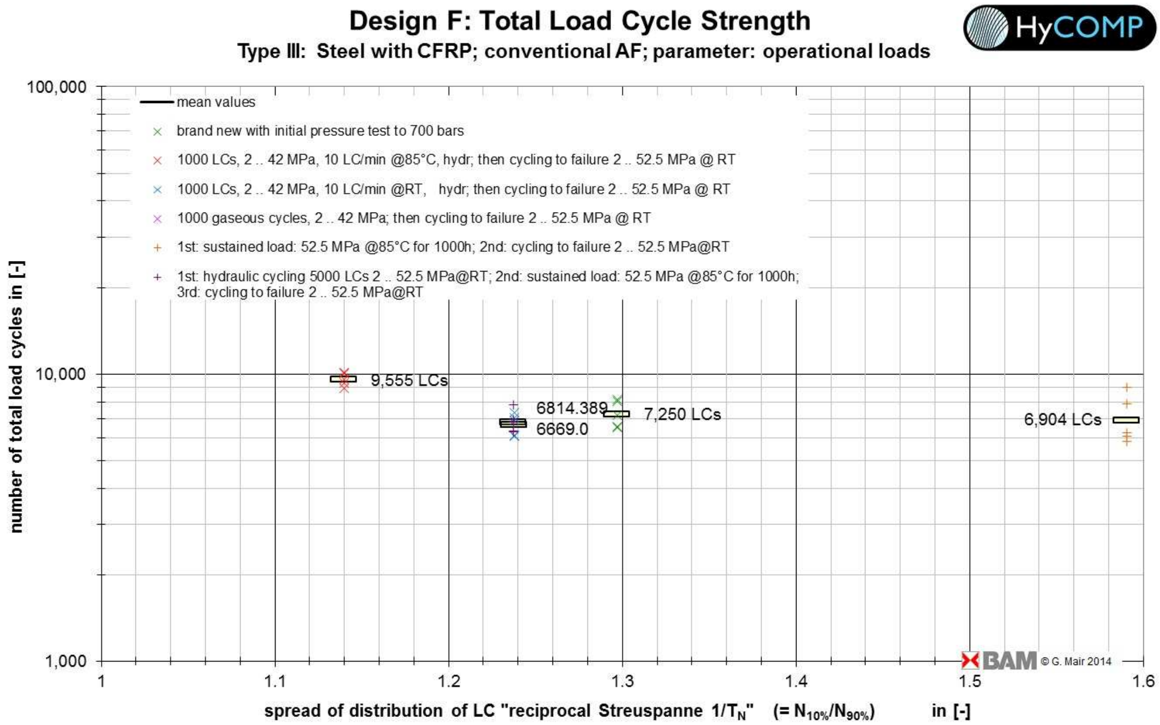

Figure 3: Influence of artificial aging on sample behaviour indicated by total load cycles (median of LND and scatter).

The service life and therefore the reliability of pressure cylinders depends on the spread of the residual stress out of the autofrettage process and are influenced by the manufacturing process and the material parameters, e. g. the spread of the carbon FRP properties, and the in service conditions [7-9]. Since there is no feasible method to measure mechanical stresses in a heterogeneous structure of fiber composite pressure vessels the state of deformation is taken to assess the state of stress. To survey the deformation of a pressure vessel optical fibers (OF) promise to show good results in matters of strain resolution, global surveillance of the cylinder, incorporation and measurement execution.
The measurements on 6 type 3 cylinders with OF showed good results, the state of deformation in different layers was measured, the position of cracks leading to final failure of the cylinder were found by evaluating changes of strain during cycle testing to failure. The tests included the autofrettage process, temperature loads without pressurization and cycle tests. The results show a distinct influence of high temperature loads at ambient pressure on the fatigue resistance of type 3 pressure vessels.

2.1.3 Type 4 specific

For failure modes analysis and conditions of type 4 cylinders under different kind of loads, 700 bar cylinders with a volume of 36 liters were used. 16 cylinders in four groups were pressure cycled at ambient temperature conditions for 20k cycles in four pressure range cases, see table 1. The idea was to check if the deep of pressure level during cycling may have an influence on thick wall cylinders. Each of these cycling tests should create a variety of damage states. The cylinders were subjected to a constant internal pressure for ~2 weeks. The pressure value was set as 80% of the mean burst pressure of these cylinders.
During the test Acoustic Emission of the cylinder composite wrapping was registered. It was planned that the results of this test would allow the determination and evaluation of a methodology to characterize and quantify the state of damage of a composite pressure vessel (according to a methodology of “a master curve of AE events” carried out in WP2).

The results after the first cycle and sustained load tests were not conclusive. It was assumed that the damage out of this load scenario was not enough to produce measureable results, therefore the tests were extended by another 30k load cycles and sustained load procedure, see figure 4.

test case	cycle test amplitude [MPa]	sustained load test
I	2 ÷ 87.5	80% of the mean burst strength (equal 145.4MPa)
II	87.5 ÷ 105.0	
III	70.0 ÷ 87.5	
IV	35.0 ÷ 87.5	

Table 1: Parameters of test cases performed on type 4 cylinder.

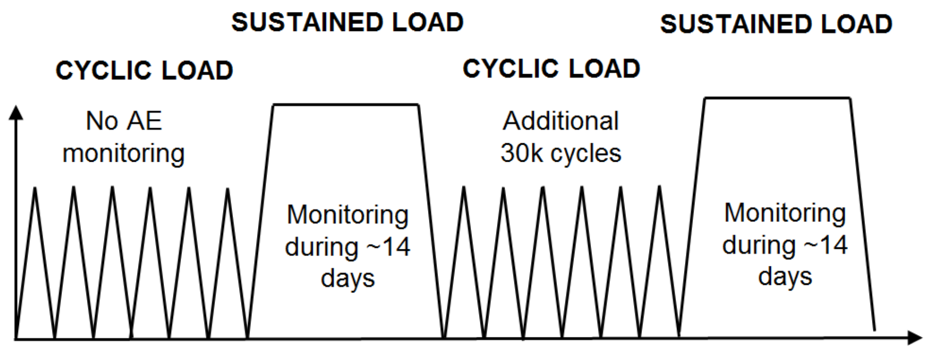

Figure 4: Test procedure for AE – measurement for failure mode analysis

Analysis of AE measurement during the first period of sustained load – AE hits amplitude in time domain

Analysis of graphs obtained from cylinders of test case IV shows that hits with amplitude higher than 45 dB occur longer after starting of the test comparing to other cases. It means that these cylinders were longer AE active than other ones, numerous hits which had higher amplitude than 45 dB occurred up to 250 h from the moment of starting AE measurements or even longer. In case of test case III cylinders the number of registered hits with higher amplitude than 45 dB decreases after 150 hours from the beginning of AE measurements.
In case of cylinders from test case I and II, the number of hits with amplitude higher than 45 dB significantly decreases after 80 h.

For few of tested cylinders during sustained load test, it was possible to observe sudden increase of amplitude, energy and number of acoustic events. It is probably caused by arising matrix cracks or development of existing defects in the AE sensors area. The hits which have small energy might be connected with cracking carbon fiber while high energy events suggest arise and/or propagation of cracks in matrix.

AE hits amplitude in time domain after additional 30k cycles

After additional 30k pressure cycles according to test case for two cylinders, the amplitude of hits just after pressurization is little smaller in comparison to AE in first part of examinations (after 20k cycles). No other significant differences can be seen on amplitude versus time graphs. In case of two cylinders of test case III, amplitude of registered hits was decreasing slower in second step of examinations. For both tanks after 100h, the AE is significantly higher after additional cycling.

Two pressure cylinders burst during the second sustained load test. One cylinder of test case I burst after one hour and one cylinder of test case IV burst after 80 hours. Both test shows that based on amplitude versus time graph, it is difficult to predict rupture.

Number of AE hits in AE amplitude domain

Generally, cylinders pressure cycled with a higher average range of pressure are characterized by a higher number of AE hits, also with larger amplitude. It shows that higher amplitude of pressure cycling is more critical for composite construction than cycling with lower amplitude but on the higher average pressure level.
However detailed analysis of the AE data registered during the sustained load test shows, that in some cases a difference in the number of recorded AE hits within the cylinder group with the same load history was observed. In case of energy graphs, there is no noticeable relationship between the energy of hits and the cyclic loading conditions.

Number of cumulative AE hits in time domain (master curves analysis)

To study AE signals registered during sustained load test the total number of AE hits in time domain was analysed. It is possible to observe that cumulative number of hits does not determine state of damage of composite cylinder. It is difficult to observe clear relationship between history of loading (amplitude of pressure cycles) and total number of AE hits.

2.1.4 Work performed on type 3 and type 4 model cylinders

The process of filling a cylinder with compressed gas and the impact of this process on the reliability is an important aspect. In the current standards it is assumed that gaseous cycles have the same impact as hydraulic cycles. This assumptions seems questionable because the different characteristics (temperature, pressurization rate) of the processes. A compression and decompression during a gaseous cycle test involves a heating or cooling of the gas and ultimately of the cylinder. During the hydraulic cycle process the temperature is approximately constant. The pressurization rate of a gaseous cycle is slower and the timespan at maximum pressure is considerably longer compared to a hydraulic cycle. The effect of hydrogen (H2) cycle process compared to the common hydraulic cycle process was evaluated by a test campaign with 30 cylinders, 15 type 3 and 15 type 4 cylinders. 3 Groups with 5 cylinders each were cycled with different parameters for 1000 load cycles, one group was H2 gaseous cycled, two groups were hydraulic cycled at room temperature and at elevated temperature of 85℃.

The hydraulic load cycle (LC) tests on the type 3 model cylinder were performed with 10 LCs/min with a pressure range of 2MPa – 1.5 times nominal working pressure (NWP) in a temperature controlled environment. The gaseous LC tests were performed with a pressurization rate of less than 1.5 LCs/h and the same pressure range of 2MPa - 1.5 NWP in a not temperature controlled environment. A similar procedure was applied on type 4 cylinder tests with one exception; the pressure range was 2MPa – ~1.2 NWP.

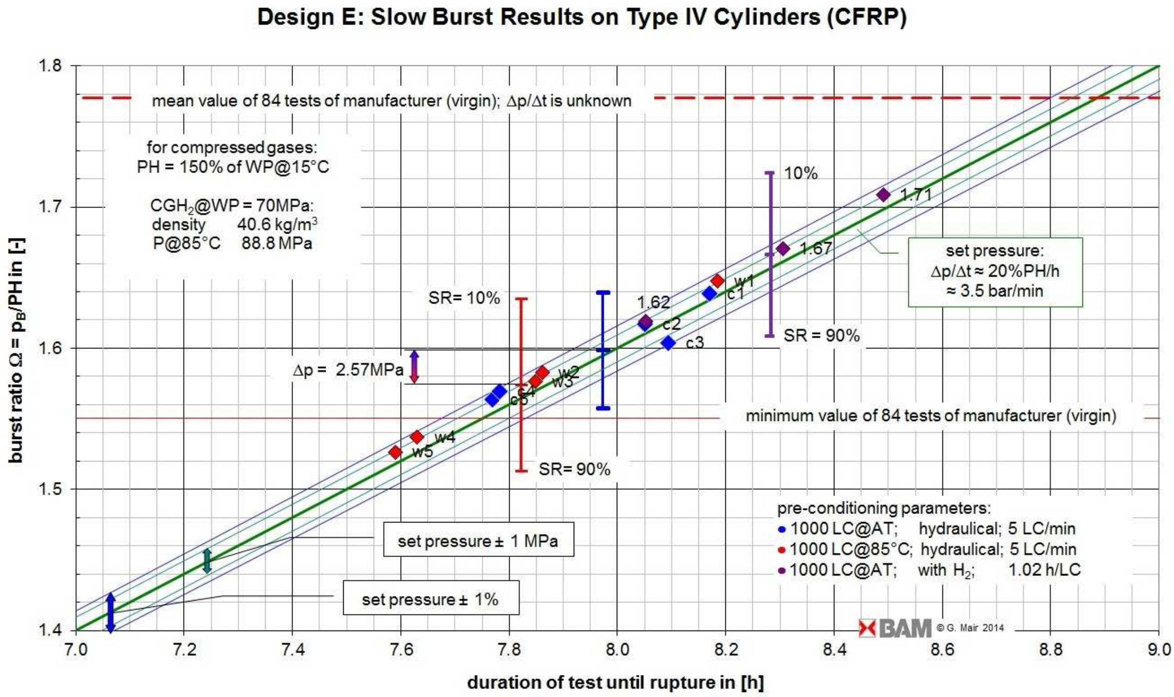

Figure 5: Influence of artificial aging on slow burst test results related to test pressure PH (=150% NWP).

Unfortunately not all pressure cylinders could be successfully cycled in the frame of the project, 1 out of 5 type 4 cylinders was damaged during the complex installation procedure of temperature sensors inside the cylinders prior to testing and on 1 out of 5 type 3 cylinders the gaseous cycle process is not finished yet. Therefore the statistical evaluation was not fully possible but a trend was derived as shown in figures 5 and 6.

The results in figure 6 indicate that the mean residual strength of the groups of cylinders which were gaseous cycled and hydraulic cycled at elevated temperature were approximately the same while the scatter, assuming a normal distribution of the strength results, was different. The scatter of the gaseous hydrogen cycled cylinders is 40% higher compared to the at room temperature hydraulic cycled cylinders. The scatter of the at 85℃ hydraulic cycled cylinders is 50% higher than the scatter of at room temperature hydraulic cycled cylinders.

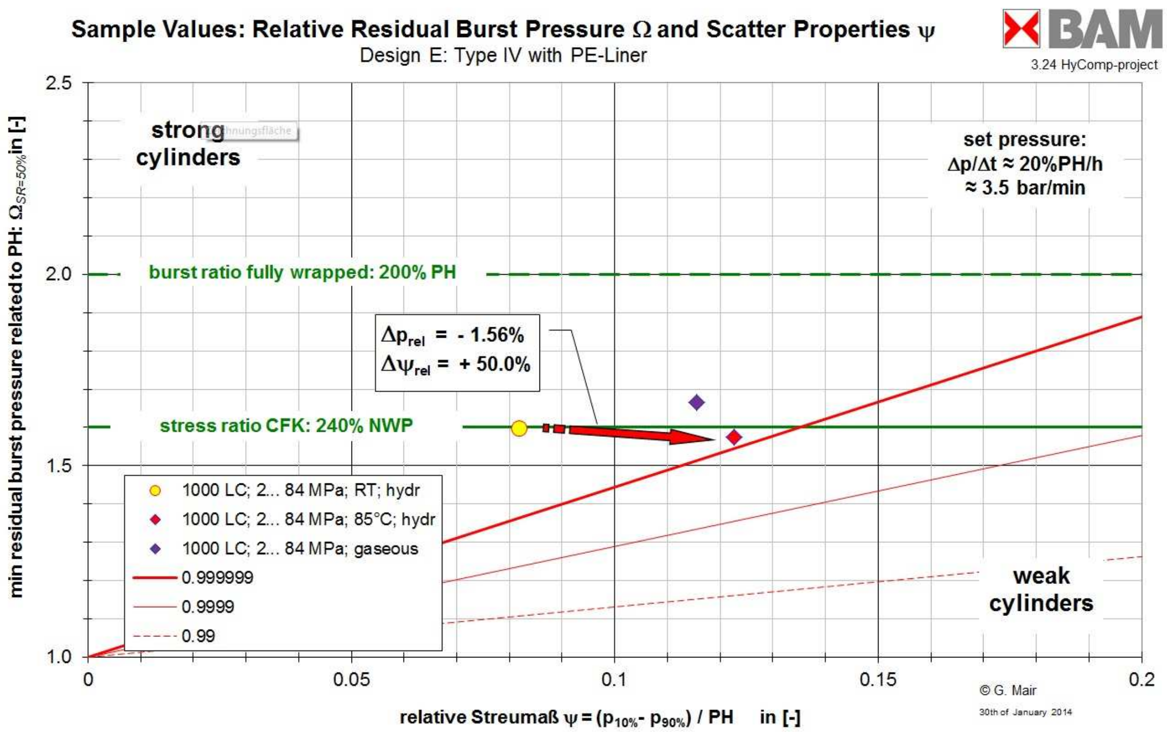

Figure 6: Influence of 1000 gaseous cycles as artificial aging on sample behaviour indicated by total load cycles (median of ND and scatter).

2.2 Questions of WP7 / Results

How can changes in the wrapping cause premature liner failure?

Regarding type 3 cylinders the project results indicate that parameters which influence the artificial residual stress state of the hybrid structure out of the manufacturing process can reduce the fatigue strength and lead to premature liner failure. These are parameters of the manufacturing process, e.g. deformation during the autofrettage process, and of service life, e.g. high ambient temperature. It has to be stated that the behaviour of the FRP wrapping and the metallic liner are linked together from the start of the manufacturing. Therefore an examination just focusing on the FRP wrapping is not sufficient.

A modification of autofrettage control parameters (pressure, rate, max. pressure holding time, volume expansion, temperature etc.) influence the internal state of stress (load sharing) and the resulting mean value and scatter of residual strength. Furthermore, a constant high temperature results in an expansion of the metal liner, thus part of load sharing of composite is temporally higher than at RT! This can change the residual state of stress of the cylinder structure and can in case of low temperature directly or in case of high temperature via compression yielding lead to a premature liner failure.

Do gaseous loads have the same effect on composite as hydraulic loads on the composite structure?

Out of the viscous material behaviour and this depends on the parameters of the test. The mechanical behaviour including the damage processes of viscous materials, like the carbon fibre composite with a thermoset epoxy matrix used in the project, depends strongly on parameters with a time- or temperature influence e.g., loading rate, time under load, ambient temperature. This rate of time dependent viscous effects will increase with decreasing difference between specimen temperature and glass transition temperature. Currently a gaseous cycle process is significantly slower compared to a hydraulic cycle process and the temperature development is different. Therefore out of this consideration a different material behaviour is possible. This question focuses on the issue, if the influence of the difference between the two processes is significant enough to be seen in the material behaviour.

Test results of type 3 cylinders indicate on one hand a similar mean value of residual cycle strength of gaseous and hydraulically cycled cylinders at 85℃ and a higher scatter of the gaseous cycled cylinders. On the other hand the difference between the hydraulic cycle tests at ambient temperature and a gaseous cycle tests is significant (around 30%).
The test results of four gaseous cycled type 4 cylinders showed a higher residual burst strength compared to cylinders cycled at room or at elevated temperature with a significant higher scatter of 40% compared to room temperature hydraulic cycled cylinders and a 7% lower scatter compared to high temperature hydraulic cycled cylinders.

What is the impact of pressure cycle amplitude on damage rate from pressure cycling in cylinder?

There are no conclusive results so far. The test campaign was performed on type 4 pressure cylinders, with different pressure cycles, in one test case up to 1.5 times the working pressure. The tests showed that for the selected experimental conditions (20k pressure cycles with different amplitudes and sustained load test at 80% of average burst pressure) cylinders are working uniquely in respect of the acoustic emission procedure applied. It causes difficulties in analysis of the results and drawing general conclusions.
The AE methodology was not sufficient to characterize an impact of pressure cycle amplitude.

How can damage accumulation be measured in a cylinder?

There are several concepts to measure damage accumulation in composite materials e.g., change of stiffness, change of results out of acoustic emission testing, change of ultrasonic attenuation, x-ray refraction, change of electric resistance and other. The two first methods were evaluated in the project.
For type 3 cylinders the change of stiffness was indicated and indirectly measured during load cycle tests, by a change of strain measured by optical fibres. The global strain over LCs appears to be promising for a life time assessment of type 3 cylinders. Nevertheless, further research is needed to understand all aspects of different influences e.g., different behavior of OF and cylinder wrapping under temperature influence, coating of the fibers, pressure load radial to the fiber direction, length of OF on local resolution etc., on this technique.

Acoustic emission testing was used to detect damage progress in composite specimens in WP2 in D2.4 it is stated: In sustained tensile loading, a steady increase in volumetric density of fibre breaks was recorded using AE. The damage is associated to time-dependent viscoelasticity property of epoxy matrix allowing the stress on the epoxy matrix to relax with time. However, AE monitoring on composite cylinders was only capable of local evaluation due to high signal attenuation of cylinder structures.

2.3 Further Remarks

NDT - None Destructive Testing

In the frame of the project 2 different NDT methods were evaluated. These two methods were an optical fibre strain measurement technique, where the OF was installed inside the composite wrapping during the manufacturing process, and an AE methodology. There are other methods to measure damage accumulation in composite materials e.g., contact and contact-free strain measurement, electrical resistance, x-ray refraction, ultrasonic attenuation and other. There is currently no system which is applicable without further adaptation on the specifics of composite pressure cylinder. It is likely that a mix of more than one measurement methodology has to be applied to compensate limitations of other methodologies.

The optical fibre is integrated into the composite wrapping in the manufacturing process. The incorporation of OF in the HyCOMP type 3 cylinder was successfully accomplished at all 6 cylinders. Only one out of twelve fiber layers was not readable due to transport damages. The crack causing the failure of the cylinders was clearly detectable. It appears favorable to implement the OF near the area of possible damages. 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.

The Acoustic Emission testing showed to be partly capable to help to understand damage processes in composite specimens and pressure cylinders. A main limitation is the limited range due to the acoustic attenuation of the carbon fiber reinforced plastic. More research is needed to overcome this limitation in order to have an efficient tool usable at an industrial level.

Influence of the manufacturing process on cycle service life of type 3 cylinder

Altogether the influence of winding and tempering on the cyclic service life of the type 3 cylinder seems limited compared to the autofrettage process. But it is not negligible that poorly chosen process parameter may influence the optimal effect of the further manufacturing process and may lead to a reduction in service life. It is recommended that no condition of pretension in the metal liner occur after winding and tempering.
The autofrettage process appears to be the main influence on the cycle life of type 3 cylinders. Here large yielding during autofrettage reduces cycle life expectancy by deterioration of the metal properties. Influenced by winding and tempering it is possible, that pre stress or the typical distribution of mechanical properties like the stiffness of the composite, lead to circumstances that affect the cycle life. It is recommended to avoid large deformation at autofrettage that affects the efficiency of the process.

Influence of service conditions on service life of type 3 cylinder

The state of residual stress and therefore the cyclic service life depends on the temperature, because the thermal expansion coefficients of the metal and the composite are different. This may lead to a debonding of the liner at low temperature [1] for designs that use a composite to liner bond in order to avoid buckling at ambient cylinder pressure. At high temperatures, compressive yielding of the metal liner may occur; especially when the induced compressive stress in the liner is close to the compressive yield stress as suggested in [4]. In addition, classical external shell buckling of the liner can occur if the thermal and residual stresses are not managed. This would lead to a reduced lifetime of the pressure cylinder.

Tests performed indicate a higher spread of cycle life after a preconditioning of 1000h at 85℃. For real service life this indicates that poo r service conditions affect the residual stress distribution and lower cycle life in some cases.
Service conditions in terms of high temperature appear to degrade the minimum cycle life. Further it is probable that the spread of load cycle strength rises.

The test results can be summarised as:

- The order of cycle loads and sustained load has a very high influence in residual strength i.e. degradation!
- A sustained load influences the residual cycle strength – especially with respect to increase of scatter!
- High temperature during a sustained load leaded to higher burst pressure while load cycle strength has been reduced.
- That means: Burst test shows safety against (one-time) overload dominated by composite – but has no relevance for service strength of metal liner!

Recommendations

As shown in [13-21] some degradation effects linked with an increase of scatter are hardly detectable without a probabilistic assessment. Therefore the following recommendations could be improved further by on statistical assessment of strength values in combination with strength tests to failure. This is especially supported by [22].

As general remarks concerning improved RC&S deduced from this field of experience it can be stated:

a) For avoiding misinterpretation or later misuse of a standardised test procedure the purpose of each test should be described as detailed as possible!

b) Hints on the maximum pressure and temperature during filling and service as well as the addressed number of full filling cycles and addressed life time should be given for dominant application.

c) For dedicated service (single gas service) the reference pressure is the maximum developed pressure at the maximum possible / allowable temperature based on the allowable amount of gas (NWP = PW = pressure at 15°C or kg gas per m3).
For use of several gases the test pressure (PH = 150% PW or TP = 150% NWP) should be used as reference or design pressure.

d) The influence of small amplitudes with simultaneous high mean pressure should be taken into account for applications, where this type of cycles has a major part in the load spectrum.

e) Where ever possible "performance based" concepts should be given preference. This means proof of sufficient safety level and especially performance of artificial ageing by using test load conditions, which are as comparable as possible to operational load conditions.

f) Performance based concepts demonstrate expected degradation to End-of-Life (EoL) by mechanical simulation of real service loads (covering all expectable load conditions); not by calculating or evaluating the performance under unrealistic high loads.

g) The performance based part of simulating load cases in the meaning of artificial aging shall be followed by a method for quantification the residual strength by a test to failure subsequent to each artificial aging.

h) Deviating from e) the test load condition may differ from the service load condition, if the test is exclusively for quantification of the residual strength. This can help to save effort – but shall not be used if the relationship of test condition to service condition or interpretation of test results is not clear.

i) The test procedure for residual strength testing should be chosen with respect to the first failure during load cycling.

j) If the test duration may have an influence on the test results, the load rate shall be limited for pressurization and depressurization.

k) If the test duration may have an influence on the test results and the test is performed for the purpose of comparison with other results, the test parameters, e.g. load rate, should be controlled and documented in detail.

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