European Train the Trainer Programme for Responders

Lecture 4

Compatibility of hydrogen with different materials

LEVEL IV

Specialist officer

The information contained in this lecture is targeted at the level of specialist officer and above.

This topic is also available at levels I-III.

This lecture is part of a training material package with materials at levels I – IV : Firefighter, crew commander, incident commander and specialist officer. Please see the lecture introduction regarding competence and learning expectations

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Summary

The present lecture gives an overview of hydrogen interaction with different types of materials and hydrogen permeation, which are extremely relevant to hydrogen storage technologies. Although hydrogen is a non-corrosive gas, the reaction of hydrogen with some metals at high temperature may form corrosive hydrides, which then generates gas bubbles within the metal lattice, known as blistering. At low temperatures, some metal could become more brittle due to the change from ductile to brittle behaviour mode, which is called cold embrittlement. The interaction of hydrogen with polymer could also lead to swelling, blistering and deterioration of the polymer, increasing the permeation rate of hydrogen through the polymer matrix. The permeation rate of hydrogen through metallic containers (i. e. Type I and Type II) or containers with metallic liners (i. e. Type III) is negligible. However, the hydrogen permeation rate through Type IV containers must be correctly controlled to a very low value, to avoid the concentration of hydrogen reaching the LFL of hydrogen in air (4.0 vol. %).

Keywords

Hydrogen embrittlement, metal, polymer, blistering, hydrogen permeation, mitigation

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1. Target audience

The information contained in this lecture is targeted at the level of specialist officer and above. This lecture is also available at levels I: Fire fighter.

The role description, competence level and learning expectations assumed at specialist officer level are described below.

1.1 Roll description: Specialist

Specialists assist Incident Commanders with advice and by directing and supervising technical operations that involve the use of knowledge, skills or equipment related to a specific risk or response activity. Typical incidents involve hazardous materials, transportation vehicles, climatic events, structural and other built environment failures and emergency response logistics.

1.2 Competence level: Specialist

A scientific, engineering and empirical foundation in the subject area with skills, extended by experience, to apply that information, knowledge in an interpreted and useful way to enable the Incident Commander and other first responders under the specialist's direction to respond effectively and safety to the emergency situation.

1.3 Prior learning: Specialist

EQF 5 Comprehensive, specialised, factual and theoretical knowledge within a field of work or study and an awareness of the boundaries of that knowledge. A comprehensive range of cognitive and practical skills required to develop creative solutions to abstract problems. Exercise management and supervision in contexts of work or study activities where there is unpredictable change; review and develop performance of self and others.

2. Introduction and objectives

The topic of hydrogen interaction and compatibility with different materials is vast. In this section of the lecture two different aspects will be considered: the interaction of hydrogen with metallic and polymeric materials, which are primarily used for the storage vessels. Due to the small size of its molecules and atoms, hydrogen can be easily absorbed by different materials including those used for hydrogen storage. This, in turn, leads to the degradation of the materials mechanical properties, which may result in unwanted hydrogen leaks and structural failures.

The aim here is to provide responders with sufficient knowledge to make relevant decisions. The interaction of hydrogen with the materials is pertinent to all FCH applications. However, in addition to being compatible with hydrogen, the materials used for storage are often subjected to high pressures, low temperatures, and cyclic or static loading. Thus, they must be selected accordingly. The selection of materials compatible with hydrogen is addressed in ISO standards applicable to FCH technologies (more detailed information on relevant RCS in the lecture of 'Regulations, codes and standards for First Responders' of the HyResponse project, http://www.hyresponse.eu/files/Lectures/RegulationCodesStandards_slides.pdf).

'Hydrogen has a low viscosity and small atoms that can be absorbed into materials, so leaks and embrittlement of certain materials are possible, which can result in structural failure' [1]. Mechanical degradation of structural materials under the influence of hydrogen is a serious problem and caused many incidents/accidents during production, storage, transportation and use [2]. The correct selection of suitable materials for the components is a crucial for safety of hydrogen storage systems. This relates to piping, walls of storage vessels, filling connectors, valves, fittings, etc. The silent movie produced in 1950s by Delft University illustrates how hydrogen bubbles emerge from steel at defects and other locations (https://www.youtube.com/watch?v=bv9ApdzalHM).

By the end of this lecture responders will be able to:

- Explain the mechanisms of hydrogen interaction with metallic and polymeric materials;
- Establish effect of hydrogen embrittlement on safety of hydrogen storage systems;
- Define the hydrogen permeation phenomena;
- Point out the safe permeation rate for hydrogen storages on-board of passenger cars and buses.

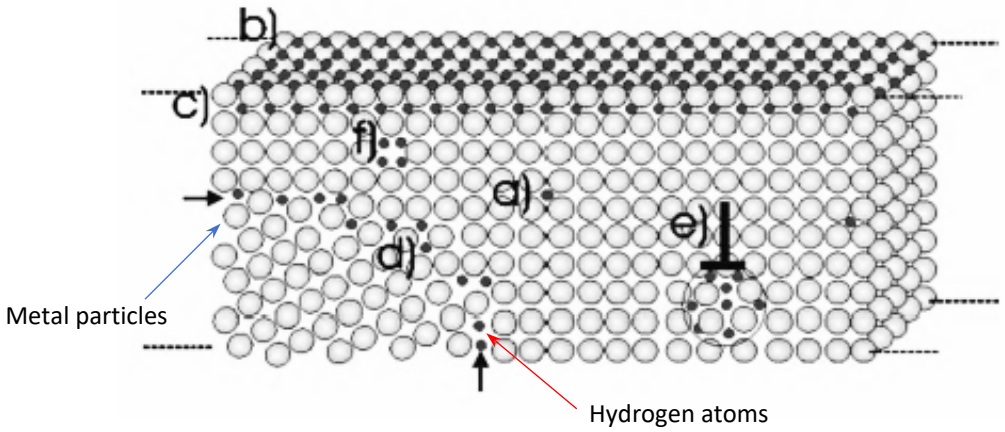
3. Interaction of hydrogen with metals

The compatibility of hydrogen with metals is affected by chemical interactions and physical effects, which include:

- Corrosion: dry corrosion (at high temperatures, *hydrogen attack*); wet corrosion (most common, caused by moisture); corrosion caused by impurities in a gas.
- Hydrogen embrittlement (HE).
- Embrittlement at low temperatures ('cold embrittlement').
- Violent reactions (e.g. ignition).

Dry corrosion is a chemical reaction between a dry gas and a metal, which eventually may lead a reduction of a cylinder wall thickness [3]. This type of corrosion is not very common, because its rate is very low at ambient temperature. However, at high temperatures hydrogen can react with some metals, forming hydrides for example. In general, wet corrosion can occur in a hydrogen storage vessel following the water entry. Please note that hydrogen is a non-corrosive gas and cannot even in wet conditions cause this type of corrosion. At low temperatures some metals can become more brittle. This is due to a change from ductile to brittle behaviour mode when the temperature is lower than a "nil-ductility" temperature, which is sometimes considerably higher than that of the cryogen. It represents a problem for installations exposed to periodic temperature cycles. Several accidents involving a cryogenic storage tank were caused by cold embrittlement.

The phenomena of hydrogen embrittlement (HE) and hydrogen attack will be discussed in more details further. Embrittlement is a process, by which various metals, mainly high-strength steels, become brittle (i.e. lose their ductility) and crack after being exposed to hydrogen [1]. It is caused by ingress of either molecular or atomic hydrogen into a metal lattice. It occurs at relatively low temperatures (e.g. at ambient). Hydrogen attack happens at higher temperatures, above 200 °C. The exact mechanism of HE is not clear. Several mechanistic routes are suggested as shown on Figure 1: (a) formation of hydrogen solution in a metal lattice; (b) hydrogen adsorption on the surface and (c) on the subsurface of a metal; (d, e, f) hydrogen accumulation in structure defects such as grain boundaries, and vacancies dislocations. Also, hydrogen can form compounds within a metal lattice such as metal hydrides or methane [4].



Source: Kirchheim and Pundt (2014) [4]

Figure 1. Suggested routes for hydrogen entry into metal/metal defects.

High-strength steels are most sensitive to the HE. The sources for the HE include:

- Some operations of steel manufacturing such as welding, electroplating, pickling, etc.
- Hydrogen formed as a by-product of wet corrosion of a metal.
- Metal surface treatments (e.g. cathode protection of metal against corrosion).
- Adsorption on a metal surface.

HE is categorised as follows [5]:

- Environmental HE - occurs when a material is being exposed to a hydrogen atmosphere, e.g. in storage tanks.
- Internal reversible HE - occurs when hydrogen enters a metal during its processing. This type of HE may lead to a structural failure of a material that has never been exposed to hydrogen before.
- Hydrogen reaction embrittlement - occurs at higher temperatures, when hydrogen chemically reacts with a constituent of a metal, forming a new microstructural element or phase such as a hydride or to generate gas bubbles also known as *blistering* (see Figure 2).

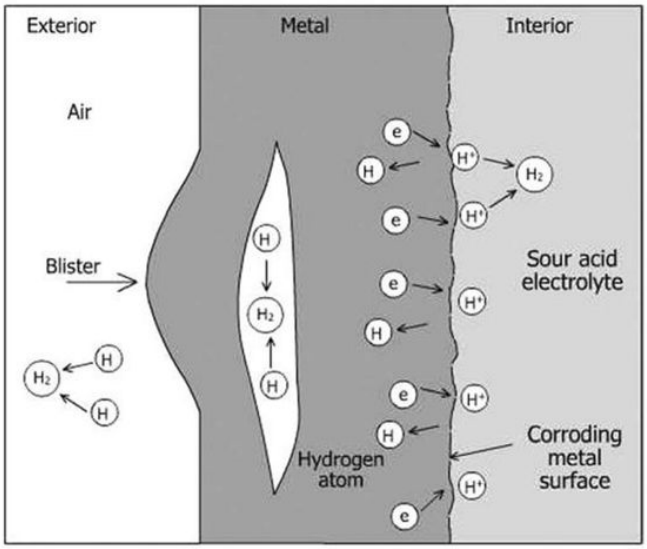


Figure 2. Formation of a blister caused by hydrogen reaction embrittlement

A whole range of factors influence the level of HE process [5]:

- Material:
  - Microstructure
  - Chemical composition
  - Heat treatment and mechanical properties

- Welding
- Cold working (strain hardening)
- Non-metallic inclusions

➢ Environment:

- Hydrogen purity
- Hydrogen partial pressure
- Temperature
- Stress and deformation
- Exposure time

➢ Design and surface conditions:

- Stress level
- Stress concentration
- Surface defects

The evaluation and selection of materials suitable for hydrogen service should be done well in advance of their use for FCH applications. A material should not be used unless the data are available to prove that it is suitable for the planned service conditions. In case of any doubt the material can be subjected to HE susceptibility testing (e.g. ISO 11114-4). As per standard ISO/TR 15916: 2004 HE is possible for any metal [6]. However, some metals can be used without any precautions. For example, brass and copper alloys (e.g. beryllium copper CuBe); aluminium and its alloys. On the contrary, metals that are highly sensitive to HE include: nickel and high content nickel alloys; titanium and its alloys. As for steels, the susceptibility to HE may depend on several factors outlined above. Many materials can be safely used under controlled conditions (e.g. limited stress, absence of surface defects, etc.) [6].

As it was mentioned earlier inadequate choice of materials will lead to incidents or serious accidents. If a material is affected by HE it may fail prematurely and sometimes in catastrophic manner when stress is applied. There are several examples of incidents associated with an incorrect selection of a material [2].

(1) A pipe failure at a hydrogen production plant.

The incident occurred in 1996. ‘A pipe rupture occurred in a steam methane reformer (SMR) process that produces hydrogen and exports steam. The rupture occurred in a 24-inch diameter stainless steel (SS) pipe used to allow the process gas flow to bypass the high-temperature shift (HTS) converter during start-up. When the pipe ruptured, process gas contained in process equipment located upstream and downstream of the break vented into the SMR plant yard area. The vented process gas was a mixture of hydrogen, carbon monoxide, carbon dioxide, steam,

and methane at 550 psig and 650 deg F (38 bar and 343 °C). The escaping high-pressure gas caused an energy release and subsequent fire. The fire was confined within the SMR plant, but equipment located near the pipe failure was damaged. The SMR plant distributed control system (DCS) worked properly to automatically shut down the process, thereby limiting the amount of process gas that escaped. Emergency response services were provided on the scene by the local county fire department within minutes of the incident and the fire was extinguished within 10 minutes. There were no injuries and no releases of any acutely hazardous materials covered by the Risk Management Prevention Plan (RMPP), EPA CERCLA, and SARA Title III regulations [2].

Metallurgical examination of the failed pipe segment revealed cracking on the pipe interior. The cracks were the result of stress corrosion cracking (SCC) of SS, caused by the presence of caustic (potassium hydroxide). The most likely source of the caustic was small amounts of potassium promoter normally present in the SMR catalyst. The section of the HTS bypass piping where the rupture occurred was normally cool and non-flowing. The piping layout allowed water and potassium hydroxide condensed from the process gas stream to collect in a low point. The SS material of construction of the bypass piping was susceptible to SCC from the concentrated caustic solution collecting in the bypass piping'. Materials incompatibility was one of the causes of the incident. As a result 'The SS 24-inch pipe that failed was replaced with 1-1/4 Cr 1/2 Mo alloy pipe that is corrosion-resistant to SCC. A revised HTS bypass piping layout was installed to prevent the hazardous conditions that lead to the failure. A detailed hazard review and evaluation of all of the materials of construction in the hydrogen plant process gas system led to preventively changing several other pieces of piping and equipment items in the SMR process to 1-1/4 Cr 1/2 Mo [https://h2tools.org/lessons/pipe-failure-hydrogen-production-plant].

(2) An explosion of hydrogen gas caused by the breakage of external gas duct at a space rocket testing facility.

An incident happened in a laboratory, on May 16, 1991 in Kakuda, Miyagi, Japan. 'An explosion occurred at testing equipment of a fuel-feeding system for a space rocket engine. During testing of a 42 MPa high-pressure hydrogen gas flow system, an exhaust gas duct outlet was damaged; hydrogen blew out and exploded. It seemed that the cause of this accident was deterioration of the exhaust gas duct outlet by thermal stress. High-pressure hydrogen was flowing during the test, and burned not in the burner (a gas generator), but in a silencer tower after flowing through the exhaust gas duct. This explosion was caused by the damage of that duct' [http://www.shippai.org/fkd/en/cfen/CC1200114.html]. The main reason for this accident is that nickel alloy of the exhaust gas duct welding became brittle after 132 tests of high-pressure and high-temperature hydrogen gas combustion over 5 years.

Mitigation measures for HE and hydrogen attack are:

- Reduction of corrosion rate (use of inhibitors or surface coatings).

- Dry conditions during welding process.
- Use of a pure gas.
- Use of a clean steel (deoxidized).
- Selection of materials (addition of: vanadium (V) to ferritic steels; rare earth elements to ferritic steels; nickel, carbon and manganese to austenitic steels).
- Alloying with chromium, molybdenum, tungsten.
- Heat treatment (baking) to remove absorbed hydrogen.
- Minimization of residual stresses [5].

4. Interaction of hydrogen with polymeric materials

As it was mentioned earlier, the polymeric materials are increasingly being used for the liners and wrapping of hydrogen storage vessels. For the wrapping of composite tanks (Type III and IV) glass, aramide or carbon fibres can be used [3]. These fibres are characterized by their tensile modulus, tensile strength and elongation [3]. Polymers are also present in some fuel cells as a material for membranes. Please read about an incident occurred on a PEM FC [7]. Two phenomena often associated with polymeric materials used in FCH applications: a *permeation* of hydrogen through the materials and the *degradation of the mechanical properties* of the polymers. From material point of view, hydrogen storage represents quite a challenge. The materials used for hydrogen storage must be light in weight but also should be able to withstand extremely high pressures whilst maintaining their integrity. There are several unwanted effects of hydrogen on polymeric materials:

(1) A *swelling* of polymers, which occurs due to a gas (or a liquid) ab-/adsorption.

This can lead to an unacceptable increase of components' dimensions (especially for O-rings) or to a formation of cracks related to a sudden outgassing when the partial pressure is decreased [8]. The significant level of swelling can be obscured by 'leaching out' of plasticisers and fillers frequently used in polymeric materials. Other important effects such as changes in a polymer mechanical strength and hardness should also be considered. According to standards a swelling of more than 15% in normal service conditions is marked as 'NR' (not recommended); a swelling less than this is marked as 'A' (acceptable) provided other risks are acceptable. Hydrogen gas under pressure may lead to swelling of several polymers used in liners of the tanks.

(2) A *blistering* of polymers, which occurs when large amount of hydrogen dissolution.

Relatively large amounts of hydrogen can dissolve in a polymeric material, thus a polymer exposure to hydrogen can cause not only swelling but also a *blistering*, similar to the one shown on Figure 2.

(3) A *deterioration* of polymers due to hydrogen *impurities*.

If gaseous hydrogen contains a certain type of *impurities*, which are not compatible with the polymeric materials, this may lead to a deterioration of mechanical properties and eventually to a rupture of a component. This risk should be considered depending on the hydrogen source.

(4) *Risk of fire*.

Hydrogen is a flammable gas and in case of a fire polymeric materials ignite relatively easy. As a result of that materials degrade and mechanical strength significantly reduces and this eventually may lead to a rupture. Type III and IV tanks cannot withstand fire for longer than 6 - 12 minutes (typically). That is why tanks are usually fitted with PRDs capable of venting the hydrogen within this time, thus resulting in long jet fires.

(5) *Hydrogen permeation*.

Barthelemy [3] stated that permeation of hydrogen through the polymeric liner is one of the main issues for the hydrogen high pressure vessels along with the HE.

In general,

- the more the polymer density increases, the more the permeation decreases,
- the more the polymer crystallinity increases, the more the permeation decreases.

Permeation is discussed in the following section of this lecture.

5. Limitation of hydrogen permeation

Permeation is an inherent phenomenon for all gases, which are in contact with polymers, and is the result of the hydrogen gas dissolution and diffusion in the polymer matrix. Due to a small size of its molecules hydrogen diffusion and thus the permeation are enhanced [3].

According to SAE J2578 (2009), permeation for CGH_2 systems could be defined as a diffusion of gas through the walls or interstices of a container vessel, piping or interface material [9]. It is worth noting that hydrogen in atomic form permeates metals, whilst for polymers permeation occurs in molecular form [10]. Current Type IV storage containers use a polymer liner, for example made of high-density polyethylene, typically overwrapped with carbon fibres set in a resin matrix. Other fibres such as glass or aramid may also be used, but most automotive systems use carbon fibre. The wrapping around the container varies in thickness, depending on the stress distribution. Type III or Type IV containers are used for most automotive applications.

The *permeation rate* through a particular material is described by Eq. (1), where J, is permeation rate (mol m^{-2} s^{-1}), T is material temperature (K), p_r is reservoir pressure (MPa), l is the reservoir wall thickness (m), P_0 is pre-exponential factor or permeability coefficient (mol m^{-1} $MPa^{-1/2}$ s^{-1}) and E_0 –is activation energy (J mol^{-1}):

$$J = P_0 \exp(-E_0 / RT)\frac{\sqrt{p_r}}{l} \quad (1)$$

Pre-exponential factor and activation energy both depend on the nature of material. The higher the storage pressure the higher is the permeation rate. The permeation rate also increases when the wall thickness is reduced. Three main phenomena will affect the dispersion of permeated hydrogen: buoyancy, diffusion, and ventilation.

For metallic containers (i.e. Type I and Type II) or containers with metallic liners (i.e. Type III), the permeation rate is considered to be negligible. However, hydrogen permeation is an issue for containers with polymeric liners (i.e. Type IV containers). For example, at 293 K, aluminium has a low permeability 2.84×10^{-27} mol m^{-1} $MPa^{-1/2}$ s^{-1}, while a polymer like Noryl has a permeability of 5.55×10^{-15} mol m^{-1} $MPa^{-1/2}$ s^{-1}, i.e. 12 orders of magnitude larger [10].

The economic and technological viability of on-board hydrogen tanks depends amongst other things on their gravimetric and volumetric capacity as mentioned earlier in this lecture. The gravimetric capacity is currently improved using high pressures for cGH2 storage, which dictates choice of light-weight, non-metallic Type III and Type IV tanks. However, as it was just described, Type IV tanks are characterised by a strong permeability.

Permeation may be categorised as a long-term slow hydrogen release from a CGH_2 system. Controlling the permeation has long been recognised as a key enabler in the development of hydrogen storage technologies [11]. The permeation from on-board hydrogen tanks is a safety issue for enclosures as hydrogen can accumulate over a period of time to create a flammable mixture with air. In sealed enclosures without ventilation the LFL of 4 vol. % of hydrogen in air can be reached as a result of permeation over quite a long time. In order to estimate the time for reaching LFL locally, e.g. under the garage ceiling, or homogeneously throughout the garage, it is important to know how hydrogen will be distributed in the enclosure.

The formation of a flammable mixture is a major safety concern for indoor use of FCH systems. There are two safety questions: *How safe is the permeation of hydrogen through a polymeric liner used in an on-board storage and its dispersion in a garage*? *Is natural ventilation sufficient to eliminate potential hazard of flammable hydrogen-air mixture formation?* The distribution of permeated hydrogen within an enclosure such as the garage, when LFL of hydrogen in air (4 vol. %) is reached, is important to define the *maximum allowable permeation rate* for FC vehicles equipped with cGH_2 tanks on-board [10].

Let us consider the dispersion of permeated hydrogen from a storage tank in a typical garage with adiabatic walls and still air. The following parameters were considered: storage tank size: L = 0.672 m, D = 0.505 m, hemisphere at each end (V = 0.2 m^3); permeation rate J equal to 1.14 NmL/hr/L of tank volume (below the allowable by the European Law [12] permeation rate limit of 6 NmL/hr/L (at 20 ºC); typical garage size $L \times W \times H = 5 \times 3 \times 2.2$ m (volume V = 33 m^3); still air; floor clearance of 0.5 m; temperature of 298 K. Analytical solution demonstrated that the time to reach LFL of 4 vol. % in the closed garage with chosen tank and permeation rate will be 240 days. On the other hand, time for hydrogen diffusion through the

height of the garage is 62051 s or 0.7 day, which means that permeated hydrogen will make a uniform mixture across the considered garage height.

A CFD study with the view to investigate the uniformity of permeated hydrogen dispersion in a garage-like enclosure was also carried out [13]. Numerical simulations were performed based on an original approach of a hydrogen mass source term introduction in the hydrogen conservation equation in control volumes around the tank surface. The permeation leak was simulated as a volumetric release of hydrogen in a thin layer of control volumes around the tanks surface of 1.87 m^2. CFD simulations indicated a negligible stratification of hydrogen. It is shown that the maximum hydrogen concentration in an enclosure is always on the top surface of the tank and it never reaches 100 vol. %. Both the analytical analysis and numerical simulations have demonstrated that diffusion and buoyancy contributions to the hydrogen transport from the tank surface are balanced within 1 minute from the beginning of the process. The quasi-steady state conditions within the enclosure with approximately linear distribution of hydrogen from the top to the bottom are established in about 1 hour for the considered permeation rates. For example, the maximum concentration of hydrogen at time of 133 min on the tank top surface was 8.2×10^{-3} vol. % (Figure 3). The observed difference in hydrogen concentration between the ceiling and the floor is about 3×10^{-3} vol. % and this difference conserves with time. Both values are significantly lower that LFL of 4 vol. %.

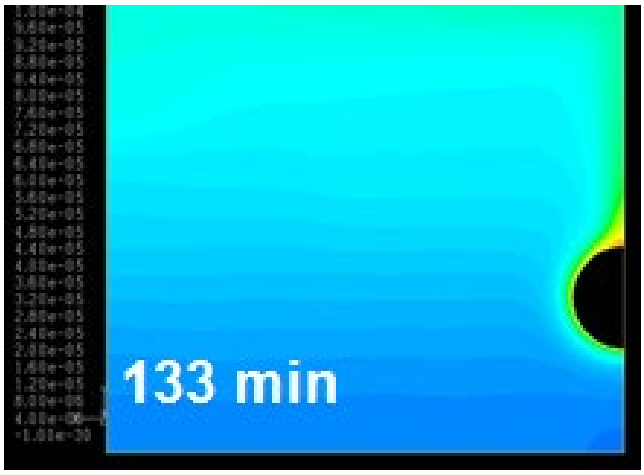


Figure 3. A snapshot of permeated hydrogen dispersion in a garage-like enclosure from Ref [13]

As a result of a permeation-induced leak, hydrogen releases in very small amounts, and spreads equally along the surface of a storage vessel. Due to a practically uniform dispersion of hydrogen permeated through the walls of the polymeric liner of the on-board storage tank on a FC vehicle parked in a garage, the perfect mixing of hydrogen and air is considered. Based on this the maximum allowable permeation rate can be calculated as [10, 14]:

$$Q_{perm}^{\max} = \frac{Q_a \cdot \mathrm{C}_{\%}}{100 - \mathrm{C}_{\%}} \cdot \frac{60 \cdot 10^6}{V \cdot f_a \cdot f_t}, \qquad (2)$$

where $C_{\%}$ - concentration of hydrogen in air, vol. %;

Q_a and Q_g - air flow and hydrogen gas leakage rate, respectively, m^3/min;

V – water capacity of hydrogen storage, L;

f_a – aging safety factor, taken to be 2, for unknown aging effects;

f_t – test temperature safety factor (3.5 for tests at temperature 20 ºC, and 4.7 for tests at 15 ºC).

The values of maximum allowable permeation rate for a passenger car and a city bus are calculated and presented in Table 1, taking into account the following:

- The permeation rate is specified in NmL/hr/L water capacity.
- Permeated hydrogen can be considered to disperse homogeneously.
- Worst-credible natural ventilation rate for a domestic garage is 0.03 air change per hour (ACH).
- Maximum permitted hydrogen concentration is 1 vol. %, i.e. ¼ of LFL.
- Maximum long term material temperature is 55 ºC.

Table 1. The calculated safe permeation rates of hydrogen

Minimum Testing Temperature (ºC)	**Maximum allowable permeation rate (mL/hr/L)**	
	Passenger car	City bus
15	6.0	3.7
20	8.0	5.0

With the levels of permeation rate indicated in Table 1 the hydrogen dispersion in typical garage would not represent a problem. These allowable permeation rates are regulated by the European Law [12]. Different limits on hydrogen permeation rate are accepted elsewhere. For example, please compare the following levels:

- Japan Automotive Research Institute: **5** NmL/hr/L (15 ºC).
- Society of Automotive Engineers J2579, end of life, 55ºC: **150** NmL/min/vehicle.
- ISO/TS15869: 2009 at end of life (20 ºC): **75** NmL/min/container.

6. A new standard for polymer hydrogen application compatibility

Today, there is a lack of test methods for evaluating polymer properties in hydrogen applications for determining design robustness. The polymer compatibility should occur on a material level. A new standard called “CHMC 2 – Test Methods for Evaluating Material Compatibility in Compressed Hydrogen Applications – Polymers” has been created and published (August 2019) by ANSI / CSA [15]. The results of these tests are intended to provide

a basic comparison of polymer materials performance in applications utilizing compressed hydrogen. A list of priority test is proposed. The first one is the hydrogen permeation where the issue is to show if the polymer is unable to contain hydrogen through the material. The second one is the physical stability to check if the polymer is unable to maintain dimensions (swelling or shrinking) and/or mass. The third test is a rapid cycling test where the issue is material degradation (extrusion, cracks or blisters) due to hydrogen exposure. Dedicated tests have been selected to follow polymer property changes to check in the material is unable to maintain mechanical properties for design and compression. On test is rheologic. A dynamic frictional wear is dedicated to follow if the polymer is unable to maintain interface sealing and design with mating surface. Finally, the last critical test is material contamination test where the issue is materials release constituents causing impurity of the hydrogen.

Acknowledgement

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