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Deliverable Report

D7.1
Preliminary WP7 Report

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1. Executive summary

1.1 Summary of deliverable content and initial objectives

The objective of this Preliminary WP7 Report is to summarize the findings and recommendations concerning the safe storage of compressed hydrogen in composite cylinders and to disseminate the project results for enhanced design requirements and testing procedures so that they can be used by the international hydrogen and fuel cell community.

At this time, only draft reports from WP2, WP3 and WP4 have been received. The D2.4 and D3.4 reports have been reviewed and a brief summary of their findings has been included in section 2.2 below.

D7.4 is the preparation of the Final Project Report which will build consensus with the other WP leaders and will include final WP2 to WP6 reports as Annexes.

1.2 Partners involved

CCS, AL, AR, BAM, Faber, Hex

1.3 Relation with other WPs / Tasks

The output of the work of all completed WPs will be fed into this report.

2. Main body

2.1 Description of work done (including difficulties encountered and solutions proposed if relevant)

2.1.1 Task 7.1 has been completed. It has prepared a list of active and published RCS and along with WP6/Task 6.1, it has identified RCS issues and the specific information that this project can deliver to address these issues.

2.1.2 The difficulties in completing this D7.1 preliminary WP7 report is due to the delay in the test program and indeed in the lack of actual RCS findings and/or improvements from the WP2 to WP6 to-date.

2.2 Discussion and conclusion

Following is a brief summary of findings with respect to enhanced design requirements and testing procedures.

2.2.1 WP2 - D2.4 Report Summary of findings

WP2 is focused on the material scale and all results reported below must be considered for composite material purposes although some references are given for composite pressure vessels.

The WP2 concludes that large-scale fibre rupture clusters are the most critical damage to the structural integrity of composite materials dedicated to CPV cylinders. It was determined that the strength of the laminates was dependent on the total content of fibre rupture clusters developing in the material. The long-term lifetime behaviour of the composites is highly dependent on the type of the epoxy resin used and the curing condition applied in the composites, which affect visco-elasticity of the matrix. There are other factors that can affect the growth rate of damage clusters such as, applied stress level and environmental conditions.

Findings for manufacturing composite pressure vessel cylinders

Use epoxy matrix polymer with better resistance to stress relaxation (creep) process should effectively reduce the growth rate of fibre rupture clusters so as to retard material degradation process in the structure and improve the lifetime performance

Use epoxy matrix with the material properties, having lower dependency on the elevation of temperature, particularly when close to T_g. The effect of temperature must be taken into account in the safety factor analysis since the rise of the temperature can affect the lifetime of the composites.

Optimize the thickness of laminate ply of CPV cylinders for the consideration of fibre rupture clustering process. It was found that fibre rupture clustering process was more favourable in the laminate containing one thick 0° ply than multiple thin 0° plies.

Findings for acoustic emission evaluation

There is no standardization for AE systems from different manufactures. In general, the parameters (such as energy) used for setting-up AE acquisition are not identical between different systems.

For non-destructive evaluation of composite pressure vessels, to perform full-field monitoring on CPV cylinders, numbers of AE probes (or sensors) distributed over the cylinder surface have to be applied to facilitate quasi full-field monitoring on composite cylinders.

2.2.2 WP3 - D3.4 Report Summary of findings

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 and style of liner used, as well as the influence of manufacturing and environmental conditions on the failure behaviour.

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.

How can changes in the wrapping cause premature liner failure?

Regarding type 3 cylinders the project results indicate that parameters which influence the residual stress state of the hybrid structure can reduce the fatigue strength and lead to premature liner failure. The behaviour of the FRP wrapping and the metallic liner seem to be linked together from the start of the manufacturing.

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

The mechanical behaviour including the damage processes of viscous materials, like the carbon fibre composite with a thermo set 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.

The test results of type 3 cylinders indicate similar residual cycle strength of cylinders tested between gaseous and hydraulically cycled cylinders and a higher scatter of gaseous cycled cylinders. The test results of gaseous cycled type 4 cylinders showed a higher residual burst strength compared to cylinders cycled at room or at elevated temperature.

How can damage accumulation be measured in a cylinder?

There are several ways to measure damage accumulation in composite materials e.g., change of stiffness, change of results out of acoustic emission testing and other. The two first methods were evaluated in the project.
For type 3 cylinders the change of stiffness was measured during load cycle (LC) tests, by a change of strain measured by optical fibres. It was concluded that the global strain over LCs appears to be promising for a life time assessment of type 3 cylinders. Nevertheless, further research is needed to continue the development of this technique.

General remarks concerning improved RCS development

a) Where ever possible performance based concepts should be used.

b) Specify the maximum pressure and temperature during filling and service as well as the number of full filling cycles and life time for each application.

c) For dedicated service (single gas service), the reference pressure (or design pressure) should be the maximum developed pressure at the maximum possible / allowable temperature based on the allowable amount of gas (NWP = PW = pressure at 15℃ 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 stationary applications, where this type of cycles has a major part in the load spectrum.

2.2.3 WP4 - D4.4 Report Summary of findings

The Work Package 4 “Manufacturing quality assurance” has been split in three tasks.
The first task aimed to identify the manufacturing parameters that could influence cylinder performance. The parameters that have been identified as the most affecting the cylinder performance are:

- MF1: Winding geometry,
- MF2: Improper Resin mix
- MF3: Improper curing
- MF4: Carbon fibre mechanical characteristics.

Next, an experimental test plan has been defined and carried out in the second task, to evaluate the influence of the above parameters on the performance of the cylinder. The test plan has been developed with the aim to evaluate the cylinder performance in the short term and in the long term.

The third task of the work package aimed to evaluate the NDE (non-destructive examination) methods for production monitoring. Acoustic emission (AE) test has been performed during the first pressurization of the cylinders, searching for an NDE capable to evaluate the long term cylinders performance with a single test at the beginning of the cylinder life (i.e. at the manufacturing stage).

Finally, an evaluation of the results has been done to understand if the requirements of current standards and regulations for materials, production test, batch test and mandatory process control are adequate, excessive or inadequate to guarantee the product performance.

Test results

Manufacturing Variation MF1
A variation of winding parameters, simulated with an offset modification of one bandwidth, shows a negligible effect in the case of Type 3 cylinder and a significant effect on type 4 cyl-

inders. In the case of type 3 cylinders, the absence of significant effects can be explained because of the relevant contribute of steel liner on the shoulder and base ends. In case of Type 4 cylinder the effect is more relevant in terms of a much higher scattering of burst test results.
Present regulations, norms and standards generally prescribe that the winding parameters must be defined and for this reason can be considered adequate.

Manufacturing Variation MF2
A variation of resin mixture composition, simulated by an increase of 20% of hardener, shows no significant change in cylinder performance in either Type 3 or Type 4cylinders.
Present regulations, norms and standards that prescribe the process control of the resin mixture can be considered adequate.

Manufacturing Variation MF3
A variation of composite curing, simulated by leaving the winded cylinder in curing at room temperature, shows an important effect in the case of Type 3 cylinder and of type 4 cylinders. In the case of type 3 cylinders, it affects mainly the scattering of burst test but also the cycling test performance has been affected with an increment of scattering in the test results. Type 4 cylinders have been affected mainly in terms of scattering of burst test after preconditioning. This suggests the need of introducing a control of this characteristic by additional tests (such as a **Barcol test** on each cylinder) to verify the proper curing of the resin mix.

Present regulations, norms and standards prescribe tests in production, during batch certification and also address curing parameters to be defined and controlled as process control.
Due to the importance of the effect additional tests to check the curing can be recommended.

Manufacturing Variation MF4
A variation of fibre mechanical characteristics, simulated by replacement of T700 fibre with T300 having lower mechanical characteristics, as expected shows a very important effect in both cases of Type 3 cylinder and type 4 cylinders. In the case of type 3 cylinders, it affects mainly the burst test value but also the cyclic test performance has been influenced. Type 4 cylinders have been affected in terms of burst test result in the case of new cylinders and also in case of preconditioned cylinders.

The burst test prescribed by present regulations, norms and standards would detect such variation, being the resistance of the cylinder proportional to the mechanical characteristics of the fibres.

Acoustic Emission (AE)
In HyComp the capability of AE method was verified on type 3 and type 4 cylinders with artificially introduced defects described above. The influence of the four manufacturing deviations for both cylinder types was detected.

2.2.4 Closing

As explained in the report, at this time, only draft reports from WP2, WP3 and WP4 have been received and a brief summary of their findings has been included in this section.

The WP5 - **D5.3** and WP6 - **D6.4** reports are not available at this time but will be completed shortly.

A brief summary of their findings will be included in D7.4 which is the Final Project Report of the HyComp project. The Final Project Report will include brief summaries of findings of all WPs and their reports will be attached as Annexes.

2.2.5 Acronyms

AE	acoustic energy
CPV	composite pressure vessel
FCH	fuel cell and hydrogen
FRP	fibre-reinforced plastic
JTI	Joint Technology Initiative
NDE	non-destructive examination
NDT	non-destructive testing
NWP	normal working pressure
RCS	regulations, codes and standards