Joint Research Centre

the European Commission's in-house science service

Serving society
Stimulating innovation
Supporting legislation

European Commission GTR-13 Phase II: ITEMS TO BE CONSIDERED

Pietro MORETTO

Beatriz ACOSTA

October 2017, Brussels

European Commission

Objectives of this presentation

This presentation aims only at summarising recent R&D and PNR work, and proposing discussion topics.

It is not a consolidated proposal from EC.

Topics will require are prioritisation and ranking also in term of a timeline

European Commission
JRC = Joint Research Centre
Directorate Energy, Transport & Climate

JRC site in Petten - The Netherlands

European Commission

Some items for discussion

- Verification tests for service terminating performance in fire
- Material qualification & hydrogen compatibility
- Verification test for baseline metrics: initial burst pressure and pressure cycle life
- Verification test for on-road performance: sequential pneumatic tests
- Surface damage test – Impact test
- Hydrogen sensors

FCH JU project FireCOMP

Broad focus: automotive application, stationary application, transportable cylinders, bundles and tube trailers.

- ✓ Better understanding of heat transfer mechanisms and the loss of strength of composite high-pressure vessels in fire conditions.
- ✓ Modelling of the thermo-mechanical behaviour of these vessels developed and validated by full scale fire tests.

Example of results:
36 litre type 4 tank exposed under different hydrogen pressures to extreme engulfing fire conditions: the tank burst in 200 sec when pressurized at 70 MPa and in 300 sec when pressurized to 50 MPa; when filled with 30 MPa and 20 MPa the tank leaked after 400 and 500 sec respectively.

FireCOMP recommendations

The proposal is a new bonfire test procedure, based on a separation into two independent sub-tests:

Test 1 concerns composite cylinder material characterization. It is done on cylinder only without any protections, with objective of getting information about time to failure and failure mode.

Test 2 concerns the protective devices selected for the application considered. For example, if the risk analysis underlined the need for a TPRD, this test will aim at checking its good opening within due time, its flow rate and the obtained depressurization curve.

Recommendations for improved test have been discussed with expert of ISO TC58 Gas Cylinders.

Source: FireCOMP deliverable D6.5

Further details to be discussed by and with FireCOMP partners

GTR13 - VERIFICATION TESTS FOR SERVICE TERMINATING PERFORMANCE IN FIRE

- Firefighters feedback*: If TPRD fails to be initiated or if fire affects only part of the CHSS 12 minutes would be insufficient for fire recognition/first responders actuation
- Large exit diameter of TPRD (4-6 mm) for quick H2 release => flame length & separation distance are ~50 m** and pressure peaking phenomena***
- FireCOMP project****: 12 minute guarantee for structural integrity might not be enough

*Captain S. Cardou, F. Verbecke, Colonel S. Delaunay, Colonel M. Gentilleau, et. al. Firefighters' feedbacks on real life FCEV TPRD releases in traffic accident and garage fires, ICHS 2017
**S. Tretsiakova-McNally, D. Makarov, HyResponse project, Basics of hydrogen safety for first responders, Lecture on Safety of hydrogen storage, http://www.hyresponse.eu/files/Lectures/Safety_of_hydrogen_storage_notes.pdf
***Shentsov, V., Kuznetsov, M., Molkov, V., The pressure peaking phenomenon: validation for unignited releases in laboratory-scale enclosure, ICHS 2015.
****FCH JU funded project FireCOMP: Modelling the thermo-mechanical behaviour of high pressure vessels, made of composite materials when exposed to fire conditions, http://www.firecomp.info/
****P. Blanc-Vannet, S.Jallais, B.Fuster, F.Fouillen, D.Halm, T.van Eekelen, S.Welch, P.Breuer, S.Hawksworth, Fire tests carried out in FCH JU fireCOMP project, recommendations and applications to safety of gas storage systems, ID137, ICHS 2017.

Bonfire test

Recent work (e.g. project H2FC, UU) has shown that **calibration of the fire sources** in term of radiant heat is critical. Flames with the same local temperatures do not deliver the same heating values, with completely different results for the tank degradation TPRD behaviour and pressure relief.

To be further presented by Prof. Molkov

MATERIAL QUALIFICATION & HYDROGEN COMPATIBILITY: Metallic Components

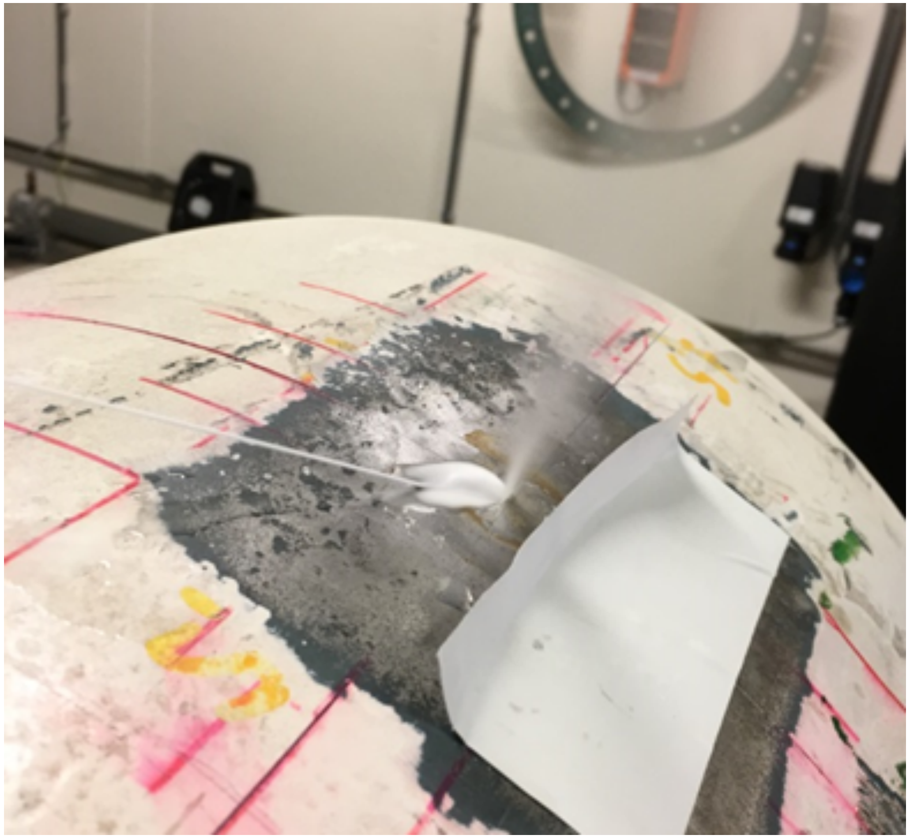
- Better understanding of crack initiation and propagation in metallic components: Different mechanical loads, effect of hydrogen pressure stress intensity factor thresholds.

  → **How to consider in standards crack initiation and propagation under H_2?**

European Commission

The MATHRYCE Project

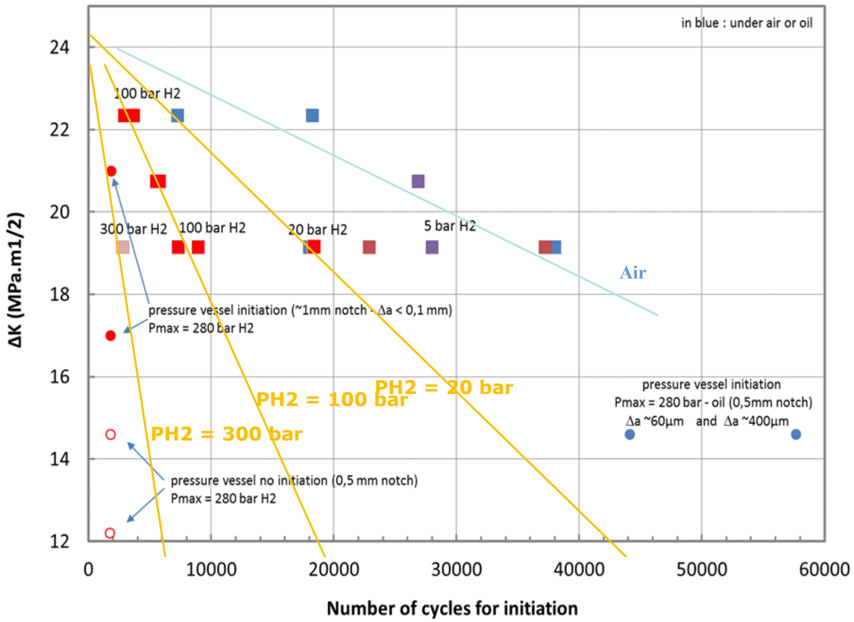
Material Testing and Design Recommendations for Components exposed to HYdrogen enhanCed fatiguE



Failure mode:
Hydrogen enhanced fatigue
for metallic vessel and metal liner of composite cylinders

European Commission

MATHRYCE Findings



- ✓ All the cylinders hydraulically tested failed by LBB, with cracks growing from the deepest notches.
- ✓ Hydrogen cycle test induced LBB in two of the four tested cylinders.
- ✓ Hydrogen accelerated the crack initiation from the deepest notches
- ✓ The number of cycles to initiation decreases as the pressure is increased.
- ✓ It seems confirmed a fatigue reduction factor of 12 at high pressure (p=300 bar).

An empirical formulation for crack initiation given

$$\ln(N) \approx 12.1 - \left(\frac{\Delta K}{16.7MPa\sqrt{m}}\right)^{3} - \left(\frac{P_{H_2}}{4MPa}\right)^{0.5}$$

A Fracture Mechanics methodology for metallic cylinders design / lifetime assessment based on lab-scale tests and taking into account Hydrogen Enhanced Fatigue is suggested

www.mathryce.eu

European Commission

Proposal Metallic Components:

- ✓ it is recommended that materials used for hydrogen containment in stationary vessels must satisfy the LBB assessment in hydraulic testing conditions, even if the design code do not recommend it.
- ✓ There is still need for proper harmonized determination of K_{IH}.

- ✓ Consider including hydrogen compatibility tests for metals (example, those of the last version of the SAE J2579).
- ✓ Consider including hydrogen compatibility requirement on other components of the CHSS like joints, valves, and seals.

European Commission

MATERIAL QUALIFICATION & HYDROGEN COMPATIBILITY: Polymers and plastic materials

- Characterization of mechanical performance of polymers under hydrogen: blistering and swelling and reversibility of these effects
- Effect of high and low temperature excursions in compressed hydrogen storage system materials

Consider revision of material tests on polymers performance under hydrogen:

- ✓ Consider including softening temperature tests and/or other similar tests for CHSS polymeric material qualification. Stablishing softening temperatures ≥ 100° C
- ✓ Consider including tests to qualify the ductile properties of the plastic materials and welds at temperatures ≤ -40 ° C (possibly at -50° C).

European Commission

MATERIAL QUALIFICATION & HYDROGEN COMPATIBILITY: Composite materials

- ➢ The resin used in the composite wrapping is an important part for the safety of the high pressure storage tank. Temperatures above the glass transition temperature, viscous flow phenomena can have an effect, resulting in stress concentration and damage accumulation in the laminate.

In the SAE J2579, the resin's shear strength and the glass transition temperature shall be determined. ISO/DIS 19881:2017, requires that the resin system materials have a glass transition temperature of at least 20° C above the maximum container temperature (i.e ≥105° C). **A minimum difference of 30° C between the maximum operating temperature and the glass transition temperature has been proposed in the FCH JU funded project HYCOMP***

- ✓ Consider including tests on resin (binder) of the composite wrapping. Consider revision of the glass transition temperature being at least 20° C above the maximum container temperature (possibly 30° C)

HYCOMP European Project, Final WP6 report to be included as appendix of WP7 report – Recommendations for Industry and RCS, C Devilliers, - Public Deliverable 6.4 (www.hycomp.eu), 2014
H. Barthélémy, M. Weber, F. Barbier, J. Furtado, B. Fuster, O. Bardoux, Hydrogen storage: recent improvements and industrial perspectives, ID 293, ICHS 2017.

VERIFICATION TESTS FOR BASELINE METRICS: initial burst pressure

- In the GTR-13 an additional requirement corresponding to minimum burst pressure ratio (ratio of minimal burst pressure to nominal working pressure) of 2.0 for unused containers has been under consideration as a screen for minimum new containers capability with potential to complete the durability test sequence requiring burst pressure above 1.8 burst pressure ratio considering < ± 10 per cent variability in new containers strength.
- However, the historical minimum, 2.25 has been adopted in this document as a conservative placeholder without a quantitative data-driven basis but instead using previous history in some Contracting Parties.

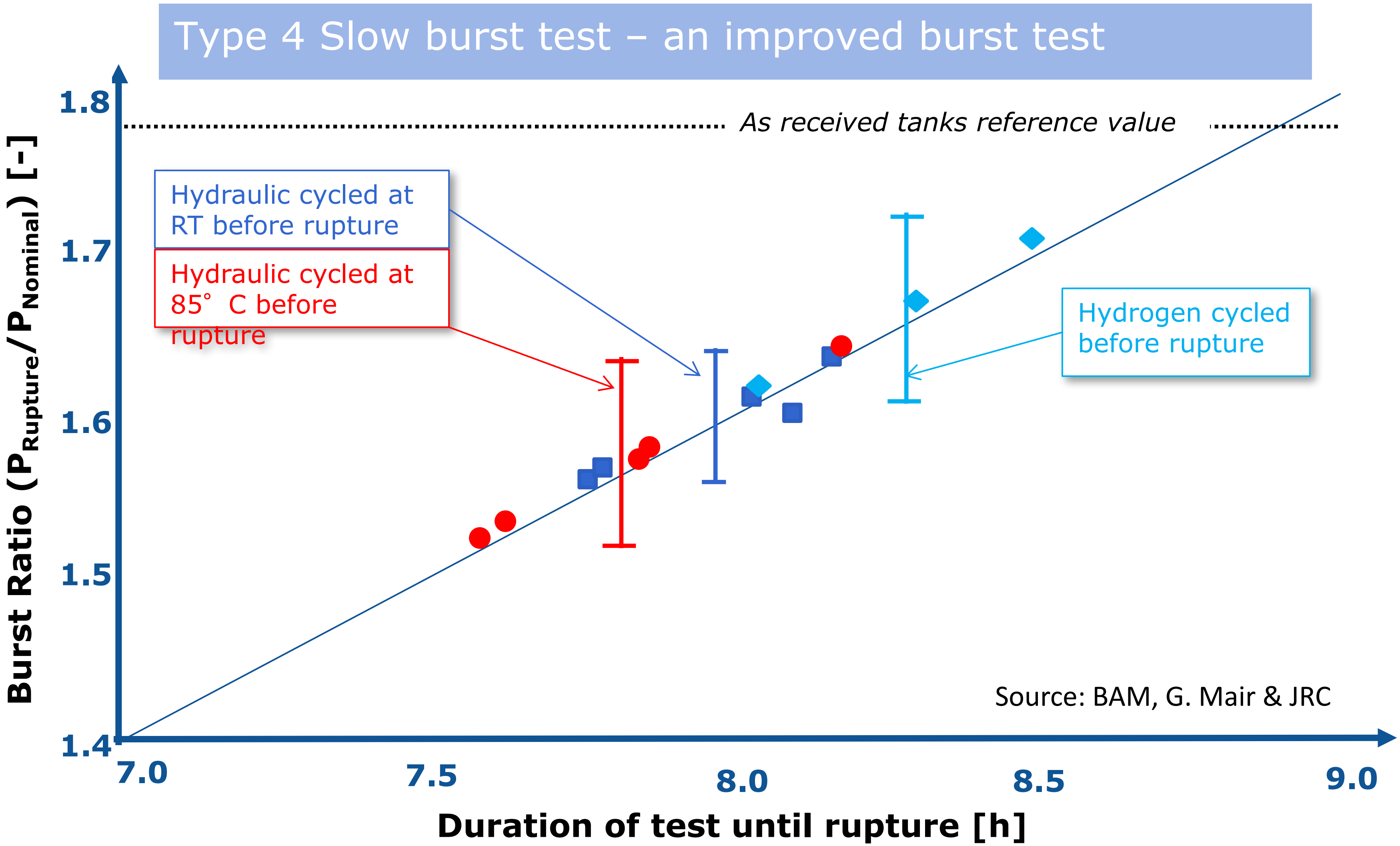
HYCOMP Project

✓Scope:

Develop a better understanding of the damage accumulation processes in the composite wrapping and the degradation rate as a function of the type of load (cyclic or sustained) and environmental conditions.

http://www.hycomp.eu/.

Pneumatic vs. hydraulic cycling



European Commission

HYCOMP conclusions of test results
Type 4 cylinders

- ✓ Cycling has a very small effect on the composite wrapping compared to metal liners! Therefore cycling to failure is not efficient, sometimes impossible.
- ✓ A sustained load influences the residual cycle strength: higher slow burst strength with constant or reduced scatter.
- ✓ Gaseous cycle loads result in a lower degradation compared to high or ambient temperature cycling.

Se further G. Mair presentation

N. de Miguel, G. Mair, B. Acosta, M. Szczepaniak, P. Moretto, Hydraulic and pneumatic pressure cycle life test results on composite reinforced tanks for hydrogen storage, Paper No. PVP2016-63568, pp. V01AT01A046; 10 pages, doi:10.1115/PVP2016-63568

Proposals for baseline metrics

- ✓ Results have shown that there is no risk of losing residual strength on Type 4 tanks after the hydrogen cycling test suggesting the minimum burst pressure ratio of unused containers could be lowered (to 2.0).
- ✓ For type 3 tanks, consider evaluating the residual strength by hydraulic cycling the cylinders until failure.
- ✓ Statistical assessment of test results for CHSS qualification will provide a better insight of the real container capability. This could imply a statistically significant number of containers.

- ✓ Consider an assessment criterion which ensures both, sufficient minimum load cycle strength and a high reliability against failure.
- ✓ Recheck the minimum number of cycles in which no leakage may occur.
- ✓ Limit the scatter of load cycle strength for approved cylinder populations.

VERIFICATION TESTS FOR FOR ON-ROAD PERFORMANCE: SEQUENTIAL PNEUMATIC TESTS

- Cycling at high temperature: Results from HYCOMP, different behaviour of type 3 and of type 3 tanks.
- Ambient and extreme temperature gas cycling tests: the fill rate is controlled based on the SAE J2601 to a constant 3-minute pressure ramp rate; the temperature of the hydrogen fuel dispensed to the container is controlled to the specified temperature. The pressure ramp rate should be decreased if the gas temperature in the container exceeds +85 °C

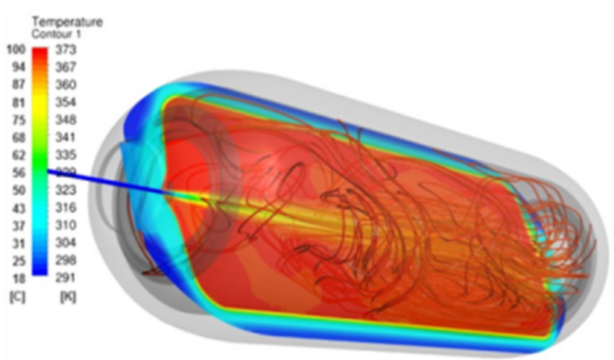
Risk Assessment on refuelling at ISO TC197 WG24 or "*Where mobility meets infrastructure*"

A first attempt to answer these questions:

- **Could it happen that the dispenser sets the refuelling condition wrongly?**
- **What does happen then at the on-board CHSS?**

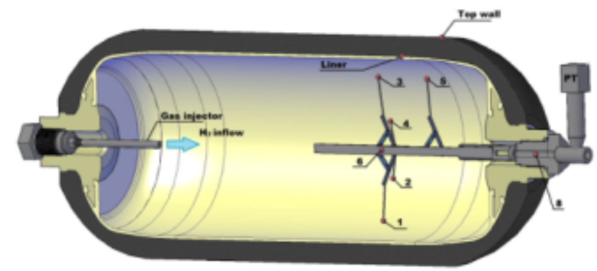
Project HyTransfer
Advanced and validated refuelling protocol

HyTransfer

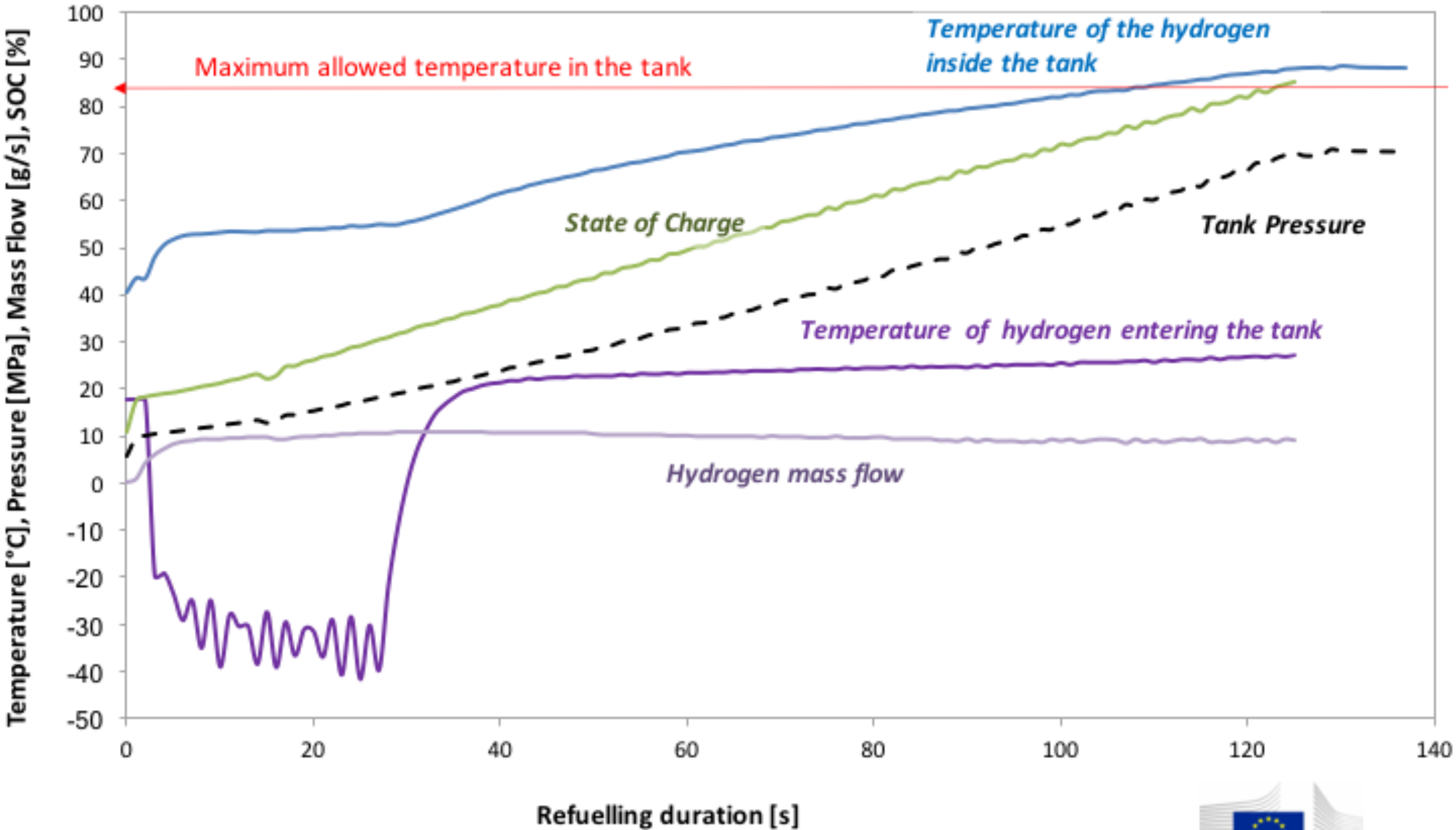


European Commission

Effect of loss of pre-cooling



Type 3 tank, 40 litres, Ambient temperature 40 °C
Mass flow 9.6 g/s
H2 at -40 °C for 20 seconds and then the cooling fails

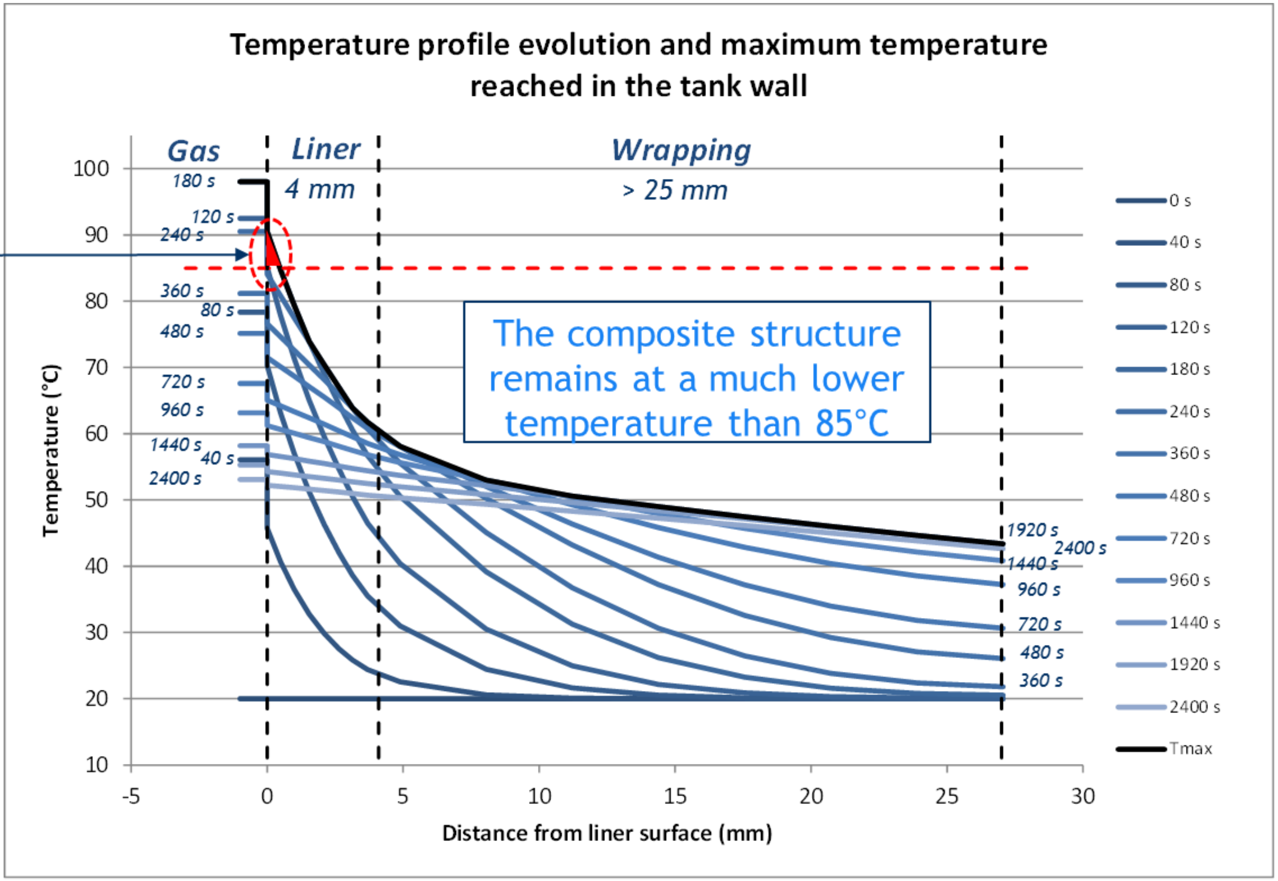


T(gas) versus T(liner)

HyTransfer

www.hytransfer.eu

Only a very small amount of material is exposed, during less than 2 min, to temperatures above 85°C, without exceeding 95°C



Coordination | Industry | Research & Testing | Regulations, Codes & Standards | Funding

European Commission

HyTransfer recommendation 2

In (very) few cases the T(gas) during refuelling can trespass the 85 °C
What to do with the tank afterwards? Dispose because not certificated anymore (see TUV)?

One option could be to take into account these (very) few cases in the type-approval testing of the CHSS.

It corresponds to the HyTransfer recommendation for Hot case - *Liner material temperature rating,* which **suggest a <u>Tank design verification test</u>: adjust the gas cycling test to verify that the tank withstands pressure cycling with a peak gas temperature of 98 °C.**

Further details to be discussed by and with HyTransfer partners, including other recommendations on T(gas) gradient

Proposals for verification test for on-road performance

- ✓ Consider increasing the ambient temperature for pneumatic cycle test to 65 ° C. It is suggested to perform analyses on the combined effects of an environment at 65° C and 95% relative humidity.
- ✓ Consider reducing the low temperature pneumatic cycling to -25° C but adding a test where the tensile properties of the plastic materials are measured at a temperature ≤ -40° C.
- ✓ Consider increasing the high temperature pneumatic cycling to 90° C and moreover adding a test where the softening temperature of the polymeric materials is measured at a temperature as high as 105° C.
- ✓ Include performance based qualification test to demonstrate that liner buckling will not occur under operating conditions. Consider to add a rapid depressurization test. Alternatively, mitigate depressurization by means of e.g. restricting valves.
- ✓ Examining the tank liner periodically (after each series of gas cycling and high pressure hold) requiring that the tank liner should not show cracks. This is in line with the proposal of hydrogen compatibility tests inclusion.

SURFACE DAMAGE TESTS: Impact

http://www.hypactor.eu/

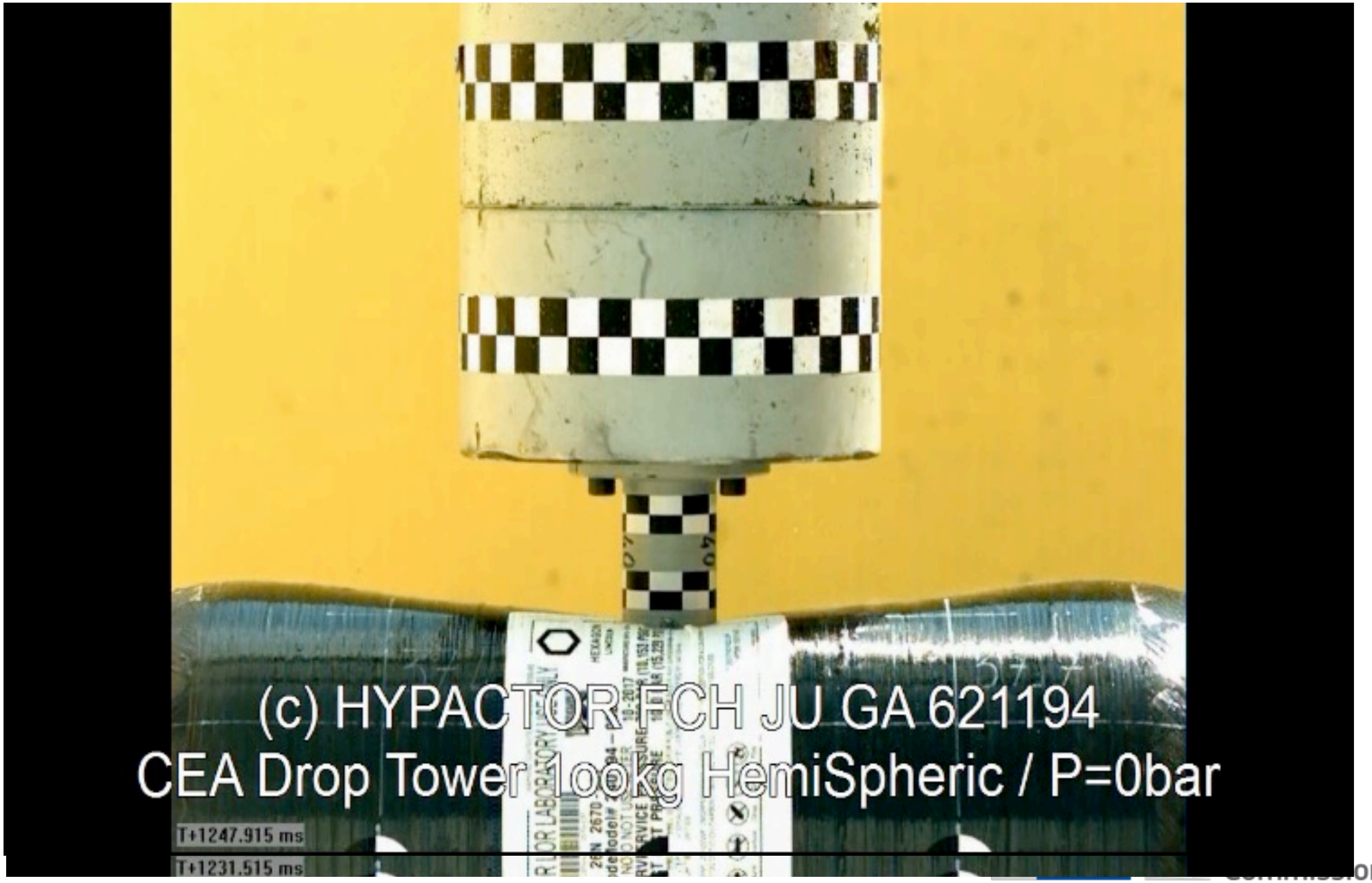
COPV resistance to mechanical impact

- Project has investigated short term (burst) and long term (cycling) performance of impacted cylinders.

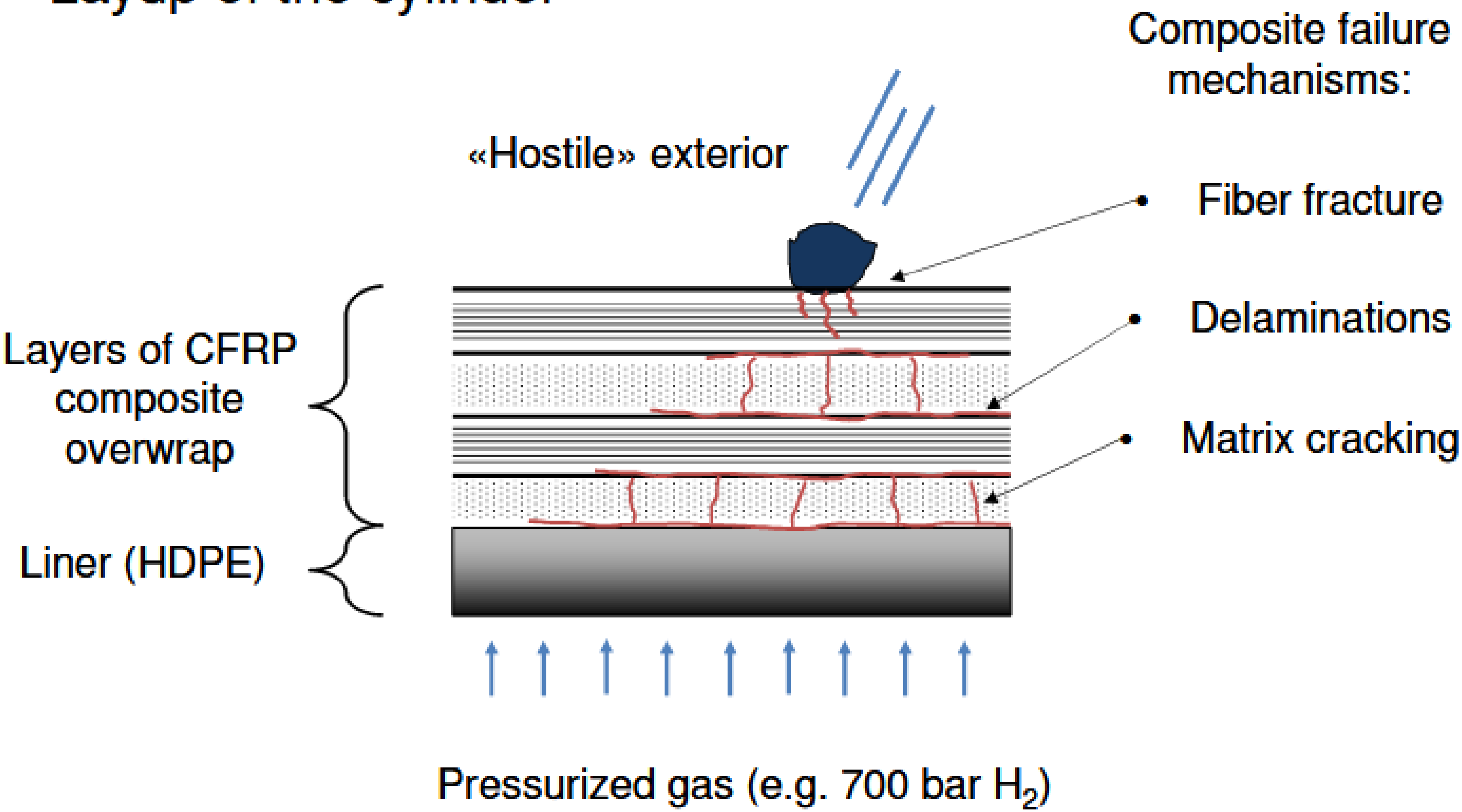
- From the results of a type 4 tank with a base burst pressure of 2.25*NWP, by using a 56 mm diameter hemispherical impactor, an impact with an energy of 1080 J or higher (on an empty tank) would result in a reduction of the burst pressure

Project HyPactor

http://www.hypactor.eu/press-room/video.html



- Layup of the cylinder



K. Lasn, Baltic Polymer Symposium 2016.

HYPACTOR recommendation for cylinder design qualification

The impact energy a composite cylinder can absorb depends mostly on:

- Structural composite thickness (driven by minimum burst requirement and diameter)
- Pressure in the cylinder when the impact happen

Requesting the same level of impact energy for all vessels from 1L to 10 000L and pressures from low pressure to more than 1000 bar does not make sense.

Type 4 cylinders have demonstrated little to no reduction in cycle performance. This might not be valid for other types of cylinders.

European Commission

HYPACTOR recommendation for cylinder design qualification

HYPACTOR recommend to introduce **modified IMPACT TESTS** in cylinder qualification test programs in order to determine the impact capacity of each specific cylinder design.

A proposal of a standard ISO/DIS 19016 (Gas cylinders - Cylinders and tubes of composite construction - Modal acoustic emission (MAE) testing for periodic inspection and testing) is under construction

European Commission

HYDROGEN SENSORS

<u>GTR text:</u> following crash tests, hydrogen releases could be measured via reduction in oxygen content

JRC & NREL have shown that this method of hydrogen detection inadequate for safety applications.

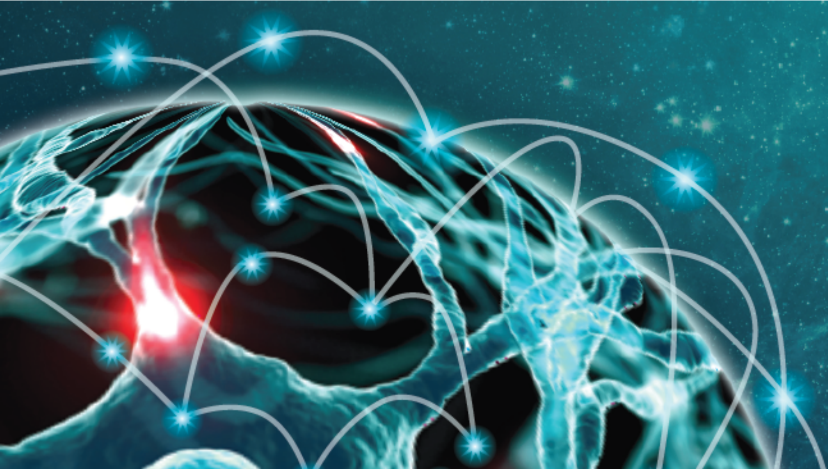
1. Normal drift in the oxygen sensor could lead to hydrogen false positive alarms, or even worse, false negative alarms.
2. In a close environment, a hydrogen release may lower the vol% O_2, but would not affect PO_2. The oxygen sensor signal is also very much dependent upon fluctuations in environmental parameters, especially temperature and pressure.

W. Buttner, C. Rivkin, R. Burgess, K. Hartmann, I. Bloomfield, M. Bubar, M. Post, L. Boon-Brett, E. Weidner, P. Moretto, Hydrogen monitoring requirements in the global technical regulation on hydrogen and fuel cell vehicles, International Journal Hydrogen Energy 2017; 42: 7664e71.

W.J.Buttner. R. Burgess, C.Rivkin, M.B.Post, L.Boon-Brett, V.Palmisano, P.Moretto, An assessment on the quantification of hydrogen releases through oxygen displacement using oxygen sensors, International Journal Hydrogen Energy 2014; 39: 20484e90

Proposals regarding hydrogen sensors

- ✓ Remove the text endorsing oxygen displacement as a means for quantifying hydrogen releases into vehicle compartments.
- ✓ Reconsider the position of hydrogen and the impact sensors in the post-crash leak test.
- ✓ Consider recommending thermal conductivity sensors for verification of fuel system integrity following crash tests.
- ✓ Consider the access to the sensor output in real time and a hold period of 1 hour following impact.



Thank you!

Stay in touch

JRC Science Hub: www.ec.europa.eu/jrc

beatriz.acosta-iborra@ec.europa.eu

Twitter: @EU_ScienceHub

LinkedIn: european-commission-joint-research-centre

YouTube: JRC Audiovisuals

Vimeo: Science@EC

European Commission