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# STUDY OF POTENTIAL LEAKAGE ON SEVERAL STRESSED FITTINGS FOR HYDROGEN PRESSURES UP TO 700 BAR

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## ABSTRACT

In order to improve risk analyses and influence the design of the future hydrogen systems, an experimental study on leaks qualification and quantification was performed. In H<sub>2</sub> energy applications, fittings appear as a significant leakage potential and subsequently explosion and flame hazards. Thus, as part of the “Horizon Hydrogène Energie” French program [1], four kinds of commercial fittings usually employed on H<sub>2</sub> systems were tested thanks to a new high pressure test bench – designed, set-up and operated by INERIS – allowing experiments to be conducted for H<sub>2</sub> pressures up to 700 bar. The fittings underwent defined stresses representative of H<sub>2</sub> systems lifetime and beyond. The associated leaks – when existing – are characterized in terms of flow rate.

## CONTEXT

Risks induced by H<sub>2</sub> use are first the leakage, and then explosion or fire due to ignition of a flammable mixture. Thus consequences having to be assessed for risk management of systems using hydrogen are the following:

1. In case of leakage, two possible scenarii depending on release properties:
  - indoor build-up: reached hydrogen concentration versus ATEX formation fixed at 4% H<sub>2</sub>,
  - self-ignition: jet fire characteristics.
2. If explosive atmosphere is formed:
  - reached levels of overpressure in case of ignition and explosion of flammable mixture versus distance from explosion ignition point, and associated consequences on:
    - structures (e.g. collapse),
    - and humans (e.g. overpressure effects, missiles).
  - jet fire follows.

The aim of the present study is to improve risk assessment of H<sub>2</sub> energy systems – existing (see Figure 1) or for future developments – by better knowing the leak causes and the associated flow rates.

Presently, assumptions are taken on the leak source – critical parameter for risk analyses of potentially hazardous systems – in order to assess a representative H<sub>2</sub> concentration.

The few data available in the literature concern hypotheses on leak diameter for build-up assessment of flammable gases, but they are generally not complete since they do not cover all the technical configurations that may exist (large variety of connection types, diameters of pipes and equipment, external stresses on equipment...). The IGC DOC 134/05 issued by EIGA (European Industrial Gases Association) provides a release source hypothesis of 0.1 mm leak diameter at a pressure of 200 bar [2] which is also mentioned in several references [3-4].

Experimental tests can bring important information and knowledge on this subject, which could be integrated in available modeling tools.



Figure 1. Examples of H2E hydrogen-based existing systems.

In view of the H2E applications, the fittings – by their function, their principle and their large number in the systems – appear as a primary source of leak. That is the reason why in this work several kinds of tube fittings and of stresses were considered to establish a realistic test matrix close to the real-life conditions of the hydrogen-based applications in order to determine associated flow rates.

This type of experiments was performed at moderate pressures ( $< 35$  bar) and mainly for 1/4'' pipe fittings [5] as normally used in mobile applications for moderate powers. It showed that these fittings have a very low leak rate in normal conditions ( $< 10^{-3} \text{ cm}^3 \cdot \text{s}^{-1}$ ), but could be an important source of release in specific cases (e.g. leak rate up to  $800 \text{ cm}^3 \cdot \text{s}^{-1}$  at 20 bar for hand tightening of the fitting).

Work in the present study aims at extending knowledge on this thematic to other possible applications considering not only other fittings (with a larger diameter range) but also higher pressures (up to 700 bar) and other fatigue conditions in particular when much stresses are applied (during mounting or H<sub>2</sub> based-system use for instance).

Expected outcomes of this study are a better definition of fittings resilience and of maintenance frequency to ensure an appropriate level of safety for hydrogen-based systems.

In the following sections, firstly the tests carried out on stressed fittings are described, then the characteristics of the test bench specifically designed are presented along with the operating conditions, and to conclude the results obtained in this work are discussed.

## 1.0 TESTED CONFIGURATIONS

### 1.1 Studied fittings

Test configurations were chosen based on H<sub>2</sub> energy existing systems and their conditions of use.

Four kinds of commercial fittings usually employed on H<sub>2</sub> systems, described in Table 1, were experimentally tested thanks to a specific H<sub>2</sub> high pressure test bench.

Table 1. Selected fittings for H<sub>2</sub> high pressure leakage experiments.

Fittings	External tube size	Maximal working pressure recommended by the constructor	Maximal tested pressure
Swagelok <sup>®</sup> double ring tube fitting (316 SS <sup>*</sup> )	6 mm	420 bar	700 bar
Rotarex <sup>®</sup> double ring tube fitting (316 SS)	6 mm	420 bar	700 bar
Swagelok <sup>®</sup> medium-pressure double ring tube fitting (316 SS)	1/2''	1035 bar	700 bar
Maximator <sup>®</sup> threaded ring tube fitting (316 SS)	9/16''	1500 bar	700 bar

<sup>\*</sup> Stainless Steel

In this work, note that the 6 mm double ring tube fittings (Swagelok<sup>®</sup> and Rotarex<sup>®</sup>), although recommended for a maximal working pressure of 420 bar, are tested up to 700 bar H<sub>2</sub> only in order to investigate their sealing behaviour for higher pressure conditions.

Figure 2 presents three of the four fittings which were tested; Swagelok<sup>®</sup> and Rotarex<sup>®</sup> (see Figure 2 (A)) 6 mm double ring tube fittings being very similar in terms of both design and sealing principle.

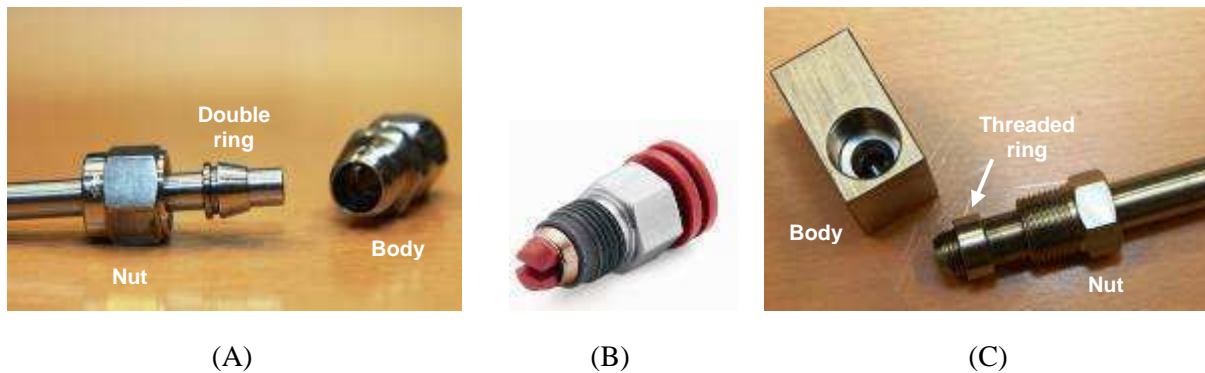


Figure 2. Pictures of three of the studied fittings.

(A) Rotarex<sup>®</sup> 6 mm double ring tube fitting, (B) Swagelok<sup>®</sup> 1/2'' medium-pressure double ring tube fitting (source: Swagelok<sup>®</sup>), (C) Maximator<sup>®</sup> 9/16'' threaded ring tube fitting.

## 1.2 Stress configurations

A reliability approach, through fault tree analyses, was used to determine all causes of leak for these kinds of fittings. The primary cause of leakage should be strongly linked to mounting and maintenance operations and most of the present work was devoted to these aspects.

For a few configurations, the stress had to be applied when the fitting was under pressure. This special situation is referred to as “dynamic stress”. In most cases, the fitting was conditioned (e.g. screwed, unscrewed several times) before the pressure was applied (“static stress”).

An analysis of the H<sub>2</sub> systems extreme conditions of use enabled to determine magnitude orders of probable maximal stresses on these fittings. Furthermore conditions for the fittings qualification tests of the constructors were considered. Based on this information, a test matrix was established and defined stresses were experimentally reproduced in order to quantify the level of leak they led to.

Note that maximal stress levels which were tested are significantly higher than the conditions encountered by the systems in use or than the conditions of qualification tests provided by the fittings constructors.

Table 2 presents in details scenarii which were retained for experimental tests. The normal conditions of use recommended by the constructors, and the maximal tested stress levels for each fitting and stress were reported hereafter.

“Dynamic stresses” were applied only on 6 mm tube fittings. Actually the maximal force available on the experimental test bench for these conditions of stress, although high, is not strong enough to have significant effects on the 1/2” and the 9/16” tube fittings.

Table 2. Selected stress scenarii.

	Stresses	Normal conditions of use	Tested stress level considered as maximal in situation
Static stresses	Assembly-dismantling cycles	Around 20 cycles, for 6 mm double ring tube fitting	100 cycles
		< 20 cycles, for high pressure fittings (double and threaded ring tube fittings)	50 cycles
	Under-tightening	1 1/4 turn, for 6 mm double ring tube fitting	1 turn
		1 1/4 turn, for 1/2” double ring tube fitting	3/4 turn and 1 turn
		80 N with silicone grease <sup>(1)</sup> on conical sealing surfaces, for 9/16” threaded ring tube fitting	60 N with silicone grease
	Over-tightening	1 1/4 turn, for 6 mm double ring tube fitting	1 1/2 turn
	Thermal effects	+60 to -20°C	Crimping at 100°C and leakage test at 30°C
Dynamic stresses	Counter-clockwise rotation	0 turn	1/8 turn compared to initial position
	Flexion	0°	10° from fitting initial axis
	Traction	0 N	320 N <sup>(2)</sup> in fitting axis direction

<sup>(1)</sup> recommended by Maximator® to improve the sealing

<sup>(2)</sup> representing a mass of 32 kg attached to the fitting

Figure 3 represents “dynamic stresses” applied by a pneumatic jack on the 6 mm double ring tube fittings under H<sub>2</sub> pressure (see “dynamic stress” module in Figure 6, Section 2.2).

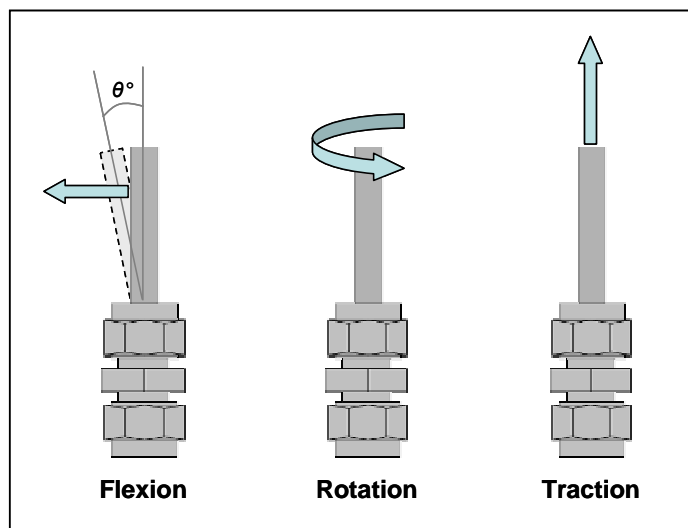


Figure 3. Scheme of “dynamic stresses” applied on 6 mm double ring tube fittings.

## 2.0 EXPERIMENTAL DEVICE

### 2.1 Main test bench

An experimental installation was specially designed and built up by INERIS enabling leakage testing under hydrogen up to 700 bar in safe conditions (see Figure 4).

Experimental test bench is mainly composed of:

- a H<sub>2</sub> compressor to reach the maximal pressure of 700 bar,
- a thermostatic 1 m<sup>3</sup> enclosure to ensure thermal stability during leakage tests,
- a primary high pressure reservoir of 2.4 L,
- a secondary reservoir of 30 L to increase the accuracy of leak flow rate determination,
- 3 Autoclave<sup>®</sup> pneumatic valves,
- 2 Kistler<sup>®</sup> pressure transducers (accuracy  $\pm 0.1\%$ ): 0-1000 bar on the 2.4 L reservoir, and 0-200 bar on the 30 L reservoir.

According to previous experiences, the temperature inside the testing chamber need to be very stable. In the 1 m<sup>3</sup> enclosure, the temperature is measured by three thermocouples (accuracy  $\pm 0.2^\circ\text{C}$ ) and regulated at 30°C; this parameter is of prime importance for gas leakage studies and determination of the associated flow rates.

Sensitivity of the test bench in terms of leak detection and quantification is assessed to be around 10<sup>-2</sup> cm<sup>3</sup>.s<sup>-1</sup>. This threshold sensitivity value is based on potential residual leakages of the test bench equipments (e.g. valves and other fittings correctly assembled...), on the uncertainties of each transducer and on a test duration of 5 h.

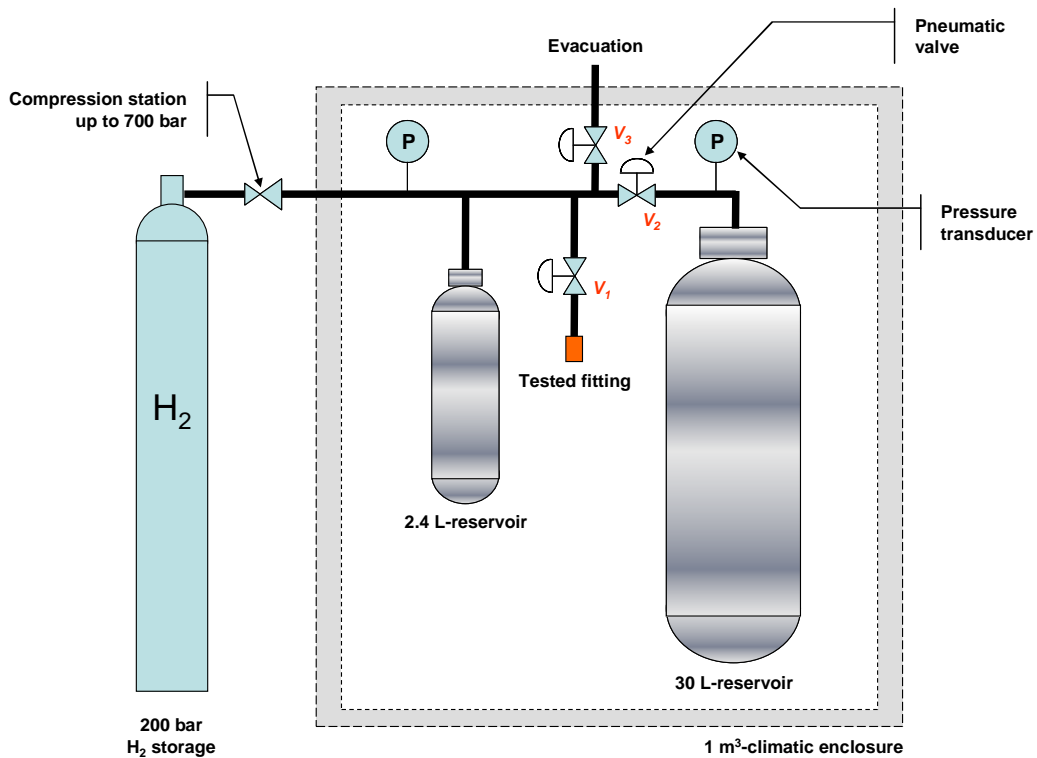
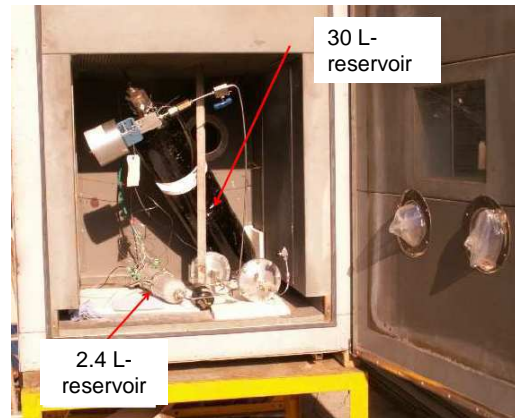


Figure 4. Scheme of the experimental installation for high pressure leak tests on stressed fittings.

For safety aspects – because tests are carried out with hydrogen and an explosive atmosphere could be generated in case of non negligible release – the enclosure is constantly flushed with nitrogen. Valves are pneumatic in order to limit ignition sources.



(A)



(B)

Figure 5. Pictures of the INERIS experimental installation. (A) climatic enclosure, (B) climatic enclosure with device for tests.

## 2.2 Additional module for “dynamic stress”

It can be recorded that in a number of situations, the fitting was conditioned before the tests (static stresses) but that in a few instances, the stress (traction, flexion, torsion) needed to be applied during the pressure test (dynamic testing). For this purpose, a specific additional module was designed to be

inserted in the climatic enclosure. This module comprises a pneumatic jack (which can produce a force up to 320 N) and a bracket to strongly maintain the fitting during the stress applied by the jack.

This module, according its settling, can be used for flexion, rotation or traction stresses (see Figure 6).

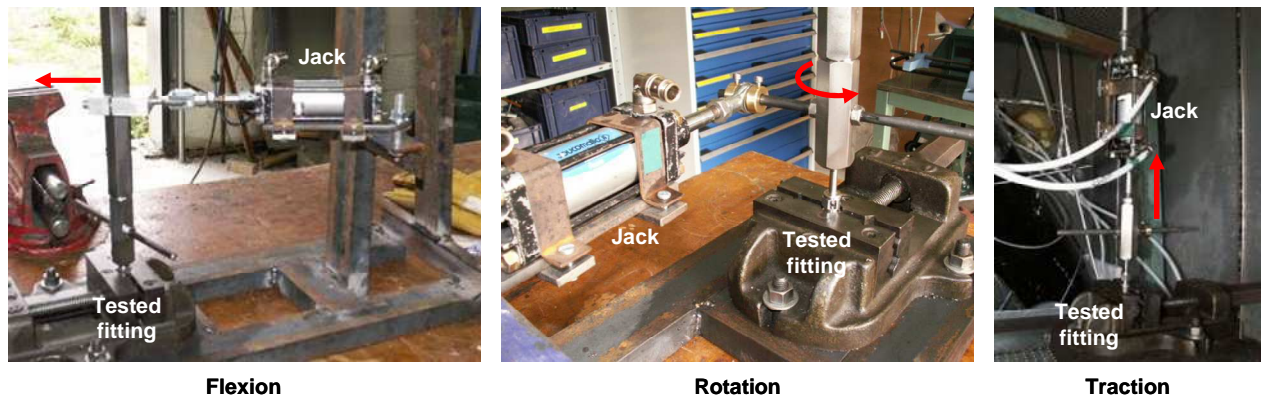


Figure 6. Pictures of the different configurations of the “dynamic stress” module.

### 2.3 Experimental procedure

The hydrogen (200 bar from a bundle of cylinders) is compressed up to 700 bar and the 2.4 L reservoir is filled. When temperature homogeneity is reached, the pneumatic valve,  $V_1$ , is opened for the leakage test on the studied fitting at the desired pressure (see Figure 4).

If the leakage is too high, the pneumatic valve  $V_2$  is opened to improve accuracy on flow rate determination by using the 30 L reservoir.

When the test is finished, the pneumatic valve  $V_3$  is opened to evacuate  $H_2$  from the gas lines of the test bench and from the reservoirs.

During the test, the pressure is controlled and recorded in order to detect leakage and assess associated flow rate.

The flow rate of the leakage is estimated by knowing the quantity of hydrogen lost during the time of the leakage. This quantity can be assessed measuring the variation of pressure in the high pressure reservoir.

### 3.0 RESULTS AND DISCUSSION

Several tests were performed as described in Section 1.0. Surprisingly, only two test configurations showed small leakages; the other stress scenarii did not exhibit detectable leakage given experimental installation sensitivity. Table 3 presents releasing configurations and associated leak flow rates experimentally determined which appear close to the threshold sensitivity value of  $10^{-2} \text{ cm}^3 \cdot \text{s}^{-1}$  previously noted in section 2.1.

These values are very low compared to the flow rates usually considered for risk analyses of potentially hazardous systems, and would be detected by normal control procedures particularly for these types of stresses. Moreover a re-tightening would certainly stop these leakages.

Note that for a predictable risk, the usual “0.1 mm - 200 bar” leak [2-4] would correspond to a release around  $900 \text{ cm}^3 \cdot \text{s}^{-1}$  and by analogy a “0.1 mm - 700 bar” would be around  $3500 \text{ cm}^3 \cdot \text{s}^{-1}$ .



Table 3. Experimental leakage flow rates determined for releasing configurations.

Fitting	External tube size	Tested stress	Pressure conditions	Measured leakage rate
Swagelok <sup>®</sup> medium-pressure double ring tube fitting	1/2''	After 50 assembly-dismantling cycles	700 bar	0.05 cm <sup>3</sup> .s <sup>-1</sup>
Maximator <sup>®</sup> threaded ring tube fitting	9/16''	Under-tightening with silicone grease: 60 N instead of 80 N	700 bar	4.3 cm <sup>3</sup> .s <sup>-1</sup>

Even at very high pressures, the fittings under considerations rarely leak. Apart from the very specific situations where the fitting is not tightened (only by hand) or when the seals are damaged as reported in literature [5], some low leaks were found after 50 of assembly/dismantling cycles for the 1/2'' Swagelok<sup>®</sup>. An additional test showed that the leakage detected at 700 bar on the Swagelok<sup>®</sup> medium-pressure double ring tube fitting disappears when pressure is decreased down to 450 bar. Note also that under similar stresses, smaller Swagelok<sup>®</sup> fittings did not leak suggesting a scale effect.

For the Maximator<sup>®</sup> fittings at tested tightening couples, sealing is not easily reached if silicone grease is not applied on sealing contact surfaces. Higher tightening couples should be tested in order to check if, beyond 80 N and without grease, the sealing for this kind of fittings can be obtained.

Note also that the 6 mm double ring tube fittings are very resilient against dynamic stresses. To some extent, this means that the “pre-stress” applied on the fittings during tightening is well above the superimposed dynamic stresses.

From this work it is interesting to retain that:

- Despite a high pressure level for the tests (700 bar), very few scenarii gave rise to leaks;
- Even if the constructor recommended limit of working pressure for 6 mm double ring tube fittings (Swagelok<sup>®</sup> and Rotarex<sup>®</sup>) is 420 bar, tests performed at 700 bar did not show detectable leak, whatever the stress;
- When existing, determined leak flow rates are very low compared to flow rates considered in risk assessment of potentially hazardous systems and associated equivalent leak diameters would be very small compared to the usual “0.1 mm”.

A follow-up to this work could be to determine critical stress levels giving rise to significant and quantifiable leaks or test combinations of stresses.

## CONCLUSIONS

A specific experimental installation was designed and set-up by INERIS in order to study potential leakages on fittings commonly used in H<sub>2</sub> existing systems. This test bench enables:

- to accurately quantify, when existing, leakages with a flow rate above 10<sup>-2</sup> cm<sup>3</sup>.s<sup>-1</sup>,
- to carry out the tests with hydrogen in safe conditions,
- to reach test pressures up to 700 bar,
- to perform stresses on fittings under H<sub>2</sub> pressure (i.e. “dynamic stresses”).

Four kinds of fittings – double ring tube fittings of different trade marks or sizes, and threaded ring tube fitting – were tested in different stress configurations representing forces or damages due to lifetime of the system, maintenance actions, non conforming assembly or use etc.

Surprisingly, but reassuringly, very few scenarii gave rise to quantifiable leaks; because nonexistent or under test bench detectable thresholds.

Only two stress configurations with the highest fitting sizes showed measurable but low leak flow rates compared to values usually employed for risk assessment:

- $0.05 \text{ cm}^3 \cdot \text{s}^{-1}$  for the Swagelok® 1/2'' medium-pressure double ring tube fitting after 50 cycles of assembly-dismantling,
- and  $4.3 \text{ cm}^3 \cdot \text{s}^{-1}$  for a 25% under-tightening of the Maximator® 9/16'' threaded ring tube fitting.

The potential hazard arising from these leaks depends on the ability of the confinement surrounding the fitting to accumulate or disperse any flammable hydrogen-air mixtures. For considered hydrogen systems in H2E project, the present leakage rates are much too low to produce locally a slightly flammable atmosphere; a leakage rate of a few tens of  $\text{cm}^3 \cdot \text{s}^{-1}$  would be required. A different conclusion may be given for other types of confinement.

To complete this work, some tests could be performed to determine critical stress levels giving rise to significant leaks, or other kinds of stresses – like vibrations for example or some combinations of stresses – could be applied on fittings in order to assess their effects.

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