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Deliverable Report

WP7
Summary report on recommendations to support Regulations Codes and Standards initiatives

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1. Executive summary

1.1 Summary of deliverable content and initial objectives

The objective of this deliverable is to summarize and prioritize the findings of WP2 to WP6 to identify regulations, codes and standards (RCS) recommendations concerning the safe storage of compressed hydrogen in composite cylinders and to propose a roadmap for bringing these recommendations to international bodies. The report represents the consensus of the HyComp group regarding RCS recommendations.

1.2 Partners involved

All partners were involved in this Work package.

1.3 Relation with other WPs / Tasks

The results of the work of WP2-6 of the project were taken into account in this report.
WP7 worked in conjunction with WP6 which looked at the results of WP2-5 to come up with proposals for improvements in design requirements and testing procedures. Finally, WP7 summarized the results of WP2-6 to identify RCS recommendations.

2. Introduction

One of the main challenges for the extensive use of hydrogen is related to storage. Currently, the most mature technology for storing hydrogen is in compressed form in high pressure cylinders. Use of lightweight composite cylinders, in particular using carbon fibre material seems to be the preferred way to store compressed hydrogen in different applications.

According to Deliverable 6.1, there are a number of gaps that need to be addressed in current RCS for hydrogen storage in high-pressure type 3 and type 4 composite cylinders. This report addresses some of these issues based on results from the HyCOMP project.

3. List of regulations codes and standards (RCS)

Here is a short list of the key standards related to the HyCOMP project:
Transportable:

ISO 11119-2:2012: Gas cylinders of composite construction – specification and test methods – Part 2:Fully wrapped fibre reinforced composite gas cylinders with load-sharing metal liners

ISO 11119-3:2013 Gas cylinders - Refillable composite gas cylinders and tubes - Design, construction and testing. Part 3: Fully wrapped fibre reinforced composite gas cylinders and tubes up to 450 l with non-load-sharing metallic or non-metallic liners, Edition 2

EN 12245:2009+A1 Transportable gas cylinders – Fully wrapped composite cylinders. November, 2011

On-board:
GTR number 13 EC/TRANS/180/Add.13, Global technical regulation on hydrogen and fuel cell vehicles, United Nations. July 2013

EU 406/2010 implementing Regulation (EC) No 79/2009 of the European Parliament and of the Council on type-approval of hydrogen-powered motor vehicles.

ISO/AWI 19881 Gaseous Hydrogen -- Land Vehicle Fuel Tanks
Status: Active. WD anticipated from ISO TC 197/WG18.

Stationary:
ISO/CD 19884 Gaseous Hydrogen – Cylinders and tubes for stationary storage Status: Active. CD circulated for comments by ISO TC 197/WG15.
Note: For a full list of active and published RCS, refer to the Annexes in D6.1 report.

4. RCS Recommendations

Early in the project, Deliverable D6.1 was prepared to inform the WP2-4 test program of the gaps in current RCS. As part of Task 7.1, WP7 in conjunction with WP6, prepared and continued to update the RCS lists of active and published standards. Additionally, WP7 prepared a list of questions to be addressed by the WPs, which were answered in the final reports of WP2-6

This strong interaction of WP7 with all the other WP (WP2-6) facilitated the progress towards arriving at the project results, which consists of the D7.2 recommendations for design and testing dedicated to industry (and D6.4) and the present D7.3 report.

This D7.3 report covers the identification and prioritization of the RCS recommendations. In summary, there are <u>four</u> RCS recommendations (#1-4) intended for immediate consideration in RCS work and <u>one</u> item (#5) that is being proposed to be used optionally.

There is also a third category consisting of <u>three</u> recommendations (#6-8) being proposed for future consideration. They are being included for information only as they will need further work.

4.1 RCS Recommendations intended for immediate consideration in RCS work

4.1.1 RCS Recommendation #1

New specification: Recommendation to use the **Maximum Developed Pressure at T_{max}** (e.g. 65°C) as the Design Pressure for <u>transportable</u> applications and for dedicated gas service (e.g. Hydrogen) instead of using the test pressure. **T_{max}** is maximum ambient temperature.
See Technical Tables prepared by D5.3, Table 1- Overview of temperature requirements.

This is a general recommendation based on analysis done by the HyCOMP group. The rationale is that hydrogen shows a low expansion compared to some other gases at high temperature. For example, for hydrogen, the maximum developed pressure (Pmax) at 65°C is 1.18xNWP which is much lower than the test design pressure as currently defined (1.5xNWP). This change needs a stringent limitation of maximum temperature including during filling to this T_{max}.

Nominal Working Pressure is pressure at 15C.

This implies that the vessel be dedicated to hydrogen service and labeled accordingly (as specified in existing applicable standards)

(For more details see D6.4, recommendation #1)

4.1.2 RCS Recommendation #2

New specification: Recommendation to add a specification on **glass transition temperature (T_g)** to be higher than T_{max} + X°C as defined by the application.

The rationale for recommending this change is that WP2 has demonstrated at a material scale that temperature is an important parameter influencing damage accumulation (fibre breaks) in the composite wrapping. As operational conditions (maximum temperature in service) cannot be changed, 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 gets close to T_{max}.

(For more details see D6.4, recommendation #3)

4.1.3 RCS Recommendation #3

Change type test: Recommendation to perform tests **at elevated temperature**, at a temperature **not less than T_{max}** for cylinders used in transportable, on board or stationary applications.

The rationale for recommending this change is that WP2 has demonstrated that temperature has a strong influence on damage accumulation (fibre breaks) in the composite wrapping. Therefore the endurance tests at elevated temperature need to be performed at **T_{max}** to ensure conservativeness.

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}.

(For more details see D6.4, recommendation #5)

4.1.4 RCS Recommendation #4

Change type test: Recommendation to perform all relevant **tests at the maximum developed pressure at T_{max}** for dedicated gas service (e.g. Hydrogen) instead of using the test pressure for cylinders used in transportable applications.
Note: Also, see RCS Recommendation #1 for new specification:

The rationale for recommending this change is that cylinders will not be pressurized in service beyond the maximum developed pressure. Need to demonstrate that cylinder will withstand the most severe loads (not more).

Change test pressure to the maximum developed pressure, instead of P_h = 1.5 x NWP.
In ISO 11119-3, this list includes:
Ambient temperature cycle test
Environmental cycling test (high temperature portion only)
Cycling test on flawed cylinder
Cycling after a drop test

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

(For more details see D6.4, recommendation #6)

4.2 RCS Recommendations intended for use optionally

In this category, there is only one recommendation for batch testing (#4.2.1). The test methodology is being proposed optionally, where the manufacturer comes to an agreement with the inspection body.

4.2.1 RCS Recommendation #5

Recommendation to control the resin mixture and curing process parameters (by monitoring temperature, time etc) and/or consider tests in standard **to verify curing of the resin** for cylinders used in transportable, on board and stationary applications.

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

The rationale for recommending this change is that WP4 has demonstrated that there is an important effect of improper curing on the cylinder performance resulting in reduced cycling performance on type 3 cylinders and increased burst pressure scatter on type 4 cylinders.

Also, WP2 performed work at a material scale which shows that the long-term lifetime behaviour of composite materials is highly dependent on the visco-elastic properties of the matrix, that are themselves dependent on the type of epoxy resin used and the curing condition applied.

(For more details see D6.4, recommendation #7)

4.3 RCS Recommendations intended for future consideration

In this category, there are three recommendations intended for future consideration, one for use of a Non Destructive Test (NDT) for example, Acoustic Emission method (4.3.1), observing scatter using probabilistic assessment (4.3.2) while the last item is for reducing safety factor (SF) for transportable cylinders (4.3.3). They are being included for information only as they will need further work.

4.3.1 RCS Recommendation #6

Statement for future consideration in periodic inspection and manufacturing QA:

Consider using a Non Destructive Test for periodic inspection and manufacturing QA for composite pressure vessels used in transportable, on board and stationary applications.

Example: Acoustic Emission (AE) seems to be a very promising technique for manufacturing QA and in-service inspection of composite pressure vessels, but significant improvement of the methodology is needed.

The reason for suggesting this change is that the actual tests proposed in RCS, visual inspection and hydraulic proof test are not suitable for composite cylinders. While the external visual inspection criteria needs to be adapted for composite cylinders (linked to studies of the effects of impact), hydraulic proof test does not give any information of state of damage in composite wrapping due to the difference of failure modes compared to metallic cylinders. AE tests have been used by HyCOMP by different partners in different WPs (WP2-4). It has been noticed that AE seems to be very operator dependant. Also, it was found that AE equipment differs from one supplier to another which can lead to discrepancies in AE results.

(For more details see D6.4, recommendation #8)

4.3.2 RCS Recommendation #7

Statement for future consideration in design and manufacturing requirements:

Add to type test. Recommendation to take into account observed scatter of performance in pass/fail criteria for cylinders used in transportable, on board and stationary applications.

Example: Make a statistical assessment of the key properties. There is a need to look further into Probabilistic assessment as proposed by BAM and HEX, and perhaps to also look at the approach proposed in the DeliverHy project (partner NTNU, Norway) in the future.

The reason for suggesting this change is that from WP3 we have learned that structural integrity can be reliably demonstrated only when scatter of properties are taken into account. Depending on quality of design and manufacturing, significant scatter of performance may be observed.

(For more details see D6.4, recommendation #4)

4.3.3 RCS Recommendation #8

Statement for future consideration in design requirements:

HyCOMP has performed theoretical and experimental studies on flat panels made of specific materials (carbon fibres) demonstrating that safety factor 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). It still needs to be proven whether the results on flat specimen can be extended to composite cylinders or not and whether filament wound structures behave locally like unidirectional composites when they are pressurized. Hence, additional margins taking into account 3-dimensional loading effects (i.e. thick wall effects) must be considered.

Note: Safety factor is defined as the ratio between burst pressure and design pressure.

For transportable applications, **there could be a potential to reduce the minimum safety factor** from 2, if justified by further test results and cylinders must pass type approval tests. Design criteria might depend on the lifetime of the cylinder.

Note: For other applications, safety factor of 1.8 is already used e.g. GTR #13, EU 406/10. See Technical Tables prepared by D5.3, Table 2 - Overview of stress ratio requirements.

The value of 1.4 must be seen as the minimum theoretical value covering intrinsic material properties (variability of carbon fibre properties). A probability of failure of 10^{-6} is considered. A value of 1.6 is found for a probability of failure of 10^{-9}. The methodology to determine this value has been described by WP2 in deliverable D2.4.

(For more details see D6.4, recommendation #2)

5. Proposed path forward

This D7.3 report also proposes a path forward on how the RCS recommendations #1-5 may be integrated into ongoing or new RCS activities.

It is being proposed that the RCS recommendations indicated in **Annex A** be forwarded to the Secretariat of the relevant technical committees for consideration by the technical committee and relevant working groups.

NOTE: Although some pathways are being proposed in the report, it will not be the work of HyCOMP to implement these RCS recommendations.

5.1 Recommendations to be forwarded to the Secretariat of ISO/TC58/SC3

The recommendations indicated in Annex A that are relevant to ISO 11119-2 and ISO 11119-3 shall be forwarded to:

Secretariat: BSI
Secretary: Mr. Stephen Read
Chairperson: Dr. Warren Hepples
ISO Central Secretariat contact: Mme Blandine Garcia

ISO 11119-2: Gas cylinders - Refillable composite gas cylinders and tubes - Design, construction and testing 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 Part 3: “Fully wrapped fibre reinforced composite gas cylinders and tubes up to 450 l with non-load-sharing metallic or non-metallic liners”

5.2 Recommendations to be forwarded to the Secretariat of CEN/TC 23

The recommendations indicated in Annex A that are relevant to EN 12245 *Transportable gas cylinders – Fully wrapped composite cylinders* shall be forwarded to:

Secretary: Mr. Stephen Read
Chairperson: Dr. Chris Jubb

5.3 Recommendations to be forwarded to the Secretariat of ISO/TC 197

The recommendations indicated in Annex A that are relevant to ISO 19984 and ISO 19881 shall be forwarded to:

Secretariat: SCC
Secretary: Mr. Jim Ferrero
Chairperson: Dr. Andrei Tchouvelev l
ISO Central Secretariat contact: Mr. Andrew Dryden

ISO/TC 197, WG15
ISO/CD 19884 Gaseous Hydrogen – Cylinders and tubes for stationary storage

ISO/TC 197, WG18
ISO/AWI 19881 Gaseous hydrogen and hydrogen blends – Land vehicle fuel tanks

5.4 Regulations

Only after these recommendations are accepted by the respective technical committees and the standards are revised, there should be an effort to have these revised standards be referred to in regulations (e.g. EU 406/10, GTR).

6. Technical tables

Note: Both these tables were prepared by WP5, D5.3.

Table 1: Overview of temperature requirements in various RCS

Limit Temperature	(°C)	Comment
Material, Min	-60	EN 12245 Extreme Cycle, Min, Temp
Gas, Min	-40	Usually treated as Ambient, Min, and used as bulk average temperature for the gas containment. Lower temperatures might occur locally and over a short time
Ambient, Min	-40	
Settled	15	State of Charge Reference
Ambient, Max	57	NGV2/JARIS001 Identified limit
	65	ADR
	85	EU406/2010
Gas, Max	85	Maximum temperature level as a result of the filling process and used as bulk average temperature for the gas containment. Higher temperatures might occur locally and over a short time

Observation: In most cases in the RCS the ambient temperature range matches or is defined as the gas (or settled gas) temperature range.

Table 2: Overview of stress ratio requirements in various RCS

Standard	Primary overall Stress Ratio related to NWP for CFRP	MAWP-Filling I Ratio	Minimum Stress Ratio in case of using CFRP
EN 12245	3.0	1.5	2.0 (PH)
ISO 11119-2	3.0	1.5	2.0 (PH)
ISO 11119-3	3.0	1.5	2.0
ISO 11439	2.35	1.3	1.8
ASME	2.25	1.25	1.8
EU 406/2010	2.25	1.25	1.8
JARI S001	2.25	1.25	1.8
GTR	2.25	1.25	1.8

The unifying parameter across the different applications is the minimum stress ratio between burst and maximum fill pressure.
Observation: In most cases in the RCS the minimum safety factor or stress ratio over maximum pressure ranges between 2.0 to 1.8.

7. Annex A Table of RCS Recommendations

RCS Recommendation	**Rationale**	**Other elements to be considered**	**Applicable Standards**
1. Add a specification. Reduce the Design Pressure to the Maximum Developed Pressure for <u>transportable</u> applications for dedicated Gas Service (*e.g. Hydrogen)* instead of using the test pressure.	Hydrogen shows a very low expansion compared to other gases at high temperature.	The standard shall require a marking be added to inform the user that these cylinders are to be used for hydrogen service only.	Applies to Transportable. Already defined in ISO 11119-3 standard. Maximum developed pressure is not defined in EN12245.
2. Add a specification for the glass transition temperature **(T_g)** to be higher than T_{max} +X°C as defined by the application.	Temperature is an important parameter influencing damage accumulation (fibre breaks) in the composite wrapping		Applies to Transportable, On-board and Stationary. ISO 11119-2 ISO 11119-3 EN12245 ISO/AWI 19881 ISO/CD 19884
3. Change type test. Perform relevant tests at elevated temperature, at a temperature **not less than T_{max}**, as defined by the application.	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}.		Applies to Transportable, On-board and Stationary. ISO 11119-2 ISO 11119-3 EN12245 ISO/AWI 19881 ISO/CD 19884
4. Change type test. Perform relevant tests at maximum developed pressure for <u>transportable</u> applications for dedicated Gas Service (*e.g. Hydrogen)* instead of using the test pressure or only NWP.	Cylinders will not be pressurized beyond the maximum developed pressure. Need to demonstrate that cylinder will withstand the most severe loads (not more).	The standard shall require a marking be added to inform the user that these cylinders are to be used for hydrogen service only.	Applies to Transportable. Already defined in ISO 11119-3 standard. Relevant tests include: Ambient temperature cycle test. Environmental cycling test. (high temperature portion only) Cycling test on flawed

RCS Recommendation	Rationale	Other elements to be considered	Applicable Standards
			cylinder. Cycling after a drop test. Maximum developed pressure is not defined in EN12245. Relevant to tests 6-10.
5. To control the resin mixture and curing process parameters (by monitoring temperature, time, etc.) and/or consider tests to verify curing of the resin.	There is an important effect of improper curing on the cylinder performance.	The test methodology (Barcol) is being proposed <u>optionally</u>, where the manufacturer comes to an agreement with the inspection body.	Applies to Transportable, On-board and Stationary. ISO 11119-2 ISO 11119-3 EN12245 ISO/AWI 19881 ISO/CD 19884

8. Symbols and Acronyms

Symbols

P_h	test pressure (= 1.5xNWP). Also known as design pressure, hydraulic proof pressure or maximum allowable working pressure (MAWP)
Pmax	maximum developed pressure at 65 °C for transportable, 85 °C for on-board
T_{max}	maximum temperature (65 °C for transportable, 85 °C for on-board)
T_g	glass transition temperature (for epoxy resin)

Acronyms

AE	acoustic emission
JTI	Joint Technology Initiative
MAWP	maximum allowable working pressure
NDT	non destructive testing
NWP	nominal working pressure at 15°C
RCS	regulations, codes and standards
SF	safety factor (ratio between burst pressure and design pressure)
iSF	intrinsic safety factor