D2.2

Review of standards and literature review on testing and qualification of metallic materials compatibility in hydrogen gas

Emilie Buennagel*	RINA-CSM
Luigi Di Vito	RINA-CSM
Cristina Rodriguez	Redexis
Alberto Cerezo	Redexis
Vanessa Gil	Fundación para el Desarrollo de las Nuevas Tecnologías del Hidrógeno en Aragón (FHa) / Fundación Agencia Aragonesa para la Investigación y el Desarrollo (ARAID)
Lidia Martínez	Fundación para el Desarrollo de las Nuevas Tecnologías del Hidrógeno en Aragón (FHa)
Matías Suárez	Fundación para el Desarrollo de las Nuevas Tecnologías del Hidrógeno en Aragón (FHa)
Marcos Parra	SIDSA
Juan Carlos Martínez	SIDSA
Virginia Madina Arrese	TECNALIA
Nicolas Oscar Larrosa Fandino	TECNALIA
Maxime BERTIN	GTRgaz

***Corresponding author**

TECHNICAL REFERENCES

Project Acronym	CANDHy
Project Title	COMPATIBILITY ASSESSMENT OF NON-STEEL METALLIC DISTRIBUTION GAS GRID MATERIALS WITH HYDROGEN
Type	Report
Call Identifier	HORIZON-JTI-CLEANH2-2022-02-01
Topic	Compatibility of Distribution non-steel metallic gas grid materials with hydrogen
Project Coordinator	Fundación para el Desarrollo de las Nuevas Tecnologías del Hidrógeno en Aragón (FHa)
Project Duration	36 months

Deliverable No.	D 2.2
Dissemination Level	PU-Public
Work Package	WP2 - Review of the state of the art of the grid material and existing standards
Task	T 2.2 – Review of standards and codes on testing and qualification for materials compatibility with hydrogen gas. T.2.3 – Collecting information from past and ongoing projects and research literature on the testing and evaluation of metallic materials compatibility in hydrogen gas.
Lead beneficiary	7 (RINA-CSM)
Contributing beneficiary(ies)	1 (FHa), 2 (GRTGAZ), 3 (Tecnalia), 4 (Redexis Gas), 5 (Redexis Serv), 9 (Sidsa)
Due date of deliverable	31/08/24
Actual submission date	DD/MM/YY

VERSIONS

Revision Version	Date	Changes	Changes made by Partner
0.1	02 August 2024	First release	E. Soileux (RINA-CSM)
0.2	21 August 2024	Input required	L. Martínez, M. Suárez and V. Gil (FHA)
0.3	30 August 2024	Input required	V. Madina Arrese and N. O. Larrosa Fandino (TECNALIA)
0.4	06 September 2024	Input required	M. Bertin (GRTgas)

Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

Table of Content

EXECUTIVE SUMMARY

The intention of the European gas industry is to enable the use of natural gas infrastructure for hydrogen. However, for the use of hydrogen in the natural gas infrastructure the consequences or the impact of hydrogen on the gas system need to be identified. Carbon steel is the alloy family of metallic materials most used in hydrogen gas distribution pipelines and is suitability in hydrogen gas is well studied and reported. On the contrary, studies on non-steel metallic materials also present in the current gas grid distribution networks are limited and the effect of hydrogen embrittlement (HE) on these materials needs to be comprehensible understood.

As gas distribution networks operate at low pressure, < 16 bar, these networks are less standardized than transport networks, CANDHy project focuses on the compatibility assessment of non-steel metallic distribution gas grid materials with hydrogen in European countries.

As part of Task 2.2 and 2.3 of CANDHy Work Package 2 (WP2) a full review of the state of the art of the standards, codes but also a literature review on testing of non-steel metallic material in hydrogen environment was performed.

Current standards cover steel materials and there is a need to provide additional information for non-steel metallic materials and components of the distribution grid. Screening tests are the most common tests to evaluate the behaviour of materials in hydrogen and mainly tensile and SSR tests were performed on non-steel metallic materials. Therefore, fracture and fatigue tests providing data on crack initiation and growth quasi-static or cyclic loading would be necessary to completely understand the effect of hydrogen gas.

Following this work, fracture tests such as K_{IH} test, fracture toughness, SSR and C-ring and fatigue test such FCGR should be conducted on materials such as ductile and gray cast iron, copper, aluminium, brass and lead. Due to the small material thicknesses of the non-steel pipes used in the distribution gas grid, testing like K_{IH} test, fracture toughness and FCGR might not be possible because bigger specimens are needed to obtain valid results; therefore, C-ring and SSR on notched specimens could be performed to study the material behavior and the sensitivity to hydrogen in presence of a notch in H_2.

1. INTRODUCTION AND OBJECTIVES

The injection of hydrogen in the natural gas grid, either as hydrogen/natural gas (H_2/NG) mixtures or 100% H_2, raises concerns about safety, infrastructure integrity, gas metering and quality. The issue of pipe integrity on non-steel metallic materials is exacerbated in existing natural gas grids, even at the low pressure of distribution grids, as hydrogen has a well-known detrimental effect on the mechanical properties of metals, causing embrittlement. Hydrogen embrittlement of materials may manifest itself as a loss in strength or ductility or both, a loss of toughness and fatigue with accelerated fatigue crack growth rates, and a reduction in fracture toughness. The degree of embrittlement strongly depends on materials, hydrogen partial pressure, hydrogen purity and environment temperature. Therefore, it is crucial to conduct long-term material integrity assessments that replicate the operating conditions of distribution grids using testing platforms.

This report detailed the work performed for CANDHy Work Package 2 Task 2.2 and Task 2.3. RINA-CSM was the leader for both Tasks.

WP2 Task 2.2 involved the following partners: FHa, Sidsa and Tecnalia. Furthermore, communication was conducted with the relevant Technical Committees in the standardization of materials for hydrogen service and stakeholders and associations present in the external advisory board (EAB). The main objective of WP2 Task 2.2 was to perform a state-of-the-art review of the standards and codes on testing and qualification for metallic materials compatibility with hydrogen gas. First, general standards, codes and guidelines on material requirements related to the delivery of 100% hydrogen or hydrogen/natural gas mixtures in low-pressure distribution grids were collected and reviewed. Secondly, testing methods and characterization techniques proposed in the standards and relevant to determine the H_2 suitability were analysed. The review concentrated on the applicability and complexity of the existing procedures for non-steel metallic materials and components in the distribution gas grid. Limitations and gaps on key properties and factors for the validation and qualification of materials or components were identified. The hydrogen readiness including EU infrastructure and assessment of defects were analysed. Area of future needs as well as the need for new standards essential to the future gas distribution systems was evaluated.

WP2 Task 2.3 involved the following partners: Redexis, FHa, Sidsa and Tecnalia. The main objective was to collect information from previous and ongoing projects/activities and research literature/documents on the testing and evaluation of metallic materials compatibility in hydrogen gas. The focus was on testing of non-steel metallic materials to identify the procedures applied and materials previously tested to not overlapping with other ongoing activities. Literature research on testing and evaluation of metallic materials compatibility in hydrogen gas was also carried out in order to identify gaps, missing information and established non-steel metallic materials whose behaviour in hydrogen service is unknown. The applicability, availability and complexity of the existing tests and qualification procedures as well as the need for new standards and component characterization techniques were evaluated.

For both Tasks, all documentation was listed and shared with all partners.

2. REVIEW OF STANDARDS AND GUIDELINES

In this section of the report, the current most relevant standards and guidelines regarding testing and qualification for materials compatibility with hydrogen gas service were collected and analysed. The review was focused on standards directly or indirectly applied for the delivery of 100% H_2 or H_2/NG mixtures in low-pressure distribution grids (< 16 MPa) and centered on non-steel metallic materials.

The analysis was carried to:

- ✓ Assess the applicability and complexity of the existing testing procedures for materials and components of the distribution gas grid.
- ✓ Identify gaps for the validation and qualification of materials and components.
- ✓ Analyze the test methods and characterization techniques and determine the areas of future needs.
- ✓ Assess the hydrogen readiness for non-steel metallic materials.
- ✓ Assess defect size allowed for existing component of the network.

2.1 Non-metallic materials of the gas distribution grid

In WP2 Task 2.1 the materials of the natural gas distribution grid network of EU countries including Ukraine and the UK were analyzed [1].

The non-steel metallic materials present together with their main composition are reported in Table 1.

Table 1 Materials and their main composition

Material	Main Composition
Ductile cast iron	Carbon: 3.0-4.0%, Silicon: 1.8-3.0%, Manganese: 0.1-1.0%, Sulfur: ≤0.03%, Phosphorus: ≤0.1%, Iron: Balance
Gray cast iron	Carbon: 2.5-4.0%, Silicon: 1.0-3.0%, Manganese: 0.2-1.0%, Sulfur: 0.02-0.25%, Phosphorus: 0.05-1.0%, Iron: Balance
Copper	Copper: ≥99.9%, Oxygen: 0.02-0.04%
Brass	Copper: 55-90%, Zinc: 10-45%, Lead: ≤3.5%
Aluminium	Aluminium: 90-99.7%, Silicon: ≤1%, Iron: ≤0.8%, Copper: ≤0.6%, Manganese: ≤0.8%, Magnesium: ≤5%
Lead	Lead: ≥99.9%,
Zamak	Zinc: 94-96%, Aluminium: 3.5-4.5%, Copper: 0.25-1.0%, Magnesium: 0.03-0.06%
Bronze	Copper: 60-95%, Tin: 1-12%, Zinc: 0-10%

Pipe materials were found to be made of cast iron and copper while components (such as valves, connector, regulator, flow meters and filter) were made of all materials reported above.

2.2 Current standards and guidelines

The following standards and guidelines regarding testing and qualification for materials compatibility with hydrogen gas service were reviewed:

- ✓ ASME B31.12 "Hydrogen Piping and Pipelines" [2]
- ✓ ASME VIII Div. 3 "Rules for construction of pressure vessels", in particular Article KD10 "Special requirements for vessels in hydrogen service" [3]
- ✓ ISO 11114-4 "Transportable gas cylinder – Compatibility of cylinder and valves materials with gas content" Part 4 "Test methods for selecting steels resistant to hydrogen embrittlement" [4]
- ✓ ANSI/CSA CHMC 1 "Test methods for evaluating materials compatibility in compressed hydrogen applications – Metals" [5]
- ✓ EIGA IGC Doc 121/14 "Hydrogen Pipeline Systems" [6]
- ✓ EPRG "Hydrogen Pipelines Integrity management and repurposing Guideline White Paper" [7]
- ✓ IGEM/TD/1 "Steel pipelines for high pressure gas transmission" [8]
- ✓ IGEM/H/1 "Reference Standard for low pressure hydrogen utilization" [9]
- ✓ CEN/TR 17797 "Gas infrastructure – Consequence of hydrogen in the gas infrastructure and identification of related standardization need in the scope of CEN/TC234" [10]
- ✓ UNI 9860 "Gas infrastructure - Pipelines with maximum operating pressure no greater than 0.5 MPa (5 bar) - Gas user derivation systems - Design, construction, testing, operation, maintenance and rehabilitation" [11]

2.2.1 ASME B31.12

This standard contains requirements for piping in gaseous and liquid hydrogen service and pipelines in gaseous hydrogen service. It covers transmission and distribution pipelines conveying hydrogen gas, including natural gas/hydrogen mixtures (H_2/NG), by volume up to 100% from a production facility to the point of final use. It includes a wide range of system components such as pipes, fittings, valves, pressure vessels and associated equipment. This standard is applicable for gaseous hydrogen up to 21 MPa (210 bar) to steel materials used for pipeline and piping systems but also to copper, nickel, and aluminum materials used in piping and pipe components.

ASME B31.12 [2] states that steel and alloy steel, copper and copper alloy and aluminum and aluminum alloy materials are acceptable for hydrogen gas; however, nickel and nickel alloys should be avoided because they are highly susceptible to H_2 and cast irons and titanium-based alloy materials are not acceptable for hydrogen gas. Aluminum alloys should be used judiciously for hydrogen gas service if water vapor is present; however, all available data indicate that the susceptibility of aluminum to hydrogen embrittlement is very low in dry hydrogen gas. According to Table IX-1A of B31.12, the specified minimum yield and tensile strength for aluminum material shall be between 17-241 MPa and 58-317 MPa, respectively. Regarding copper, oxygen-free grades of copper should be used for hydrogen gas service as copper can be embrittled due to a reaction between dissolved hydrogen and oxygen to form water, resulting in pores that promote failure. The specified minimum yield and tensile strength for copper material should be between 62-413 MPa and 206-758 MPa, respectively. For nickel materials, the specified minimum yield strength should be between 172-379 MPa and the minimum tensile strength should be between 482-586 MPa.

To evaluate the suitability of materials in hydrogen environment, ASME B31.12 uses fracture mechanics methods to provide for the measurements of both, the threshold stress intensity factor, K_{IH} and of the fatigue resistance of the material through the study of the curve da/dN-ΔK using the applicable rules provided in Article KD-10 of ASME VIII Div. 3 (see details in Section 2.3 below).

This standard is the most detailed design codes which reference hydrogen; however, it is challenging to apply practically to repurposing pipelines, particularly accounting for historic defect and damage and does not incorporate the latest knowledge with specific application to Europe.

2.2.2 ASME VIII Div.3 – Article KD 10

The ASME VIII Div. 3 standard [3] provides for the measurement of a value of the K_{IH} parameter of fracture toughness, and of the fatigue resistance of a material through the study of the curve da/dN-ΔK, using fracture mechanics methods as described in the standard. This standard highlights the need to determine the parameters of interest for each material, each different welding procedure and in the following locations: the base material (BM), the weld metal (WM) and the heat-affected zone (HAZ) of welded joints. This code applies to metallic materials such as steel and steel alloys, nickel and nickel alloys, and aluminum alloys. No information on other non-steel metallic materials such as cast iron and copper materials is available. According to Table KM-400 of ASME VIII Div. 3 [3], the specified minimum yield and tensile strength for aluminum material shall be 170 MPa and between 260-290 MPa, respectively. For nickel materials, the specified minimum yield strength should be between 170-415 MPa and the minimum tensile strength should be between 450-825 MPa.

This code provides mandatory requirements for:

- Nonwelded vessels operating at temperatures less than 95 ºC with hydrogen partial pressure exceeding 41 MPa or 5.2 MPa for materials with ultimate tensile strength exceeding 945 MPa.
- Welded vessels operating at temperatures less than 95 ºC with hydrogen partial pressure exceeding 17 MPa or 5.2 MPa for materials with ultimate tensile strength exceeding 620 MPa

Limitations:

- For aluminum alloys, the maximum design temperature shall not exceed 107 ºC.
- For all other materials, the maximum design temperature shall not exceed 205 ºC.
- Vessel parts in direct contact with hydrogen shall have an ultimate tensile strength not exceeding 950 MPa.

✓ K_{IH} test method

The K_{IH} test method is reported in section KD-1040 of ASME VII Div. 3 [3]. Testing shall be performed using applicable rules of ASTM E1681 [12] (see Section 2.3) and the additional rules specified in this code.

The K_{IH} measurement must be conducted on at least three specimens per type following the relevant indications of the standard ASTM E1681 [12] which proposed three types of specimen configurations (beam specimen, compact specimen and bolt-load specimen). Specimen configurations with dimensions is reported in Section 2.3 of this report. The specimen thickness shall not be less than 85% of the design thickness of the material that is being qualified. Smaller specimens may be used provided that the specimen validity check of ASTM E1681, para. 9.3 is satisfied. Specimen shall be notched and fatigue pre-cracked and then loaded at a constant load or a constant displacement at a value of K_{IAPP} established by the User based on fracture mechanics calculations. The standard provides a table of possible K_{IAPP} values that can be adopted for ferritic steels only. The test is performed in a high-pressure test chamber containing hydrogen gas to a pressure equal to or greater than the design pressure of the vessel at room temperature. For ferritic steels and martensitic stainless steels, the duration of the test is at least for 1000 hours at room temperature, and at least for 5000 hours (208 days) for austenitic steels. No value of K_{IAPP} and test duration is given for non-steel metallic materials.

At the end of the test, any propagation of the fatigue crack is measured and if the average value does not exceed 0.25mm and:

- if the test was conducted at constant load, $K_{IH} = K_{IAPP}$;
- if the test was conducted at constant displacement, $K_{IH} = 1/2\ K_{IAPP}$.

If the average crack growth exceeds 0.25 mm, K_{IH} is calculated as required by ASTM E 1681 in par. 9.2.1 and 9.2.2 [12]. A value of K_{IH} is therefore obtained in each case.

✓ Fatigue crack growth rate (FCGR) test method

The ASME standard provides FCGR test in the form da/dn-ΔK in gaseous hydrogen at design pressure with a frequency test lower than 0.1 Hz. Testing shall be performed according to standard ASTM E 647 [13] (See Section 1.3) on at least three notched specimens for each of the following locations: BM, WM and HAZ.

2.2.3 ISO 11114-4

The standard ISO 11114-4 [4] specifies test methods and the evaluation of results from these tests in order to qualify steel suitable for use in the manufacture of gas cylinders for hydrogen and hydrogen bearing embrittling gases.

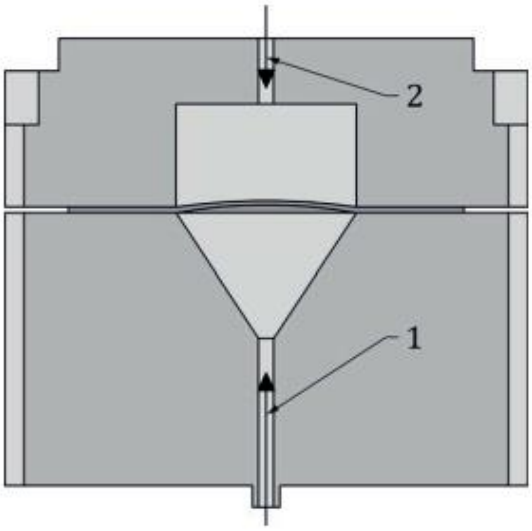
It only applies to seamless steel gas cylinders and is applicable if the hydrogen working pressure is more than 20% of the test pressure of the cylinder and the hydrogen partial pressure is more than 5MPa (50 bar). This standard is more generic in its selection of compatible materials and does not state non-steel metallic materials.

The standard recognized the following three test methods:

- ✓ Method A Disc test

Method A in Fig.1, also standardized by the standard ASTM F1459 [14], measures and compares the bursting pressures of a material in an inert atmosphere and in hydrogen. The gases used in the test require a high level of purity (O_2< 1 mL/L and H_2O < 3 mL/L). ISO 11114-4 requires a series of pressurization tests to be conducted in helium and hydrogen at different pressure growth rates, to identify the highest load condition sensitivity. It is therefore possible to obtain an evaluation of the susceptibility to the various pressurization speeds of the chamber and identify the $Pr'He/Pr'H_2$ ratio between the membrane rupture pressure in helium vs that in hydrogen. For the material to be compatible with hydrogen, this ratio must not exceed 2.

The test is conducted on a disk of a diameter 58mm with a thickness of 0.75 mm, which is not very representative of the real dimensions of the components that are generally intended to be analysed, at rate between 0,1 bar/min and 1 000 bar/min. The specimen shall be free of defects and the test does not provide an engineering parameter that can be used in design but only an embrittlement coefficient whose critical value (=2) is defined on empirical bases.



Key

1 disc is subjected to P_{H2} on the lower side

2 disc is subjected to P_{N2} on the upper side

NOTE The disc is loaded with $\Delta P = P_{H2} - P_{N2}$.

Figure 1. Principle of pressure disc test

- ✓ <u>Method B: Fracture mechanic test for determining the threshold stress intensity factor, K_{IH}, for susceptibility to cracking of metallic materials in gaseous hydrogen</u>

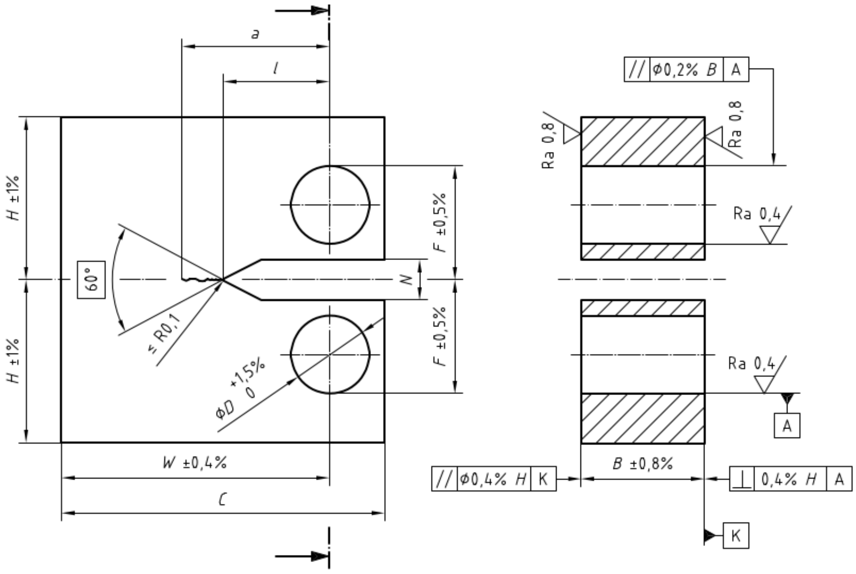
Method B is based on fracture mechanics and uses pre-cracked compact tension (CT) specimens according to the geometry given in ISO 7539-6 [15], see Figure 2. This method requires that the mechanical characterization tests be conducted in an autoclave at hydrogen pressure equal to or higher than the operating pressure, in conditions of gas purity similar to those described for method A. The specimens must have a thickness not less than 85 % of the component thickness being qualified, and at least three specimens shall be tested. Specimens shall be notched and pre-cracked prior to testing.

The test is performed using an increasing load in steps of amplitude equal to 1 MPa.m$^{1/2}$ at a constant rate of 2 x 10^{-3} kN s^{-1} until failure. The K_I value at which the crack begins to propagate is indicated as K_{IH}, which is determined as:

$$K_{IH} = YP/BW^{0.5} \qquad \text{(Equation 1)}$$

where Y is the stress intensity factor coefficient for a particular specimen geometry, P is the load applied to the specimen before final increment that caused failure, B is the specimen thickness and W the width.

The material is acceptable if K_{IH} is equal to or greater than the limit set by the standard, equal to 60/950 x Rm (MPa.m$^{1/2}$) where Rm is the average maximum tensile strength measured on two tensile specimens of the material under examination. In this case the K_{IH} parameter is of engineering relevance and allows the subsequent design of a pressure vessel.



Key

net width = W

total width, C = 1,25 W minimum

thickness, B = 0,5 W

half height, H = 0,6 W

hole diameter, D = 0,25 W

half distance between hole outer edges, F = 1,6 D

notch width, N = 0,065 W maximum

effective notch length, l = 0,25 W to 0,40 W

effective crack length, a = 0,45 W to 0,55 W

Figure 2 Configuration of compact tension specimen

- ✓ <u>Method C: For determining the resistance of steel materials to hydrogen assisted cracking</u>

Method C is simpler to apply and involves the imposition of a constant load or constant displacement on specimens with CT geometry. The thickness, orientation, number and pre-cracking methods of the specimens are the same as those of method B but here the specimens are static-loaded such that K_I value applied at the beginning of the test is K_{IAPP} = 1.5x60/950 x Rm (MPa.m$^{0.5}$). The duration of the test is at least 1000 hours. In this case, the material is considered suitable for use in hydrogen if:

- the average crack propagation does not exceed 0.25 mm;
- the average propagation exceeds 0.25 mm but the final K_I is at least equal to 60/950 x Rm (MPa.m0.5).

Method C is less precautionary than method B.

2.2.4 ANSI/CSA CHMC 1

The standard ANSI/CSA CHMC 1 [5] provides for the application of a series of test methods for a general evaluation of the compatibility of a metallic material in hydrogen. As per the previous standards, high purity levels of hydrogen in the autoclave are always required but the test temperature, which in all the previously mentioned tests is ambient, must be chosen in such a way as to achieve the conditions of maximum hydrogen embrittlement (Temperature of Maximum Hydrogen Embrittlement, TMHE). The standard describes how to identify this temperature.

Chapter 5 describes the test methods, while Chapter 6 indicates the qualification process of a material. Initially the material is characterized by means of a "slow strain rate test" (SSRT) on notched specimens. Smooth specimens can be used only for austenitic stainless steels or aluminum alloys. For notched specimens, the judgment criterion is based on the value of the relationship between the average tensile strength in hydrogen and in an inert environment. For smooth specimens, it is based on the ratio of reduction of area. If this ratio is greater than or equal to 0.90 for austenitic steels or aluminum alloys, the material is considered adequate. Otherwise, the general criterion for all other materials is that the ratio between the tensile strength of the notched specimens in the two environments is greater than or equal to 0.50. In case of a positive outcome further fatigue and fracture toughness, K_I or J_I, tests shall be performed on the material to obtain relevant engineering parameters to be included in the calculation of the structures of interest.

This standard does not consider welded joints, instead it characterizes the fatigue behavior of materials not only with fracture mechanic tests (da/dN-ΔK) but also with traditional curves S-N and also considers the use of the J_{IH} parameter as a measure of toughness in hydrogen thus also allowing the evaluation of more modern high yield materials with high toughness values in the elastic-plastic region. It is adapted for all metallic materials.

The following tests are reported:

- ✓ <u>Slow strain rate tensile testing</u>

This test method assesses the tensile properties and notch sensitivity of materials in gaseous hydrogen. This method is frequently used as a comparative method and appropriate for primary screening of materials.

Specimens shall be prepared according to ASTM G142 [16] following the guidance in Clause 10 "Test specimens" and the geometry specified therein. If the product form does not accommodate this specimen geometry, the specimen dimensions shall be consistent with ASTM E8 [17] with a gauge length of (at least) four times the gauges diameter. Notched specimens shall be designed according to ASTM G142 or shall have a notch stress concentration (Kt) greater than 3.

Tests shall follow the basic given in ASTM E8 and ASTM G142 (see Section 2.3) and be performed at constant displacement rate. For smooth specimens the nominal strain rate shall be 10^{-5} s^{-1} while for notched specimens the strain rate is nominally 10^{-6} s^{-1}. Tests with strain rate within a factor of 2 of these nominal values are acceptable.

- ✓ Hydrogen assisted cracking threshold stress intensity factor, K_{IH} or J_{IH}

This test method assesses the fracture threshold properties of material in gaseous hydrogen.

Compact tension or single edge bend specimens shall be prepared and tested according to ASTM E1820 [18]. Tests shall be conducted on notched and pre-cracked specimens under displacement control using either crack opening displacement or machine actuator position for the control variable. Displacement rate shall be selected such that the crack tip stress intensity factor K increases at a rate between 0.1 MPa√m/minute and 1 MPa√m/minute during the nominally elastic region of specimen loading. Compared to the test method described in ASTM E1681 which uses constant loading, this test method uses the rising loading method.

- ✓ Fatigue crack growth rate

This test method is used to characterise a material's resistance to stable crack propagation under cyclic loading within gaseous hydrogen environment. As determined over a range of cyclic stress intensity factor (ΔK), crack growth rate (da/dN) data can be used to determine material compatibility or for life cycle analyses.

Compact tension, middle tension and eccentrically-loaded single edge crack tension specimens shall be manufactured, pre-cracked and tested according to ASTM E647 [13]. The final maximum precracking stress intensity shall not exceed the initial maximum stress intensity of fatigue crack growth rate testing. FCG testing shall be conducted at constant force amplitude with R ratio of 0.1 and frequency of 1Hz.

- ✓ Fatigue life tests.

This test method is used to measure total fatigue life of metallic materials exposed to gaseous hydrogen.

Test specimens shall be prepared and tested in accordance with ASTM E466 [19] for force controlled fatigue tests and in accordance with ASTM E606 [20] for strain controlled tests. For notched specimens, the notched tensile geometry of ASTM G142 [16] is recommended. The notch severity, Kt, shall be greater than or equal to 3.

For force controlled fatigue life tests, tests shall be conducted with constant nominal stress amplitude throughout the test. The R ratio shall be equal to 1 for notched specimens and for smooth specimens equal to -1 and 0.1, respectively, to characterize tension compression and tension-tension fatigue behaviour. Test frequency shall be no greater than 1Hz for tests in the low cycle fatigue (LCF) regime and no greater than 20Hz for tests in the high cycle fatigue (HCF) regime.

For strain controlled fatigue life test. Tests shall be conducted with constant total axial strain amplitude or plastic axial strain amplitude. Test frequency shall be as detailed for force controlled fatigue life tests.

2.2.5 EIGA IGC Doc 121/14

EIGA document [6] provides an approach aimed at evaluating the suitability of metallic transmission and distribution piping systems, originally designed to transport natural gas, to carry pure hydrogen or hydrogen mixtures. It is limited to gaseous products with a temperature range between -40°C and 175°C and total pressures from 1 MPa (10 bar) up to 21 MPa (210 bar).

The information provided in this document applies only to future installations and not to existing installations or those in the project phase.

Steels and welds used in hydrogen pipeline service should have a maximum hardness of approximately 22HRC (Hardness Rockwell C) or 250 HB (Hardness Brinell) equivalent to a tensile strength (UTS) of 800 MPa. Seamless steel may have a UTS up to 950 MPa. Non-steel metallic materials stated in this document are nickel, copper and cobalt alloys. EIGA reports that nickel alloys should be avoided unless the user verifies the alloy is suitable for hydrogen gas service. Copper alloys are subject to hydrogen damage when copper contains oxygen; however, when deoxided copper alloys generally do not suffer hydrogen embrittlement (HE) and then are suitable for use in hydrogen. Cobalt alloys are considered acceptable for hydrogen gas service.

To evaluate the suitability of material to resist hydrogen gas embrittlement the document refers to test methods available in ISO 11114-4 (See Section 2.1.4 above) and in Section B4 of this document.

The following test methods for HE are reported:

- ✓ Tensile and notched tensile properties

The susceptibility of metals to hydrogen embrittlement can be evaluated by conducting tensile tests on smooth or notched specimens in hydrogen environment at different pressures. The test method is described in ASTM G142 [16] as reported in standard ANSI/CSA CHMC 1 [5] and Section 2.3 of this document.

- ✓ K_{IH} test

This test is a fracture mechanics test to evaluate the threshold stress intensity factor for hydrogen stress cracking. A pre-cracked specimen is loaded in tension and in gaseous hydrogen environment. The minimum applied stress intensity factor that can cause crack propagation (K_{IH}) can be used to evaluate the resistance to hydrogen assisted cracking. No specific test method is reported but ASTM or ISO test procedures can be used.

- ✓ SSR test

Since the hydrogen attack is a time dependent process, a slow strain rate test can be employed to evaluate the strain rate sensitivity of the materials in hydrogen environment. In the slow strain rate test, the test is conducted at strain rates as low as 10^{-7}. The test can be done in hydrogen environments or specimen can be pre-charged with hydrogen and subsequently tested in air or hydrogen. A general procedure of SSR tests is described in ASTM G129 [21] (see Section 2.3 below).

- ✓ Disk pressure test

Disk pressure test measures susceptibility to hydrogen embrittlement of metallic materials under high pressure hydrogen. A thin disk sample placed as a membrane in a test cell is subject to high pressure helium and hydrogen. The ratio between the helium burst pressure and the hydrogen burst pressure indicates that susceptibility of the material to environmental hydrogen embrittlement. A test method is described in ASTM F1459 [14] as reported in ISO 11114-4 [4].

2.2.6 EPRG

EPRG "European Pipeline Research Group" has developed a repurposing guideline white paper [7] which provides guidance for repurposing existing pipelines to near pure and blend hydrogen services and for new hydrogen pipelines based on ASME B31.12 [2].

To evaluate the suitability of materials in hydrogen environment this guideline recommends the following tests:

- ✓ Fracture toughness test

This test shall be performed according to ASTM E1820 [18] or E1681 [12] or equivalent (see Section 2.3). ASME B31.12 uses ASTM E1681 for K_{IH} testing; however, this guideline would recommend replacing it with ASTM E1820 especially where fracture mechanics calculations are required.

- ✓ SSR test

SSR test is recommended to be carried out according to ASTM G129 [21] described in EIGA document [6] and in Section 2.3 or equivalent.

- ✓ Fatigue test

Fatigue test shall be performed according to standards ASTM E647 [13] as described in standard ANSI/CSA CHMC 1 [5] and in Section 1.3 or Sandi [22]/ASME B21.12 [2] (see Section 2.1.2) fatigue curves.

2.2.7 IGEM/TD/1

This standard covers the design, construction, inspection, testing, operation and maintenance of steel pipelines and certain associated installations for the transmission of hydrogen, including H_2/NG mixtures, and for the repurposing of natural gas pipeline to hydrogen service, at minimum operating pressure exceeding 0.7 MPa (7 bar) and not exceeding 13.8 MPa (138 bar). Any H_2/NG mixture above 10% mol is considered to be equivalent to 100% hydrogen. For blends below 10% mol there is no evidence to confirm that blends containing up to 10% hydrogen do not cause material degradation and therefore this standard considers that the risk is low.

Grades higher the L485 (X70) shall not be used unless the pipe base and weld material are qualified for the intended service. The maximum UTS for both, the pipe and weld metal shall not exceed 690 MPa.

The standard states that to demonstrate that the material does not crack and that there is no significant reduction in toughness, smooth specimens shall be tested in a hydrogen-charged environment and pre-cracked specimens shall be used in an inert and hydrogen-charged environment.

A test using a smooth specimen will load the specimen at a constant load or displacement, or at a slow strain rate, in a hydrogen-charged environment for a specified period of time or until cracking occurs. It is a variant of a standard tensile test.

The tests can be used to:

- establish if a material will crack and the stress at which it cracks;
- establish a threshold stress for cracking;
- bound the limits of applicability of a material.

A test using a pre-cracked test specimen will load the specimen at a constant load or displacement, or at rising load or displacement, in a hydrogen-charged environment for a specified period of time or until cracking occurs or growth occurs. It is a variant of a standard fracture toughness test.

The tests can be used to establish:

- the threshold stress intensity factor for hydrogen assisted cracking, K_{IH};
- crack initiation toughness;
- tearing resistance;
- limit on stable ductile tearing in hydrogen-charged environment.

2.2.8 IGEM/H/1

This standard aims to identify and discuss the principles required for the safety and integrity of low pressure Hydrogen installation and utilization in premises. It covers only pure hydrogen (100%).

This standard reports the suitability of the following non-steel metallic materials for use in 100% hydrogen at less than 0.2 MPa (2 bar) pressure:

- Aluminum depending on alloy (1000, 2000, 7000 series alloys are suitable)
- Copper (oxygen free copper)
- Lead free solder/leaded solder
- Leaded brass (<2.5%)

Cat iron materials could possibly be suitable at low pressure but further data are required.

No data were found for lead, >2.5% leaded brass and chromium brass so the suitability is unknown and data would be required.

2.2.9 CEN/TR 17797

This technical report was written in preparation of future standardization and provides guidance on the impact of introducing hydrogen into gas infrastructures, from the point of entry into the transmission network to the point of entry of the utilization devices, providing ratings for progressive hydrogen concentrations from 2 % up to 100 % Vol.

This document suggests that the existing natural gas infrastructure within the pressure range up to and including 16 bar is able to accept hydrogen concentration up to 100 % hydrogen as far as the pipe and its various bends and tee-pieces are concerned.

Hydrogen tolerance of piping material used in natural gas infrastructure is reported for steel, stainless steel, copper alloys and polymer materials. No effect is expected for copper material with a hydrogen concentration ≤ 2 Vol. % H_2 at a pressure < 1MPa (10 bar) and with a hydrogen concentration ≤10 Vol. % H_2 at a pressure < 0.5MPa (5 bar).

Recommendation is given on assessing separately other pipe and fitting materials in historical use such as grey cast iron and/or ductile cast iron to ensure safety during hydrogen service. However, no testing methods are provided.

2.2.10 Summary of suitable tests and test standards to evaluate H_2 suitability

Following the review of the standards, the main tests and test standards to evaluate the suitability of materials in hydrogen environment are provided in Table 2.

Table 2 Summary of test and test standards

Tests	Standards
K_{IH}	ASME B31.12 ASME VIII Div.3 Article KD10 ASTM E1681 ASTM E1820 ISO 7539-6

Fracture toughness	ASTM E1820
Slow strain rate	ASTM G142 ASTM G129 ISO 7539-7
Fatigue crack growth	ASTM E647
Fatigue life	ASTM E466 ASTM E606
Disk pressure	ASTM F1459

2.3 Test method to evaluate the material behaviour in hydrogen

Following the review of standards and guidelines regarding the transport of H_2 or H_2/NG mixtures, there are various test methods to evaluate the suitability of materials in hydrogen environment. In this section, detailed test methods (including specimens' geometry and dimensions) and additional standard test methods most relevant to determine the H_2 suitability were reported.

The following test methods were described:

- ✓ K_{IH} testing
- ✓ Fracture toughness testing *K*, *J* and CTOD
- ✓ Slow strain rate testing
- ✓ Fatigue crack growth rate testing
- ✓ Fatigue life
- ✓ Disk rupture testing
- ✓ C-ring testing

The review of the test methods was based on ASTM and ISO standards.

2.3.1 K_{IH} test

K_{IH} test is a very common test to evaluate the toughness of materials in the presence of hydrogen and more accurately the threshold stress intensity factor. This test is stated in all standards previously studied (Section 2.2) and its test method is described in ASME B31.12 [2], ASME VIII Div.3 - Article KD 10 [3], ASTM E1681 [12], ASTM E1820 [18] and ISO 7539-6 [15].

ASME B31.12

According to ASME B31.12, a material shall be qualified for adequate resistance to fracture in hydrogen gas by following the rules and testing procedure reported in article KD-10 of ASME BPV Section VIII, Division 3 [2], where the K_{IH} must be measured in accordance with the test method reported in KD-1040 which is based on ASTM E1681 standard "*Standard test method for determining threshold stress intensity factor for environment-assisted cracking of metallic materials*" [12].

A set of three specimens shall be tested from each of the following locations: the base metal, the weld metal and the HAZ of welded joints. Specimens shall be in the T-L direction (i.e. extracted in the pipe transverse direction and notched thought thickness parallel to pipe longitudinal direction).

The value of K_{IH} measured shall not be less than 55 $MPa.m^{1/2}$ and the lowest value shall be used in the pipeline design analysis.

ASME VIII Div.3 - Article KD 10

At least three specimens shall be tested to obtain a set of three K_{IH} measurements. Specimens thickness shall not be less than 85% of the design thickness of the material to be qualified. Specimens shall be extracted in the T-L direction, pre-cracked and loaded by a constant load or a constant displacement method.

For a test conducted using the constant load method, the fatigue – pre-cracked test specimen is loaded to a stress-intensity determined from the fracture analysis.

For a test conducted using the constant displacement method, the fatigue – pre-cracked test specimen shall be loaded to a stress-intensity, K_{IAPP}, at least 1.5 times greater than the estimated K_{IH} but less than 198 MPa.m$^{1/2}$. The standard provides values of stress-intensity for ferritic steels only. No stress-intensity values are provided for non-steel metallic materials.

Specimens must be tested for at least for 1000 hours, at room temperature, in a test chamber pressurized with hydrogen gas.

On test completion, the crack growth shall be measured at the following three positions: at the center of the crack front, midway between the center of the crack front and the ends of the crack front on each side surface.

If the average measured crack growth does not exceed 0.25 mm and the test is performed using the constant load method, $K_{IH} = K_{IAPP}$.

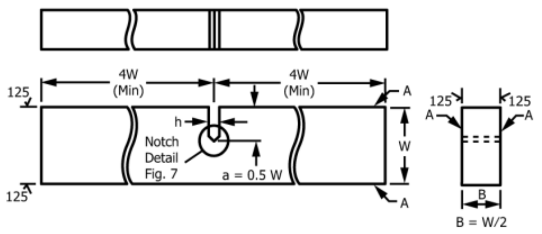
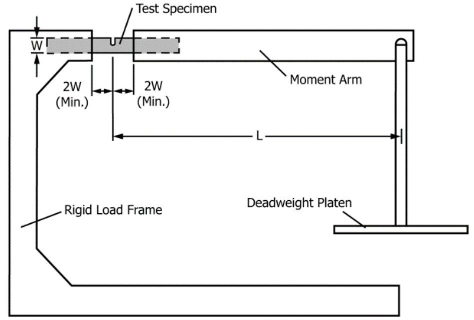
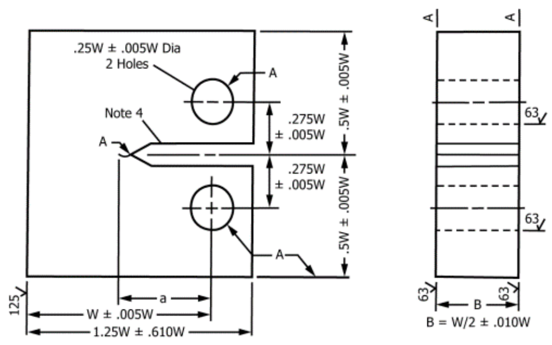
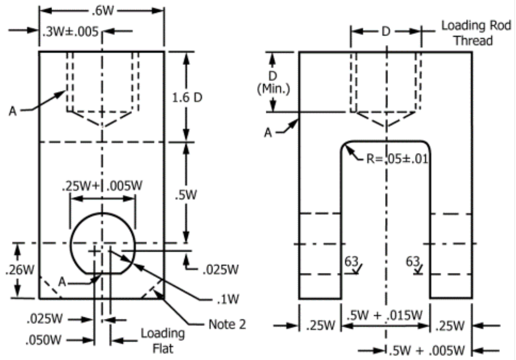
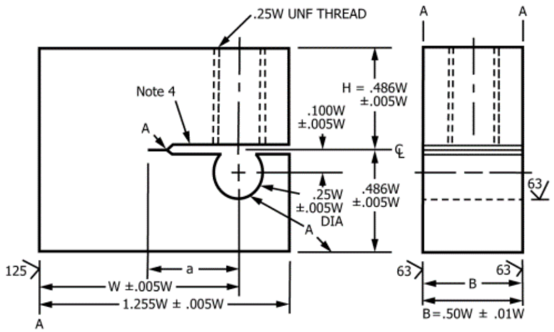
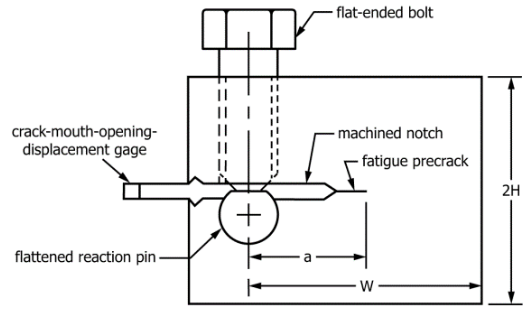
If the average measured crack growth does not exceed 0.25 mm and the test is performed using the constant displacement method, K_{IH} is equal to 50% of K_{IAPP}.

ASTM E1681

ASTM E1681 reports three specimen configurations (Table 3):

- Beam Specimen (SENB): specimen is tested with constant force. Specimen should be loaded with one end clamped in a stable rigid fixture and the other end clamped to a horizontal moment arm to which a force is applied. In such a fixture, the long axis of the specimen is placed horizontally with the notch opening facing upward.
- Compact Specimen (CT): specimen is tested with constant force. Both ends of the specimen are held in a clevis and loaded with pins to allow rotation of the specimen during testing.
- Bolt-Load Compact Specimen (CT-WOL): this specimen is used for constant displacement tests. The displacement is applied to the specimen containing a machined notch and fatigue pre-crack, with a bolt acting on a pin. The crack opening is measured using a crack mouth opening displacement device (CMOD).

Table 3 Summary of K_{IH} specimens

Specimen Type	Specimen configuration	Fixture	N° of specimens generally required	Stress intensity factor (K) calculation
Beam Specimen			4 – 6	$K_I = \frac{M}{B(W)^{\frac{3}{2}}} f(a_o/W)$
Compact Specimen			4 - 6	$K = \left[\frac{P}{BW^{\frac{1}{2}}}\right] f\left(\frac{a_o}{W}\right)$
Bolt-Load Compact Specimen			2 - 4	$K_I = [V_m E/W^{1/2}] f(a/w)$

A summary of the dimensions required for each specimen configuration is reported in Table 4.

Table 4 Specimen dimensions and crack length

	Beam Specimen	Compact Specimen	Bolt-Load Compact Specimen
W/B	1 - 2	1 - 2	1 - 2
a/W	0.25 – 0.75	0.25 – 0.75	0.3 – 0.95
$a_{pre\text{-}cracking}$	Greater between ≥0,10B and 1mm		

Note: W: width
B: thickness
a: crack length

Specimens shall be extracted in the T-L direction (same orientation as reported in the ASME standards). The number of specimens suggested per specimen type are reported in Table 3.

As ASME VIII Div.3, the specimens shall be pre-cracked and loaded using a constant load or constant displacement method determined with the equations reported in Table 3. The fatigue pre-crack shall extend to a depth of not less than 0.10B, or 1 mm, whichever is greater, beyond the tip of the machined notch as measured on each face of the specimen.

For this type of test, the exposure environment has a strong influence on the resulting K value. Once exposed to the environment, the specimen is loaded until one of the followings occurs:

- Rupture
- Presence of visible subcritical crack
- End of pre-set test time

ASTM E1681 only provides test duration for testing in ambient-temperature solutions of sodium chloride. No test duration is reported for non-steel metallic materials.

On test completion, the specimens fracture surfaces shall be examined, the fatigue pre-crack and crack growth (if present) measured, and the stress-intensity calculated following the equation provided in Table 3. The fatigue pre-crack shall be measured at the following three positions: at the center of the crack front, midway between the center of the crack front and the ends of the crack front on each side surface similar as AMSE VIII Div.3.

For the beam and compact specimens, the value of K_{IH} determined by ASTM E1681 test method is the highest applied *K* level that did not cause a fracture or evidence of subcritical crack growth in a specimen after reaching the recommended test duration.

For the bolt-load compact specimens, the value of K_{IH} determined by ASTM E1681 test method is the lowest applied K level that shows evidence of subcritical crack growth in a specimen after reaching the recommended test duration.

ASTM E1820

ANSI/CSA CHMC 1 standard [5] and EPRG document [7] recommend to use the test method as per ASTM E1820 "*Standard Test Method for Measurement of Fracture Toughness*" [18] to determine K_{IH} which uses the increasing load method which is different from the test method to determine the threshold stress intensity factor according to ASTM E1681 [12] which uses static-constant loading. The test method is described in the next section on fracture toughness (see Section 2.3.2).

ISO 7539-6

ISO 7539-6 "*Corrosion of metals and alloys – Stress corrosion cracking – Part 6: Preparation and use of precracked specimens for test under constant load or constant displacement*" [15] provides a test method similar to ASTM E1681 [12].

The test involves subjecting a pre-cracked fatigue specimen to a constant load or constant displacement at the load points during exposure to a chemically aggressive environment in order to determine the threshold intensity factor.

The same type of specimens as ASME E1681 (see Table 3) are recommended: those intended for constant load tests such as beam and compact specimens and those intended for constant displacement tests such as loaded specimens with a modified wedge opening (bolt-load compact) and double-cantilever beam (DCB).

Compared to ASTM E1681, beam and compact specimens have the same total width/thickness ratio, W/B = 2 while for the compact bolt-load specimen W/B = 2.55. For all specimens, the usable crack length, a, should be 0.45W to 0.55W.

Specimens shall be pre-cracked by fatigue loading until the crack propagates to at least 2.5% of W or 1.25 mm beyond the notch on the side surfaces.

The test shall be performed in a test chamber containing the chosen test environment for a minimum time of:

- Titanium: 10 hours
- Ultra-high-strength low alloy steels: 100 hours
- Lower-strength steels, high alloy steels and aluminium alloys: 1000 hours

The stress intensity factor is calculated using the equations reported in Table 5 .Y is a dimensionless stress intensity coefficient related to crack length expressed in terms of a/W, or a/H for (W-a) indifferent specimens, where W is the width and H is half-height of the specimen.

The stress value is equal to the highest of the various values determined for the same material.

Table 5 Stress intensity factors

Specimen configuration	Stress intensity factor
Compact tension or C-shaped	$K = \frac{YP}{B\sqrt{W}}$
T-type wedge-opening-loaded	$K = \frac{YP}{B\sqrt{a}}$
Double-cantilever-beam	$K = \frac{YP}{B\sqrt{H}}$

2.3.2 Fracture toughness testing *K*, *J* and CTOD (δ)

This test is used to evaluate the fracture toughness of metallic materials in terms of *K*, *J*, CTOD (δ) and R curves in the presence of hydrogen. It is performed on a pre-cracked specimen subjected to a monotonically increasing load, while the displacement of the crack opening is monitored. The test method is described in ASTM E1820 "*Standard Test Method for Measurement of Fracture Toughness*" [18]. ISO test methods not mentioned in the previous reviewed standards could also be used for determining the fracture toughness such as ISO 12135 "*Metallic materials – Unified method of test for the determination of quasistatic fracture toughness*" [23] and ISO 15653 "*Metallic materials – Method of test for the determination of quasistatic fracture toughness of welds*" [24]. Both ASTM and ISO standards are described below.

ASTM E1820

In this test method, two procedures can be used:

- ✓ A basic procedure directed toward evaluation of a single value without the use of crack extension measurement equipment.
- ✓ A resistance procedure directed toward evaluation of a complete fracture toughness resistance curve using crack extension measurement equipment.

The standard recommends three specimen configurations:

- Single edge bend specimen - SENB (Figure 3): is a single edge notched and fatigue cracked beam loaded in three-point bending with a support span, S, equal to four times the width, W.
- Compact specimen -CT (Figure 4): is a single edge notched and fatigue cracked plate loaded in tension.
- Disk-shaped compact specimen - DCT (Figure 5): is a single edge-notched and fatigue cracked plate loaded in tension as the CT.

For all specimens the recommended dimension W/B =2. Alternative size can be considered with 1 ≤ W/B ≤ 4 for the SENB and 2 ≤ W/B ≤ 4 for the CT and DCT specimens.

The crack size (total average length of the crack starter configuration plus the fatigue crack) shall be between 0.45 W and 0.70 W for J and CTOD determination. The fatigue pre-crack shall not be less than 0.5h where h is the notch height or 0.25mm.

Specimens shall be in the T-L direction (i.e. extracted in the pipe transverse direction and notched thought thickness parallel to pipe longitudinal direction).

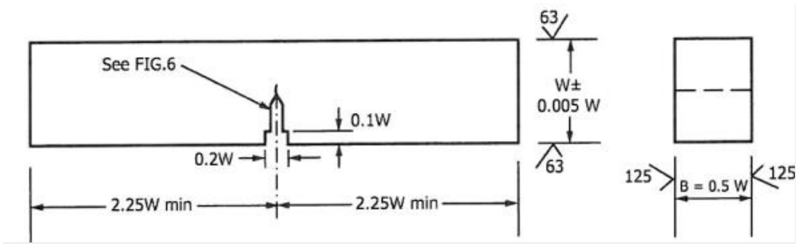


Figure 3 Recommended single edge bend specimen.

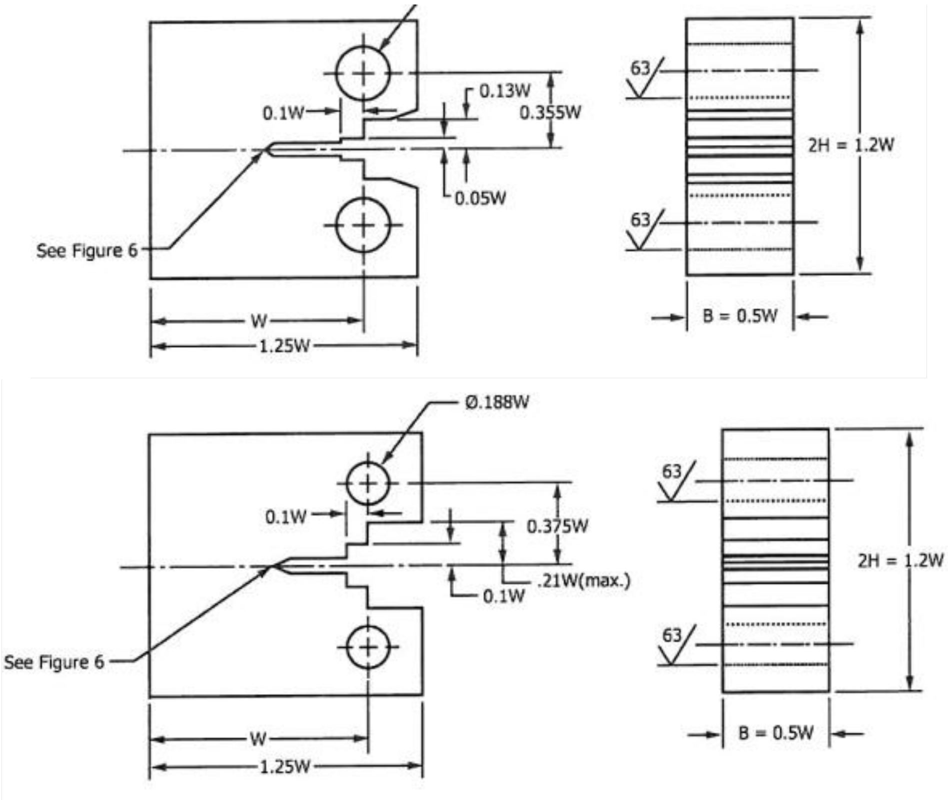


Figure 4 Two compact specimen designs

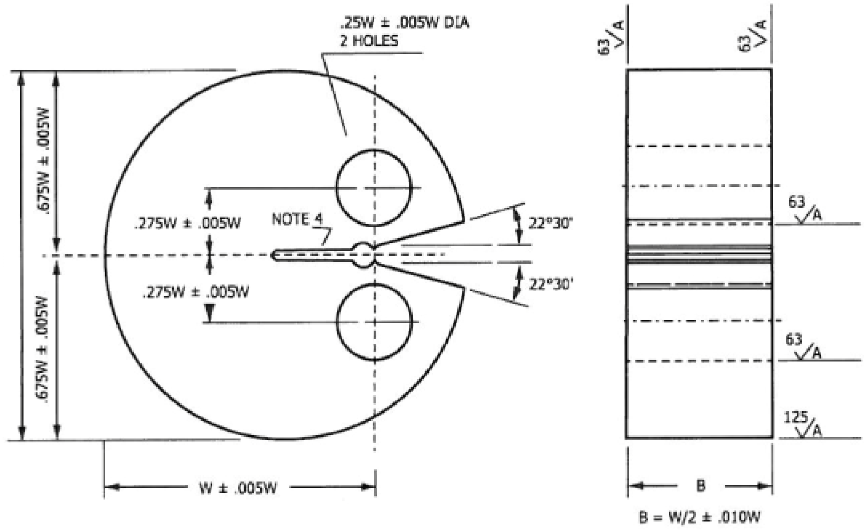


Figure 5 Disk-shaped compact specimen design.

The objective of this test method is to load a fatigue pre-cracked test specimen to induce either or both of the following responses:

- Unstable crack extension, including significant pop-in, referred to as “fracture instability” in this test method;
- Stable cracks extension, referred to as “stable tearing” in this test method.

Specimen shall be loaded under displacement gage or machine crosshead or actuator displacement control at a constant rate, using one of the following two procedures:

- Basic procedure which involves loading a specimen to a selected displacement level and determining the amount of crack extension that occurred during loading. This procedure is directed toward obtaining a single fracture toughness value such as J_c, K_{JIC} or δ_c.
- Resistance curve procedure which involves an elastic unloading procedure or equivalent procedure to obtain a *J*- or CTOD-based resistance curve from a single specimen.

A tearing resistance curve, or R-curve, represents a material's resistance to progressive crack extension. It is a plot of fracture toughness (J) against crack extension (Δa), as shown in Figure 6.

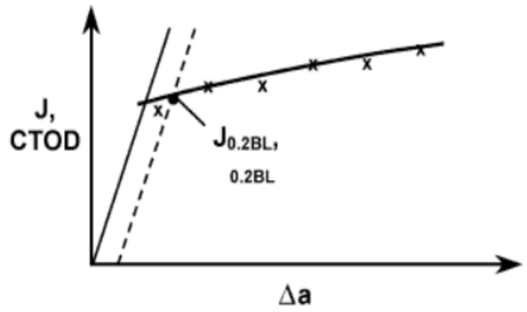


Figure 6 Example of tearing resistance curve, or R-curve [25]

Tearing resistance curves are only of use if a material exhibits stable tearing behaviour and cannot be generated beyond the onset of a brittle fracture event. In many ductile, work hardening materials, the size of the plastic zone at the crack tip increases as the crack extends. This type of behaviour is known as a 'rising R-curve'.

Data that can be generated from a tearing resistance curve include:

- the equation for the entire resistance curve;
- the value of J or CTOD at initiation of stable ductile tearing ($\delta_{0.2BL}$, $J_{0.2BL}$);
- the value of J or CTOD at maximum load (δ_m, J_m).

On test completion the original (pre-crack) and final crack size shall be measured at nine measurement points and shall not differ by more than $0.1(b_0B_N)_{1/2}$ from the average a_o for original crack size and a_p for final crack size, where b_o is the original remaining ligament ($b_o = W\text{-}a_0$).

The toughness parameters *K*, *J* and CTOD (*δ*) shall be calculated according to the equations provided in ASTM E1820 [18].

ISO 12135

In this standard, notched and pre-cracked specimens are subjected to quasi-static loading i.e. are tested under slowly increasing displacement.

The fracture toughness is determined for individual specimens at or after the onset of ductile crack extension or at the onset of ductile crack instability or unstable crack extension.

The recommended specimens are bend specimen, straight-notch compact specimen and stepped-notch compact specimen of size W/B = 2. Alternative sizes for bend specimen are 1 ≤ W/B ≤ 4 and for the compact specimen are 0.8 ≤ W/B ≤ 4.

Dimensions and tolerances of specimens shall conform to Figure 7 to Figure 9.

The test specimens shall be in the Y-Z direction which is the same as T-L direction provided in ASTM E1820 (i.e extracted in the pipe transverse direction and notched thought thickness parallel to pipe longitudinal direction).

The crack size (total average length of the crack starter configuration plus the fatigue crack) shall be between 0.45 W and 0.70 W for *J* and CTOD determination and between 0.45 W and 0.55W for K determination. The minimum fatigue crack extension shall be the larger of 1.3 mm or 2.5 % of the specimen width W.

All specimens for R-curve testing shall be side grooved, usually performed after fatigue pre-cracking, to a depth no greater than 0.2B.

When testing in gaseous environment, a soaking time of at least 60 s/mm of thickness shall be employed. The minimum soaking time at the test temperature shall be 15 min. The temperature of the test specimens shall remain within 2˚C of the nominal test temperature throughout the test.

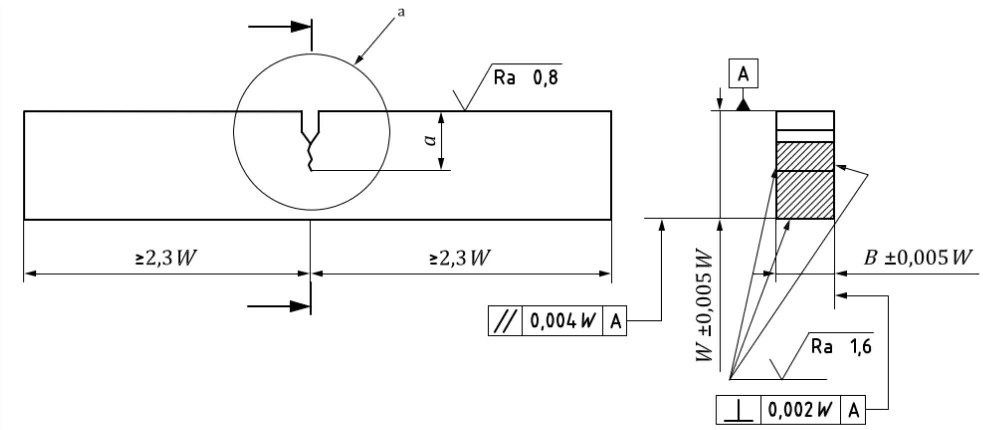


Figure 7 Dimension for bend specimen.

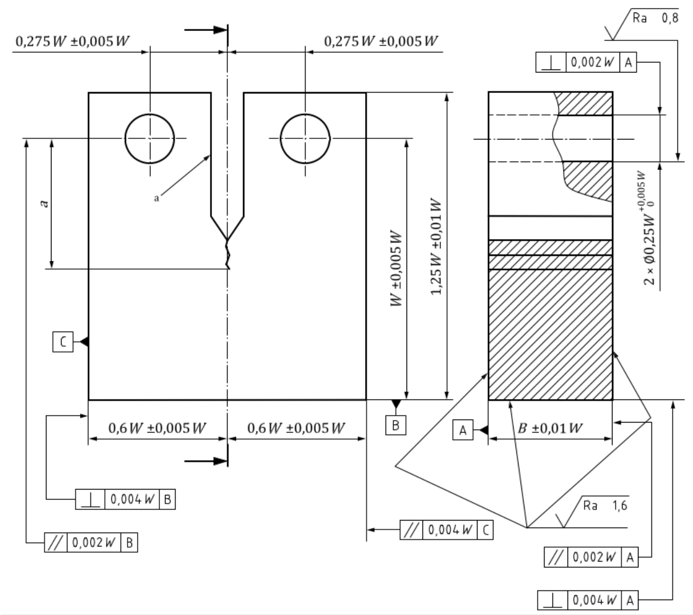


Figure 8 Dimensions for straight-notch compact specimen.

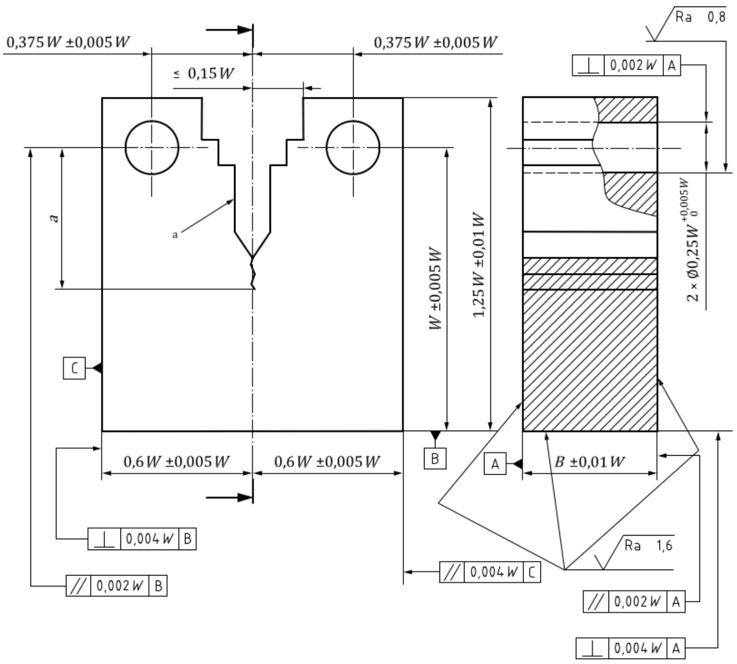


Figure 9 Dimension for stepped-notch compact specimen.

The specimens shall be tested with three-point bend or tension loading at a constant rate between 0.2 MPa $m^{1/2}$ s^{-1} and 3.0 MPa $m^{1/2}$ s^{-1}.

The fracture toughness parameters *K*, *J* and CTOD (δ) shall be calculated according to the equations provided in ISO 12135 [23].

ISO 15653

This standard provides methods for determining fracture toughness of welds in metallic materials. It complements ISO 12135 [23] described above which need to be used in conjunction with this document. Specimens design shall be of compact or single-edge-notched bend configuration as defined in ISO 12135 (see Figure 7 to Figure 9) and may be plain-sided or side grooved.

The specimens are recommended to have the dimension B or W equal to the full thickness of the parent metal adjacent to the weld to be tested and shall be extracted in the NP orientation (specimens normal to weld direction and notch parallel to weld direction).

Specimens shall be pre-cracked and tested as per ISO 12135.

2.3.3 Slow strain rate testing (SSRT)

This test method performed in a hydrogen gaseous environment evaluates the loss of material ductility and strength caused by hydrogen embrittlement. It is basically a tensile test performed on a smooth or notched specimen, slowly pulled at a constant strain rate until failure while exposed to the environment of interest. The mechanical properties obtained in a hydrogen containing environment are then compared to those obtained in a non-embrittling environment providing a general index of susceptibility to cracking versus the material's normal mechanical behaviour.

A test method is provided in ASTM G142 [26], ASTM G129 [27] and ISO 7539-7 [28].

ASTM G142

ASTM G142, "*Standard Test Method for Determination of Susceptibility of Metals to Embrittlement in Hydrogen Containing Environments at High Pressure, High Temperature, or Both*" [26], covers a procedure to determine tensile properties of metallic materials in high pressure or high temperature, or both, in gaseous hydrogen-containing environments. The results can be used to evaluate the effects of material composition, processing and heat treatment but also the effects of changes in environment composition, temperature and pressure.

Two test specimens can be used: smooth tension specimen and notched tensile specimen. The dimensions of the specimens are provided in Figure 10.

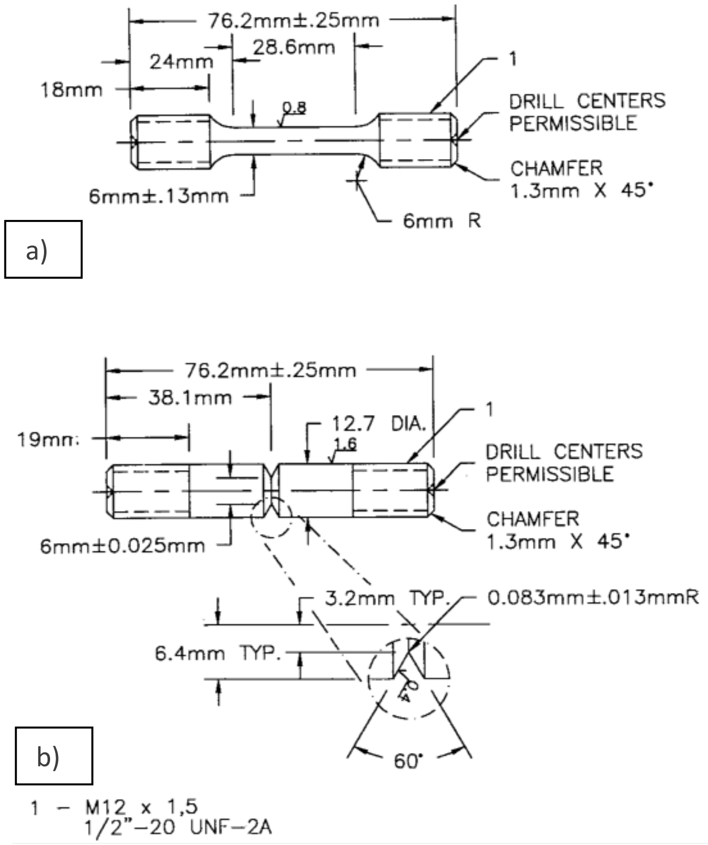


Figure 10 Standard tension specimen: a) smooth and b) notched.

Once the specimen is mounted in the test cell and the test cell is sealed, the oxygen level in the test cell is reduced to the desired level using alternate vacuum /inert gas purges. For testing in hydrogen containing environment, it is recommended that at least three vacuum/inert gas cycles be used.

Prior testing, the test cell shall be pressured tested with inert gas to at least the intended test pressure and held for at least 10 min. Upon completion of the pressure test, another vacuum to the inert gas shall be applied. The test gas shall be then back-filled into the test cell and pressurized to the intended pressure.

Smooth specimen shall be pulled at a constant extension rate of 0.002 mm/s ± 10% based on extension in the gage section of the specimen. For notched specimens an extension rate of 0.02 mm/s ± 10% based on the testing machine cross-head extension shall be used. The test is performed until failure of the specimen.

At the end of the test, the values of the parameters such as the plastic elongation (i.e. the elongation from the elastic limit to failure), the ultimate tensile strength and the reduction in area are determined from the load displacement curve, both for specimens smooth and carved.

These parameters are then used to evaluate the test results and to evaluate the susceptibility to cracking in hydrogen containing environments. It is common to use the ratio of these parameters with the corresponding data obtained for the same material in the control test conducted in an inert gaseous environment at the same temperature and pressure as the test in hydrogen. Values of these ratios close to unity indicate high resistance to hydrogen embrittlement while lower values indicate susceptibility to embrittlement.

ASTM G129

The test method provides in ASTM G129 "*Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking*" [27] is similar to the one from ASTM G142 described above. Smooth or notched tensile specimens and also pre-cracked specimens are axially loaded to study the resistance of metallic materials to environmentally assisted cracking.

Tension specimens should conform to the dimensions and guidelines provided in Test Methods ASTM E8/E8M [17]. An example of round tensile specimen is shown in Figure 11. The specimen nominal diameter, D, can vary between 2.5mm and 12.5mm with a reference length equal to four time the nominal diameter.

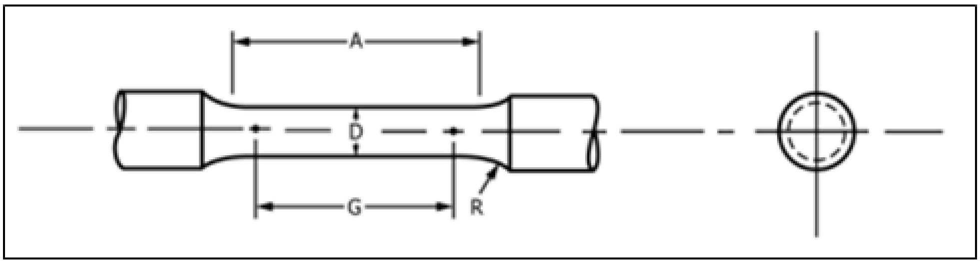


Figure 11 Example of round tensile specimen.

Fatigue pre-cracked specimens should conform to dimensions and guidelines developed for plane strain conditions in Test Method E 399 [29] or the size requirements for predominately linear elastic conditions as stated in Test Method E 647 [30]. Fatigue cracking should be conducted in accordance with ASTM E1681 [12].

For both specimens, non-standard specimens can be used, and results obtained should be compared to specimens with similar geometries.

Specimens shall be tested at very slow strain rates, which are achieved by constant extension rate while monitoring load and extension rate on specimens, until fracture. Strain rates shall be used in the range from 2.54 x 10-3 and 2.54 x 10-6 mm/s. For smooth tension specimens the strain rate shall be used in accordance with Test Method E8 [17].

As per ASTM G142, the results shall be compared to corresponding test results for the same material in a control environment.

ISO 7539-7

ISO 7539-7 *"Corrosion of metals and alloys – Stress corrosion testing – Method for slow strain rate testing"* [28] covers procedures for conducting slow strain rate tests for investigating susceptibility of a metal to stress corrosion cracking, including hydrogen-induced failure.

Tests may be conducted in tension or in bending, on plain or notch specimens. A variety of specimen shapes and sizes can be used such as compact tension, C-shaped, Disc, 3-4 point bend specimens, etc.

Test shall be conducted at strain rates between 10^{-5} s^{-1} and 10^{-7} s^{-1} in the test environment required until failure.

On test completion, to assess the susceptibility to stress corrosion cracking, comparison between identical specimens exposed to the test environment and to an inert environment may be used. The ratio of the results from specimen in test environment/results from specimens in inert environment should be applied to one or more of the following parameters of the same initial strain rate: time to failure, plastic strain failure, ductility (reduction in area or elongation to fracture), maximum load achieved, area bounded by nominal stress/elongation curve and percentage of stress corrosion cracking on the fracture surface.

2.3.4 Fatigue crack growth rate testing (FCGRT)

FGCRT helps to determine the resistance to fatigue failure of a material or component during its service life. For fatigue testing, a cyclic compression tension or bending stress is applied to pre-cracked notched sample. The fatigue life of a material or component in a hydrogen gas environment can be determined by the growth rate of a preexisting crack.

The test method described in ASTM E647 "*Standard Test Method for Measurement of Fatigue Crack Growth Rates*" [30] is recommended by various standards and guidelines. A test method is also provided in ISO 12108 *"Metallic material – Fatigue testing – Fatigue crack growth method".*

ASTM E647

This test method covers the determination of fatigue crack growth rates from near-threshold to K_{max} controlled instability, involving cyclic loading of notched specimens which have been pre-cracked in fatigue.

In this standard, the fatigue crack growth da/dN addresses regions I and II of the crack growth curve. The crack growth is mainly determined for ductile materials.

The crack growth test can be performed on three different specimen geometries: compact tension, C(T), middle-tension, M(T), or eccentrically loaded single edge crack tension, ESE(T), as shown in Figure 12 to Figure 14.

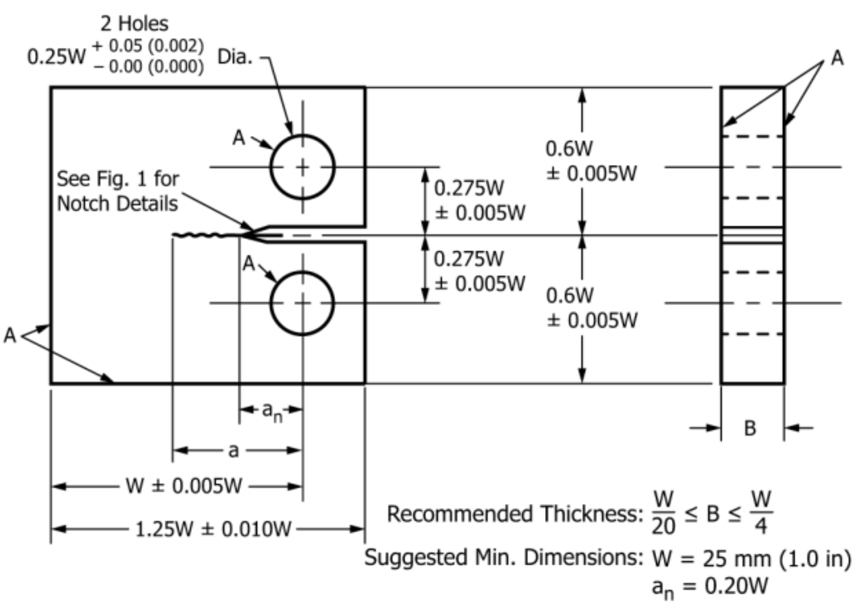


Figure 12 Standard compact specimen.

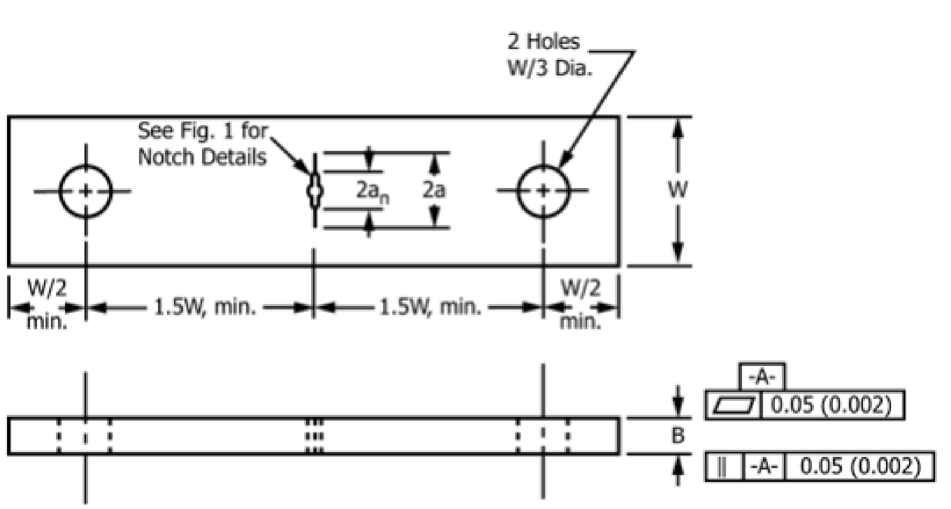


Figure 13 Standard middle tension specimen.

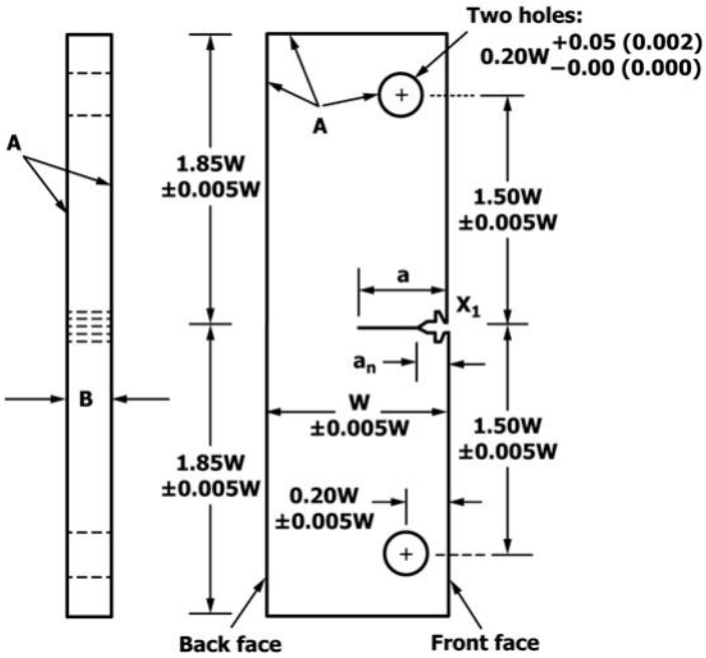


Figure 14 Standard eccentrically loaded single edge crack tension specimen.

Pre-crack is introduced by cyclic loading. It is important to ensure that the last maximum K (K_{max}) in pre-cracking is less than the K_{max} used at the start of a FCGR test. To achieve this, pre-cracking is often conducted at a stress ratio lower than that used in the subsequent FCGR test.

FCG tests can be carried out under increasing or decreasing ΔK.

The K-decreasing test method is used to determine the threshold value Δ_{Kth} (region I) and the Paris curve (region II). This method is based on decreasing stress intensity factor (SIF) range in small steps (shall not exceed 10% of the previous maximum stress-intensity factor to avoid crack growth retardation phenomenon) or continuously, as the crack grows. The fatigue crack growth threshold is reached when the crack propagation rate achieves the value of 10^{-7} mm/cycle. During K-decreasing testing, the test can be performed at constant stress ratio R during the entire test period. Load reduction is done through a non-constant K shedding rate C which is known as normalized K-gradient and should be greater than -0.08 mm-1 for steel and aluminum alloys.

Another approach is to test at constant maximum stress intensity, K_{max}, while ΔK is decreased by increasing K_{min} until the rate of crack growth reaches the threshold value. In order to maintain a constant K_{max} value the applied loads are varied. In this load reduction test procedure, the stress ratio can vary.

The K-increasing test method or constant-force-amplitude test as mentioned in this standard is used to determine the general crack growth curve (region II) and the Paris curve. The determination of the threshold value (Δ_{Kth}) is not possible. In this method, the maximum and minimum loads are kept constant so ΔK increases with increasing crack size.

On test completion, the crack length is measured as a function of elapsed fatigue cycles and these data are subjected to numerical analysis to establish the rate of crack growth. To characterise a material's resistance to stable crack extension under cyclic loading the results are expressed in terms of the fatigue crack growth rate (*da/dN*) as a function of ΔK, as defined by the theory of linear fracture mechanics. The material constant as well as validity check of the FCGR data obtained including crack front curvature and minimum un-cracked ligament shall be determined.

ISO 12108

ISO 12108 provides a similar test method as ASTM E647 describing a method of subjecting a pre-cracked notched specimen to cyclic force. Specimen size can vary over a wide range and geometries are the same as ASTM E647 (see above).

The main differences is the definition of the crack growth threshold, Δ_{Kth}, which correspond to a value of 10-8 mm/cycle (compared to a value of 10-7 mm/cycle for ASTM E647) and the specification of the parameter C which should be greater than -0.1 mm^{-1} (compared to a value of -0.08mm^{-1} for ASTM E647).

2.3.5 Fatigue life testing

This test is used to determine the total number of load cycles to failure of a specimen subjected to repeated loading. Specimen tested in hydrogen gas are compared to specimens tested in inert environments to determine the effect of hydrogen on materials.

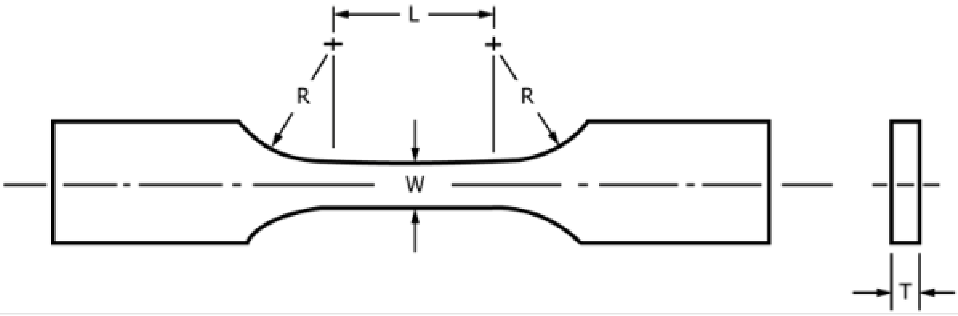
The test method is reported in ASTM E466 “Standard Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials” [19] and ASTM E606 “Standard Test Method for Strain-Controlled Fatigue Testing” [20].

ASTM E466

This test method is to obtain the fatigue strength of unnotched and notched metallic materials subjected to an axial force controlled constant amplitude.

The specimen can be round or flat as Figure 15 and Figure 16 such that failure occurs in the test section.

Testing shall be performed at stress amplitude previously determined as per ASTM E647 (see Section 2.3.4) at a frequency of 10^{-2} to 10^{+2} Hz.



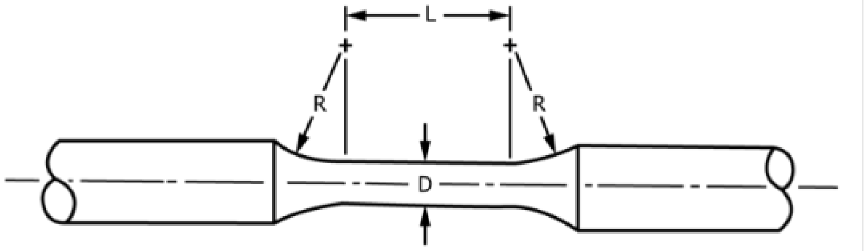


Figure 15 Round specimen

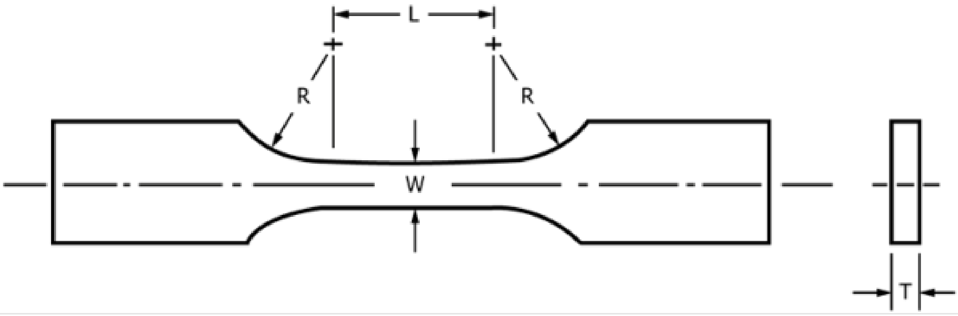


Figure 16 Flat specimen

ASTM E606

This test method covers the determination of fatigue properties by the use of test specimens subjected to uniaxial strain-controlled forces.

The recommended test specimen is shown in Figure 17. The cross section shall have a minimum diameter of 6.35 mm in the test section. Flat specimens can also be machined (Figure 18) for material that is less than 6 mm thick. A minimum of ten specimens shall be used to generate a fatigue strain-life curve.

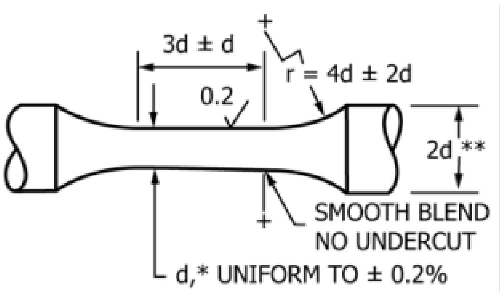


Figure 17 Standard fatigue specimen

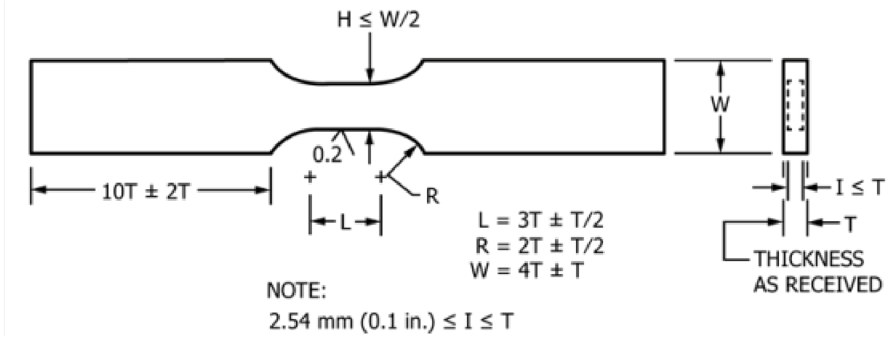


Figure 18 Flat fatigue specimen

2.3.6 Disk rupture testing

Disk pressure test measures susceptibility to hydrogen embrittlement of metallic materials under high pressure hydrogen. A thin disk sample placed as a membrane in a test cell is subject to high pressure helium and hydrogen. The susceptibility of a material is then evaluated by identifying the ratio between the test in helium vs in hydrogen.

The test method is presented in ASTM F1459, "*Standard Test Method For Determination Of The Susceptibility Of Metallic Materials To Hydrogen Gas Embrittlement (HGE)*" [14].

The disk pressure technique involves a disk-shaped specimen, of specified size, which is pressurized on one side until the disk ruptures. Failure pressure measured using hydrogen gas (P_{H2}) is compared with the failure pressure using an inert gas (usually helium, P_{He}) to determine an embrittlement index (P_{He}/P_{H2}). P_{He}/P_{H2} ratio is plotted versus pressurization rate over a range of rates; the maximum value of P_{He}/P_{H2} is recorded.

2.3.7 C-ring testing

This kind of test was not mentioned in the standards previously reviewed but is a common test used to also evaluate the susceptibility of a metallic material to hydrogen gas. The test method is provided in ASTM G38 "*Making and Using C-Ring Stress-Corrosion Tests Specimens*" [31] and ISO 7539-5 "*Corrosion of metals and alloys – Stress corrosion testing – Part 5: Preparation and use of C-ring specimens*" [32].

ASTM G38

In this test method C-ring specimens shall have an outside diameter higher than 16mm with typical dimensions as shown in Figure 19.

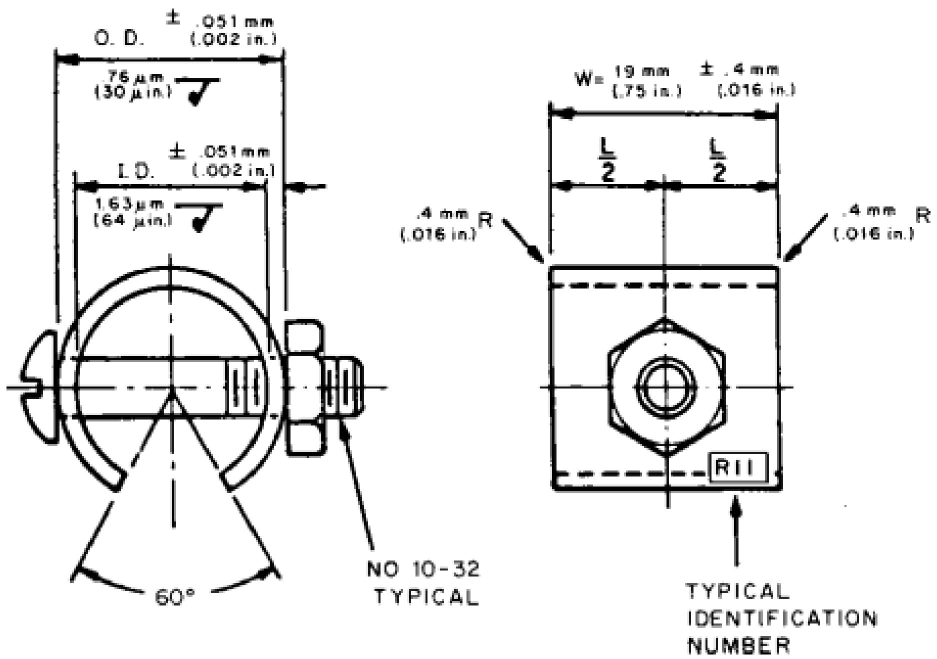


Figure 19 Standard C-ring specimen.

C-rings specimens can be loaded with tensile stress produced on the exterior of the ring using constant displacement (constant-strain) by tightening a bolt centered on the diameter of the ring or constant load by using a calibrated spring placed on the loading bolt. C-rings specimens can also be loaded with stress produced on the internal surface by spreading the ring. C-rings are bolt loaded to the desired stress level, usually comprised between 75 and 100% of the elastic limit.

C rings are stressed to known values, which are usually reported as a percentage of the yield strength. Deflections should be limited to stresses below the material elastic limit. The deflection necessary to obtain the desired stress on the C-ring test specimen shall be calculated using the following equations [30]:

$$OD_f = OD - \Delta \qquad \text{(Equation1)}$$

$$\text{With } \Delta = f \pi D^2 / 4EtZ$$

Where:

OD = outside diameter of C-ring before stressing

ODf= outside diameter of stressed C-ring

f= desired stress

Δ= change of OD giving desired stress

D = mean diameter (OD-t)

t = C-ring wall thickness

E = modulus of elasticity

Z= a correction factor for curved beams

Before exposure, the specimen should be instrumented, in the middle of the arc, with strain gauges located circumferentially and transversally to the surface stress in tension. When several rings of the same material and dimensions are to be loaded, a calibration curve of circumferential stress versus ting deflection shall be determined to avoid instrumenting each individual specimen.

For notched specimen, a nominal stress shall be assumed using the ring outside diameter measured at the root of the notch.

C-ring must be exposed to the test environment immediately after being stressed or stored in such a way as to avoid contamination or deterioration until the time of exposure.

Hydrogen embrittlement susceptibility with the C-ring tests specimen is usually determined by time to cracking during the test. Determination of cracking time is a subjective procedure involving visual examination that under some conditions can be very difficult and depends on the skill and experience of the inspector. In any case, specimens could be examined metallographically at the conclusion of the tests for evidence of cracking.

ISO 7539-5

This standard referred to similar specimen design and test method as ASTM G38.

2.3.8 Summary of test methods

K_{IH} test determines the threshold stress intensity factor of a material. It uses pre-cracked specimens type beam, compact and bolt-loaded compact specimens, of thickness > 85% of the material nominal thickness to be qualified, loaded at constant load or constant displacement for a defined period a time. According to ASME B31.12, ASME VIII Div.3 and ASTM E1681, multiple specimens can be tested at the same time. Crack growth shall be less than 0.25 mm for a material to be considered suitable to hydrogen gas. K_{IH} value is then determined been equal to K_{IAPP} or 50% of K_{IAPP}, respectively, for test conducted using constant load and constant displacement method.

This test provides information on the threshold stress intensity factor but it is not possible to determine the crack growth initiation. To overcome this, the industry tends to prefer the application of an increasing load method as per ASTM E1820 but to date the test speed to be adopted to better capture the effect of hydrogen and the most suitable method to identify the crack growth initiation have not been defined. Compared to the other standards previously mentioned with this test method only one specimen at the time can be performed.

Fracture toughness test provides the toughness values in hydrogen of a material. The test also uses pre-cracked specimens similar as K_{IH} test. Here the specimens are continuously loaded and tested one by one. Toughness parameters are determined from a single test.

SSRT evaluates the susceptibility to hydrogen embrittlement through the determination of standard mechanical properties in tension. These are then compared to those determined in a non-embrittling environment providing a general index of susceptibility to cracking versus the material's normal mechanical behaviour. Due to the accelerated nature of this test, the results are not intended to necessarily represent service performance, but rather to provide a basis for screening, for detection of an environmental interaction with a material, and for comparative evaluation of the effect of metallurgical and environmental variables on sensitivity to known environmental cracking problems.

FCGRT determines the resistance to fatigue failure of a material or component during its service life. A pre-cracked notched specimen is subjected to cyclic force in compression tension or bending and the fatigue life is determined by the crack growth rate of the fatigue crack.

Fatigue life test determines the total number of load cycles to failure of a specimen subjected to repeated loading. A smooth or notched specimen is subjected to uniaxial cyclic forces until failure.

Disk rupture test evaluates the susceptibility to hydrogen embrittlement by subjecting a thin disk sample to high pressure hydrogen. The same test is also performed in high pressure inert gas and the failure pressure measured using hydrogen gas is compared with the failure pressure using an inert gas.

The C-ring test also evaluates the susceptibility to hydrogen embrittlement by loading the exterior or internal surface of the specimen using constant displacement or load. It is particularly suitable for making transverse tests on tubular products.

2.4 Applications, limitations and gaps

2.4.1 Standards and guidelines

Table 6 summarizes the application and limitation ranges of the revised standards and guidelines on testing and qualification of materials compatibility with hydrogen gas together with relevant tests that can be performed to evaluate the suitability of materials in hydrogen environment.

The standards and guidelines studied cover metallic materials at pressure < 210 bar and mainly steel materials which are the most used in distribution gas grids. Little information is provided on non-steel metallic materials. According to standards copper (oxygen free grade) and aluminium are suitable for hydrogen service; however, nickel (unless verified), cast iron and titanium based alloys are not suitable.

The standards foresee mechanical tests to identify materials suitable for hydrogen service and provide specific parameters to then be applied in the design of the component except for documents IGEM/H/1 and CEN/TR 17797. These documents provide information on the impact of H_2 concentration on materials which is studied in Section 2.6.

The standards provide test methods such as K_{IH}, fracture toughness test (also denoted as K_{IH}), SSR test, fatigue test like FCGR and disk pressure test based on steel materials providing limited testing parameters for non-steel metallic materials. These test procedures can be applicable to non-steel metallic materials; however, attention, thought and experience need to be given to the input/testing parameters.

Comparing the ASME standards from the standard ISO, the latter is more generic in its selection of compatible materials, labeling them as such only in following specific tests. The ASME standard, on the other hand, is more precise for the preventive choice of the material and foresees subsequently mechanical tests to provide specific parameters to then be applied in the design of the component. Furthermore, ASME standards consider welded joints as documents EIGA 121/4, EPRG, IGEM while the standard ISO and ANSI/CSA CHMC 1 do not consider welded joints. However, the latter characterises the fatigue behaviour of materials not only with fracture mechanic tests (da/dN-ΔK) but also with traditional curves S-N and compared to ASME VIII Div. 3, considers the use of the J_{IH} parameter as a measure of toughness in hydrogen thus also allowing the evaluation of more modern high yield materials with high toughness values in the elastic-plastic region.

Current standards are mainly based on steel materials which are the most commonly materials used in the distribution gas grid. Further research should be performed on non-steel metallic materials to provide information on their qualification for hydrogen gas service and therefore standards should be updated.

Table 6 Summary of standards and guidelines on testing and qualification of materials compatibility with hydrogen gas

Standards/Guidelines	**Items**					
	Components	**Materials**	**Hydrogen concentration**	**Hydrogen pressure**	**Non-steel materials**	**Tests to evaluate H2 suitability**
ASME B31.12	Transmission and distribution pipelines including pipes, fittings, valves, pressure vessels and associated equipment. Also include welded joints	Metallic materials	H_2/NG mixtures up to 100% H_2 by volume	≤ 21 MPa (210 bar)	Copper (oxygen free grade) and aluminium are suitable. Nickel, cast iron and titanium-based alloys are not suitable	• K_{IH} and FCGR (Article KD10 of ASME VIII, Div. 3)
ASME VIII Div. 3 – Article KD 10	Pressure vessels including welded joints	Metallic materials	Up to 100%	-	Apply to Nickel and nickel alloys, and aluminium alloys	• K_{IH} (KD 1040 and ASTM E1681) • FCGR (KD 1050 and ASTM E647)
ISO 11114-4	Seamless gas cylinder	Steel	-	• Working pressure > 20% of test pressure • H2 partial pressure > 5 MPa (50 bar)	-	• Disc test (ASTM F1459) • Fracture toughness/K_{IH} (ISO 7539-6)
ANSI/CSA CHMC 1	Do not consider welded joints	Metallic materials	-	-	-	• SSR (ASTM G142 and ASTM E8) • K_{IH} or J_{IH} (ASTM E1820) • FCGR (ASTM E647)

						• Fatigue life tests (ASTM E466 or ASTM E606)
EIGA 121/4	Transmission and Distribution piping systems including welded joints	Metallic materials	Up to 100%	> 1 MPa (10 bar) and < 21 MPa (210 bar)	Apply to Copper, nickel and cobalt alloys. Oxygen free copper alloys are suitable. Nickel to be avoid unless verified.	• Tensile (ASTM G142) • K_{IH} (various ASTM or ISO) • SSR (ASTM G129) • Disk pressure (ASTM F1459)
EPRG	Pipelines	Metallic materials	>10% up to 100%	-	-	• K_{IH} (ASTM E1820) • SSR (ASTM G129) • FCGR (ASTM E647)
IGEM/TD/1	Transmission pipelines including welded joints	Metallic materials	Up to 100%	> 0.7 MPa (7 bar) and < 13.8 MPa (138 bar)	-	• K_{IH} • Fracture toughness • SSR
IGEM/H/1	Domestic and smaller non-domestic premises		100% only	< 0.2 MPa (2 bar)	Aluminum (depending on alloy), copper, lead free solder/leaded solder and leaded brass are suitable Cast iron, lead (>2.5% leaded	-

					brass) and chromium brass data are required	
CEN/TR 17797	Transmission and distribution pipelines	Metallic and non-metallic materials (Steel, stainless steel, copper alloys and polymers)	From 2% up to 100%	$\leq$ 1.6 MPa (16 bar) infrastructure can accept up to 100% H_2	Copper and alloys (No effect for copper with ≤ 2 % H_2 at pressure < 1 MPa and ≤ 10 % H_2 at pressure < 0.5 MPa) and polymers	-

2.4.2 Test methods

The test methods to evaluate the susceptibility of a material to hydrogen gas are performed on machined specimens and will be limited by the material size available to be tested. In general, non-steel metallic materials used in the distribution gas grid have small thicknesses which will make the extraction of specimens difficult. The analysis of the non-steel metallic materials present in the gas distribution grid conducted in Task 2.1 [1] of CANDHy project reported that ductile and gray cast iron pipe material in use have a maximum thickness of 15mm and 17mm, respectively, and copper pipe only up to 2mm. No information was provided on the size of components of the distribution grid and reviewed test methods are not applicable on full size components; therefore, testing of small size specimens will be limited in terms of sample machining, machine capacity and measurement of physical and mechanical parameters.

The test methods are for machined specimens and not applicable for components. Specimens will need to be extracted from components to be tested which could be difficult due to the size and shape of components.

C-ring test would be easier to apply on small pipes of small thicknesses while specimens for fracture toughness, SSR and fatigue tests would need thicker materials of at least 6mm for round tensile specimens and 10mm thick for CT and CT-WOL specimens.

Tests such as SSRT, disk rupture test and C-ring test only allow to evaluate the susceptibility to hydrogen embrittlement by comparing tests performed in hydrogen to tests performed in inert environments while fracture toughness and fatigue tests provide a quantitative result given a toughness value, the crack growth and determine the fatigue life of a material. SSRT performed on notched specimen would allow to study the effect of H_2 in a material in presence of a notch.

Testing provided in current test methods are applicable to metallic materials and mainly to steel materials; therefore, there is a lack of requirements/parameters for non-steel metallic materials. For K_{IH} test, the test method in hydrogen gas is based on steel materials and no stress intensity value and test duration are provided for non-steel metallic materials.

To perform tests of non-steel metallic materials, assumptions will need to be made on test parameters which might not be correct or relevant for the material tested. Therefore, test methods should be updated.

2.5 Hydrogen readiness for non-steel metallic materials

ASME B31.12 reports that non-steel metallic materials made of copper and copper alloy and aluminum and aluminum alloy materials are acceptable for hydrogen gas; however, nickel and nickel alloys should be avoided because they are highly susceptible to H_2 and cast irons and titanium-based alloy materials are not acceptable for hydrogen gas.

EIGA reports that free oxygen copper and cobalt alloys are suitable for use in hydrogen; however, nickel alloys should be avoided unless the user verifies the alloy is suitable for hydrogen gas service.

IGEM/H/1 document states that, copper (oxygen free copper) is suitable for 100% H_2 at pressure < 0.2 MPa while CEN/TR 17797 states that H_2 has no effect on copper alloys when hydrogen concentration is ≤2 Vol. % at a pressure < 1MPa (10 bar) and ≤10 Vol. % at a pressure < 0.5MPa (5 bar). For a pressure < 0.2 MPa, IGEM/H/1 also reports that aluminium (depending on alloy), lead free solder, leaded solder and leaded brass are suitable materials. However, cast iron, lead (>2.5 leaded brass) are not suitable.

MARCOGAZ which represents the European gas industry provided a report on an overview of the technical readiness of the gas infrastructure and end-use equipment to handle hydrogen-natural gas mixtures at each stage of the gas chain, focusing on material aspects and functional principles and identifying a hydrogen admissible percentage. The study reported that for ductile cast iron pipeline (< 1 bar) the results are mostly positive for a hydrogen gas percentage up to 100 %. For fittings, no significant issues were observed and therefore are suitable for hydrogen gas service.

2.6 Defect tolerance assessment

As described above, design codes, such as ASME B31.12 and ANSI/CSA CHMC 1, provide prescriptive guidelines and safety factors for the design and construction of components and systems intended for hydrogen service. These codes are based on pessimistic assumptions about material properties, operating conditions, and the expected lifespan of components, ensuring that new installations meet minimum safety and performance criteria from day one. However, even in such cases, once a component is in operation, it may no longer conform to these initial design specifications due to wear, damage, or degradation over time. This is where fitness-for-service (FFS) assessment procedures become critical. FFS assessments evaluate the current condition of an in-service component, considering defects, operational history, and environmental factors to determine whether it can continue to operate safely and effectively. Unlike design codes, which typically assume a defect-free condition, FFS procedures account for the real-world state of the component, enabling decisions on repair, continued operation, or decommissioning. Of particular interest to the CANDHY project is the assessment of the natural gas non-metallic materials to hydrogen service.

The material property requirements specified in design codes like ASME B31.12 (R1 in Figure 20) and ANSI/CSA CHMC 1 (R2 in Figure 20), such as fracture toughness and fatigue crack growth rate in hydrogen environments, can be directly applied in fitness-for-service assessments. These properties provide essential data for evaluating the residual life and safety of components that have been exposed to hydrogen over time.

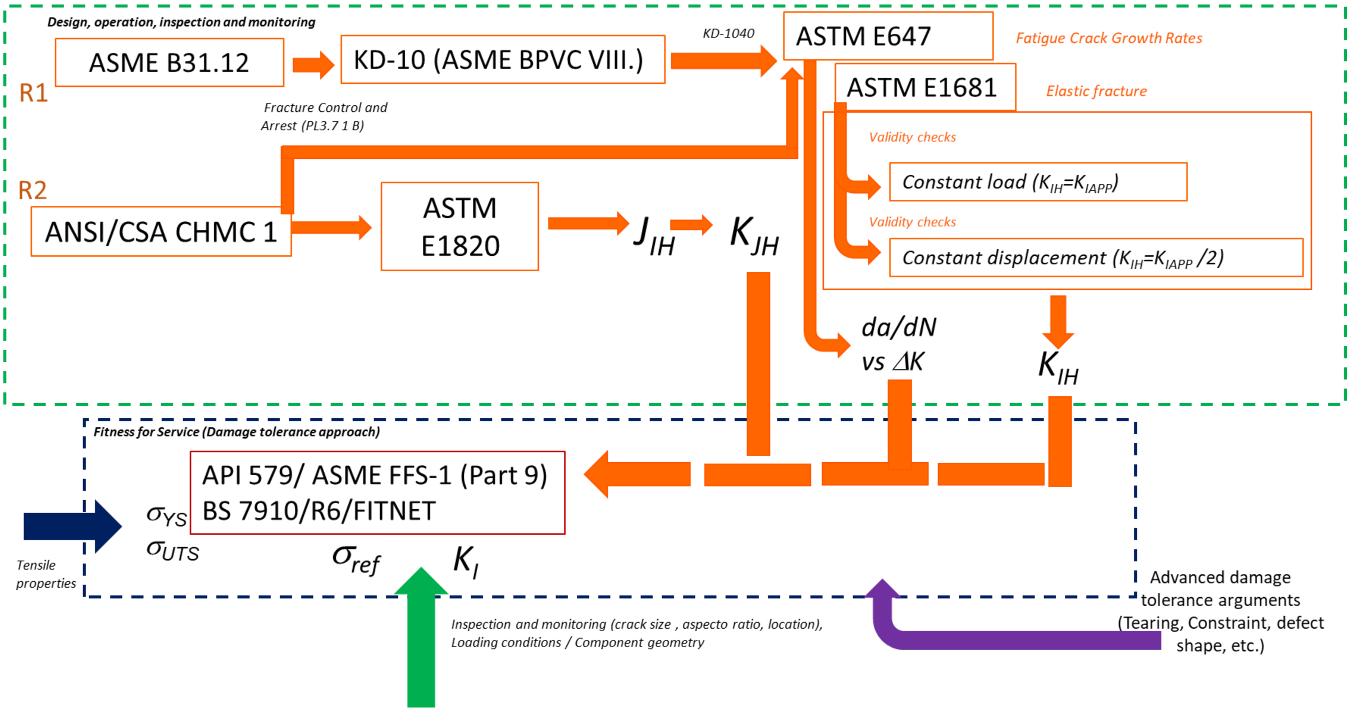


Figure 20 Schematic of the relation between design codes and fitness for service assessments.

Fracture mechanics-based approaches are typically used for defect tolerance assessments (DTA). In particular, the Failure Assessment Diagram (FADs) approach is a useful tool for evaluating the integrity of materials, with applicability to assessing critical conditions to failure and perform sensitivity analysis under elastic, elastic-plastic and fully plastic conditions with the ability of incorporating failure mechanisms such fatigue crack growth, hydrogen embrittlement and many more. In this context, under hydrogen service conditions, FADs could be used to providing insight into safe operational limits and to understand the effects of varying material properties, loading conditions and defect shape and sizes on the margins of safety.

The FAD methodology involved the assessment of two main parameters. Within the fracture avoidance demonstration, a Failure Assessment Diagram (FAD) is used for fracture analysis and the demonstration of the acceptability of defects. Two main dominant failure criteria, linear elastic fracture and plastic collapse are assessed independently and integrated using the FAD framework allowing the whole range of crack tip elastic and plastic deformations to be considered, Figure 25. The parameters required in any FAD assessment are the load parameter (L_r) and the fracture parameter (K_r), which indicate the degree of proximity to plastic collapse and fracture, respectively:

$$L_r = \frac{P}{P_L} = \frac{\sigma_{Ref}}{\sigma_Y} \; ; \; L_r^{max} = \frac{\sigma_Y + \sigma_{UTS}}{2\sigma_Y} \qquad \text{(Equation 2)}$$

$$K_r = \frac{K_I}{K_{mat}} \qquad \text{(Equation 3)}$$

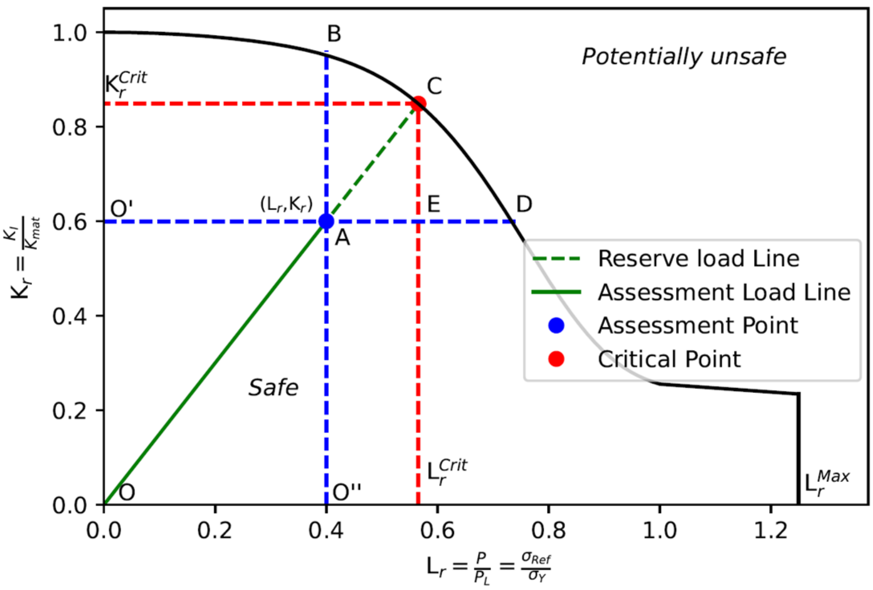


Figure 21 Schematic Failure assessment diagram (FAD) with failure assessment curve (black) assessment point, critical point and lines for reserve factor assessment.

where P is the applied load, P_L is the limit load, σ_Y is the yield strength, σ_{Ref} is the reference stress, i.e. the driving force for plastic collapse at the remaining ligament of the flawed section and K_I is the stress intensity factor, including primary and secondary loading. The failure assessment curve (black solid line) divides the safe from the potentially unsafe conditions and its shape is a result of the effect of crack tip plasticity on the fracture driving force for the case of primary stresses only. Fatigue crack growth effects can be considered by growing the crack as a function of loading cycles, moving the assessment point closer to the failure assessment curve.

The role of secondary stresses is accounted by adding a corrected value K_r^S using a factor designated either V, as given in API 579 or BS 7910:

$$K_r = \frac{K_r^P}{K_{mat}} + V\frac{K_r^S}{K_{mat}} \qquad \text{(Equation 4)}$$

The fracture toughness (K_{mat}) value implicitly accounts for the effect of the environment on the material's resistance to fracture and is typically obtained from elastic quasi-static fracture initiation testing methods such as ASTM E-399 or a value of toughness obtained from a *J*-resistance curve with some amount of tearing permitted as obtained from ASTM E-1820. Constant load/displacement testing methods, as standardised in ASTM E1681, have shown to provide no-conservative toughness values, with stronger effect on lower strength steels (Figure 20). In rising load testing methods, the level of plastic deformations ahead of the crack tip as the load level increases shows to affect the amount of hydrogen that ingresses the material locally, affecting the posterior ability to deform plastically and promoting quasi-brittle behaviour. A discussion about the appropriateness of constant load/displacement methods for this purpose is given in report developed by Sandia [33].

One key aspect of the application of the FAD curve is that it provides a graphical description of the critical crack driving force as a function of the applied load, allowing safety margins or reserve factors to be equated using simple trigonometric relationships. In Figure 21, for example, points lying underneath the FAD are in a safe condition (Point A), while points reaching the curve (Point C) or above are potentially unsafe. For this case, reserve factors on load, flaw size, toughness and strength can be straightforwardly evaluated to give an indication of the margins of safety associated with the operation of the component under the new conditions:

$$P^L = \frac{P^{crit}}{P} = \frac{\overline{OC}}{\overline{OA}}$$
$$P^a = \frac{a^{crit}}{a}$$
$$P^K = \frac{K_{mat}}{K_{mat}^{crit}} = \frac{\overline{O''B}}{O''B} \qquad \text{(Equation 4)}$$
$$P^\sigma = \frac{\sigma_Y}{\sigma_Y^{crit}} = \frac{\overline{O'D}}{O'E}$$

Next, an example of FAD-based analysis is shown for two ductile iron pipelines of different dimensions using tensile propeties from the literature and fracture toughness from ASME B31.12. The dimensions of the "vintage" ductile iron pipes that will be assessed are as follows:

Table 7 Dimensiones of distribution ductile iron pipes used in the assessment

Pipe Information Table		
Pipe	Outer Diameter (OD)	Wall Thickness (t)
Pipe 1	118 mm	8.5 mm
Pipe 2	98 mm	4.76 mm

The maximum working pressure for these pipes is 16 bar. Table 8 shows the material properties used in the assessment, which were taken from material data specifications and from minimum required values in ASME B31.12.

Table 8 Material properties used for FAD assessment. * Minimum required value in ASME B31.12

Material properties	Symbol	Unit	Value
Minimum tensile strength	Rm	MPa	420
Minimum yield strength	Rp0.2	MPa	300
Minimum elongation at break	A	(%)	10
Maximum hardness	HBW	HBW	230
Fracture toughness	KIH	MPa $(m)^{1/2}$	55*

Figure 22 and Figure 23 illustrate the minimum required toughness as a function of the normalized crack length (*a/t*) for Pipe 1 and Pipe 2 under internal pressure of 16 bar. This analysis includes both scenarios with and without the consideration of secondary stresses. For the purposes of this evaluation, using a constant residual stress profile equivalent to the yield stress of the material is as a conservative approach and should give conservative estimations of safety margins and critical pressure values.

At the specified operating pressure and considering the material and pipe dimensions in Table 7 and Table 8, the safety margins remain robust, even at crack length to pipe thickness (*a/t*) ratios greater than 0.5. For instance, from Figure 23, it is shown that if the material exhibits a critical stress intensity factor (K_{IH}) at the hydrogen environment that is equal or greater than 40 MPa(m)$^{1/2}$, Pipe 1 and Pipe 2 are capable of accommodating cracks with a normalized length (*a/t*) of up to 0.5 and 0.65, respectively.

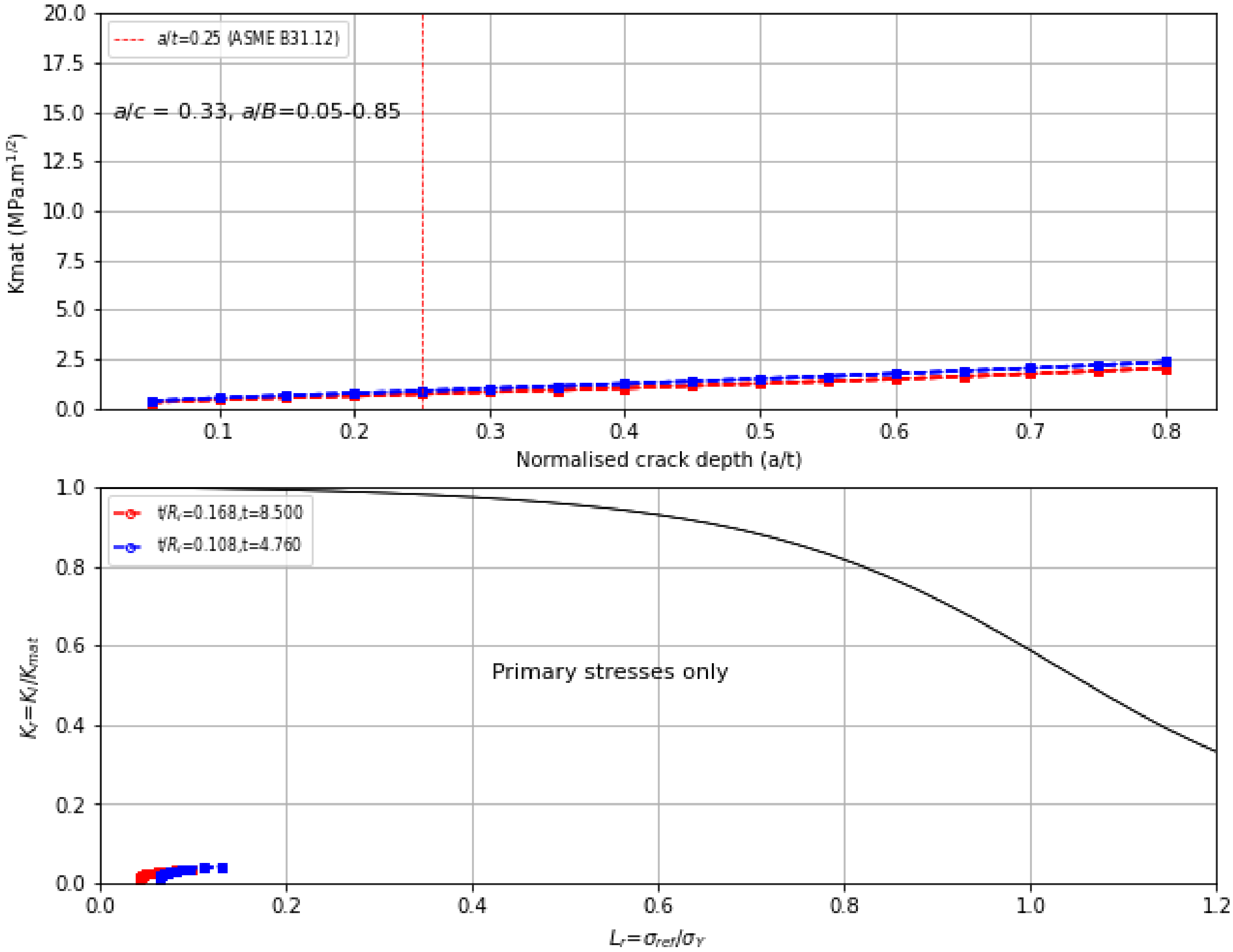


Figure 22 Minimum required fracture toughness and failure assessment for the case for Pipe 1 (red) and Pipe 2 (blue) for semi-elliptical surface cracks of different size (a/t) (a/c=0.33). Operating pressure 16 bar . No residual stresses considered.

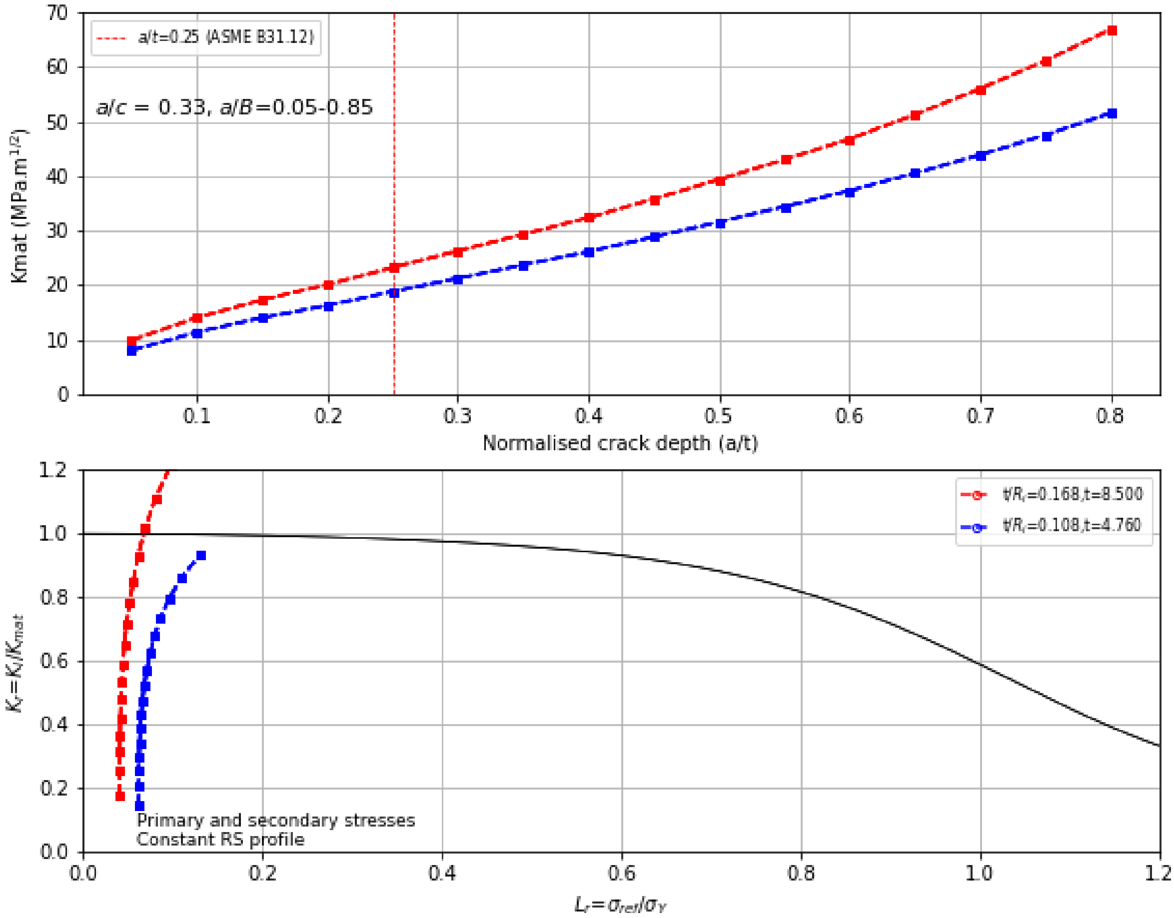


Figure 23 Minimum required fracture toughness and failure assessment for the case for Pipe 1 (red) and Pipe 2 (blue) for semi-elliptical surface cracks of different size (a/t) and aspect ratio a/c=0.33. Operating pressure 16 bar. Secondary stresses considered affecting only crack driving force sR=sY (yield strength.

Figure 24 Critical pressure and reserve factors ($\boldsymbol{P^L}$) on load for Pipe 1 (red) and Pipe 2 (blue) for semi-elliptical surface cracks of different size (a/t), (a/c=0.33). Operating pressure 16 bar . Secondary stresses considered affecting only crack driving force sR=sY (yield strength). shows the critical pressure to failure and reserve factor (P^L) for both Pipe 1 and Pipe 2 for the material properties and dimensions in Table 6 and 7 when considering constant residual stress across different normalized crack depths (a/t) is shown. The plots illustrate how these values change as the crack depth increases for each pipe, providing insight into the sensitiviy of crack size on critical pressure and margins of safety. It is observed that high critical pressures are obtained in comparison with operating pressure (>15x) as well as significant margins of safety (>15x) for *a/t*=0.25 (a/c=0.33) when K_{IH}=55 MPa(m)$^{1/2}$.

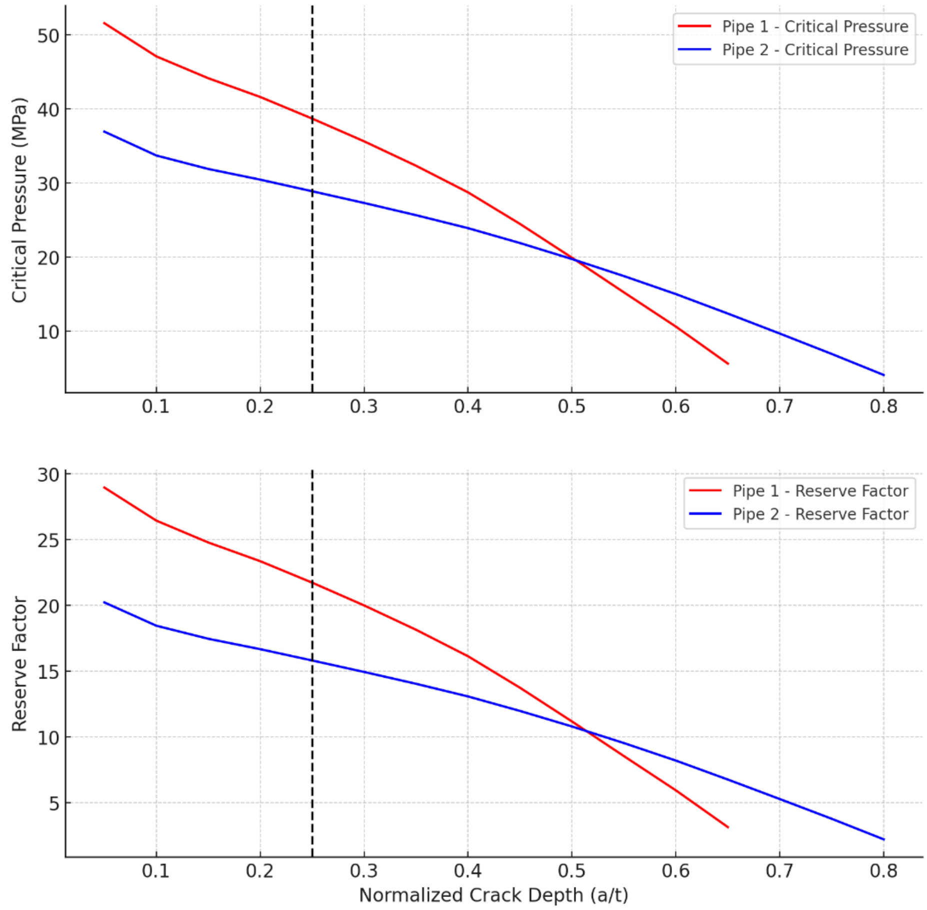


Figure 24 Critical pressure and reserve factors (P^L) on load for Pipe 1 (red) and Pipe 2 (blue) for semi-elliptical surface cracks of different size (a/t), (a/c=0.33). Operating pressure 16 bar . Secondary stresses considered affecting only crack driving force σ_R=σ_Y (yield strength).

3. REVIEW OF PROJECTS AND TESTS PERFORMED

This study aims at identifying and summarizing results of previous research, avoiding duplicates, and looking for new study fields to be investigated. This section detailed the various projects and studies carried out on non-steel metallic materials with hydrogen but also includes work performed on steel materials.

3.1 Previous and ongoing projects

3.1.1 Sedigas

This project studied the possible effect of the joint conduction of natural gas/hydrogen on the mechanical resistance of gas channeling pipes made of ductile iron [34]. Pipes were examined that had already been operated with town gas and those that had only been operated with natural gas.

During this work, testing was performed on five different tubes of geometry shown in Figure 25. Tubes were made of vintage materials, ductile nodular cast iron (UNE EN 969 standard), installed between 1969 and 1987 in different Spanish cities.

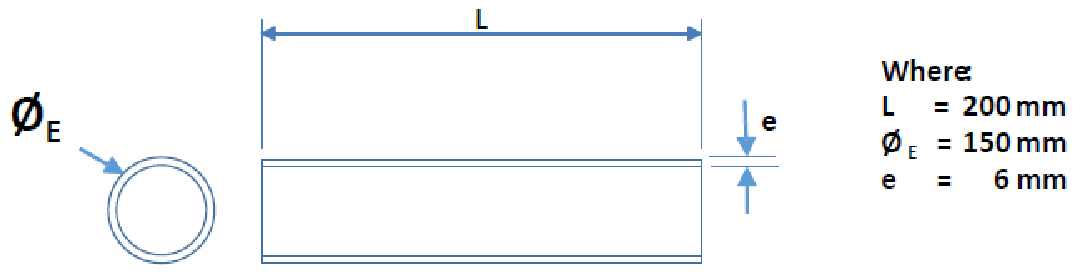


Figure 25 Dimensional sketch with nominal dimensions of the pipelines analysed

Each tube was instrumented with uniaxial strain gauges of 350 ohms to enable the measurement and recording of the tensile strains derived from the bending stress imposed on the tubes during the mechanical tests.

A test station was built to expose the pipes to different concentrations of hydrogen gas (5, 10, 50 and 100 %) at 150 mmbar during both 1 month and 6 months. The pipes were sealed at each end with caps and the hydrogen blend was injected inside them. After exposure to hydrogen, the following tests were performed to the pipes:

- ✓ Flexural mechanical characterization
  - 3 point bending test at a constant displacement load of 10 mm/min and data sampling rate of 50 Hz.
  - Failure (fracture) mode by local buckling of the material in the area of application of the load located on the upper face of the pipeline, accompanied by cracking of the material at the same point in the transverse direction.
  - All tested tubes show an ultimate resistance to bending of 25.2 kNm and a maximum elastic moment of around 20 kNm which would be higher than the minimum detailed in the UNE EN 969 standard for new 150 mm tubes in the state of manufacture.
  - The ratio between the maximum moment and the moment for the end deformation of the elastic branch is practically always equal to or greater than 1.4.

- ✓ Analysis of hydrogen absorption
  - Conducted with LECO technique, using a LECO model TCH 600.
  - The levels of hydrogen detected in all the samples (1.9-5.8 ppm) analyzed in this study exceeded the theoretical initial value of 2 ppm for ductile cast iron. However, the initial value for the pipelines in the manufacturing state was unknown.

- ✓ Visual inspection of the fracture face using SEM microscope

- High levels of plastic deformation in all the fractures were observed, which has been reflected in the form of the generation of "dimple" hemispherical cavities around the nodular graphite particles.
- All the samples analyzed have shown a purely ductile type of fracture, without showing signs of brittle fracture typical of hydrogen embrittlement.

✓ Determination of hardness

- Brinell test according to UNE EN ISO 6506 1 and ASTM E 10 standards and Vickers test according to ASTM E 92 and ASTM E 384 standards were performed.
- The HB hardness analysis showed no differences between the 5 series of cast irons, with hardness values within the specifications required by the UNI EN 969 standard and always below the established limit value of 250 HB.

In view of the results obtained, the pipes exposed to different concentrations of hydrogen during two different periods of permanence, 1 and 6 months, did not show differences in fracture behavior, mechanical resistance, deformation capacity and hardness that could reflect an affectation by hydrogen embrittlement mechanisms. The mechanical parameters were all compliant with manufacturing standard (UNE-EN 969) and the brittle fracture surfaces also showed no abnormalities.

3.1.2 H2VorOrt

This project is a multi-year planning process for the transformation of the German gas distribution networks to climate neutrality with aim of completing an investment-ready plan by no later than 2025.

91 % of the German gas distribution network have been studied, representing 506,584 km of pipelines (grid connection lines and main lines) out of 554,500 km (total length of the German gas distribution network). Analyse of the German pipeline network materials have shown that 95.9 % of the pipelines are made of H_2-compatible steel or plastic. Gray cast iron, representing 0.2%, is not ready for H2. Ductile cast iron, representing 1.5%, are in working progress. No final statement regarding H2 suitability can be made at present; however, initial tests in the UK seem to indicate positive results. The remaining 2.4% are unknown materials.

3.1.3 HyDeploy

HyDeploy [35] is an English project to establish the feasibility of supplementing natural gas in the GB distribution network with 20% vol. H_2. The following tests were performed:

✓ Mechanical squeeze off tests of PE 80 pipes after soaking them in 100 % H_2 for 6 weeks @2bar.

- After soaking, test samples were held for 45 mins in an ice bath at 0 °C, removed for squeeze-off, re-cooled for 1 h and rounded using an approved tool.

- The sample was hydrostatic tested at 9.7 bar (4.5 MPa hoop stress) at 80 °C for 165 hrs. A second pipe without hydrogen soaking was also tested.
- Samples were inspected using DPI for internal stress crack onset, and X-Ray for mid-wall voids.
- No detrimental effects due to hydrogen were observed in the squeeze-off trials.

✓ Tensile tests were conducted on steel, brass, copper, lead and cast-iron materials after soaking in 100% CH_4, 20% Vol. H_2 in CH_4 and 100 % H_2 in vessels at 2 barg for 6 weeks.

- For Copper, Brass and Lead materials, the test results showed no significant change in mechanical properties after soaking the materials in various test gases.
- For cast iron, the results showed a drop in UTS and yield strength after soaking the material in 100% hydrogen. However, scatter was observed to be caused by microstructural differences like grain size, graphite flake size and shrinkage. Also, as the specimens were machined from cast block, differential thermal cooling was considered to affect the results.
- The results also showed that a blend of 20% Vol. H_2 did not affect the performance of cast iron material while 100% of H_2 had a possible effect.

✓ Tensile testing and charpy impact tests were conducted on steel, brass and cast-iron materials following soaking in pure hydrogen vessels at 8 barg for different lengths of time (1 week, 4 weeks and 5 weeks).

- Although the fracture energies observed from the charpy impact tests showed an increase in scatter with longer hydrogen exposure, it is concluded that the impact energies for the materials were not influenced by the hydrogen charging.
- The tensile testing of hydrogen exposed samples revealed no discernible difference between the un-exposed and soaked condition with the cast iron.
- Fractographic assessment showed that the presence of hydrogen did not change the failure mechanisms of the materials in that ductile microvoid coalescence fracture of the steel and brass was observed, whereas more of a brittle fracture surface was observed for the cast iron.

3.1.4 H2SAREA

Nortegas, the second natural gas distributor in Spain (operating 8,100km of distribution networks), has launched the H2SAREA project on developing advanced technological solutions for the safe distribution of hydrogen in the natural gas network [36].

In this project several compatibility tests were performed. Specimens were extracted from pipes of the Nortegas' grid made of steels of grade B and X42 (base and weld material), ductile iron, copper and non-metallic materials (such as PE100 and PE80 pipes, NBR joint, Carboard joint, elastomeric flat joint, membrane of pressure regulator), and prepared to assess the impact of hydrogen.

The following test were performed:

✓ Absorption tests:

- Specimens (rods of 3 mm diameter and 10 mm length) were exposed to 100% H_2 for 1000h at 16 bar.
- Specimens were kept in liquid nitrogen and the quantity of H_2 absorbed was quantified afterwards. A LECO TCH-600 analyser was used to perform the measurements.

✓ Embrittlement tests:

- SSRT tests were conducted, in accordance with EN ISO 6892-1, in air and after exposure to H_2 at a strain rate lower than 0.0003s-1.
- For each material, at least two tensile test specimens were machined. The first specimen was tested in air and the average necking value, Z_1, was determined. The second specimen, prior testing, was exposed to pure hydrogen atmosphere for 1,000-hour. After exposure the specimen was stored in liquid nitrogen and then low-strain rate tensile test was conducted. The average necking value after exposure, Z_2, was determined.
- Once the values Z_1 and Z_2 were known, the Z_2/Z_1 ratio was calculated, and the following criterion was applied: If $Z_2/Z_1 > 0.85$, the material was considered not to have undergone hydrogen embrittlement.

✓ Hardness test according to UNE-EN ISO 6507-1: 2018 and UNE-EN ISO 9015-2: 2016 standards.

- Metallographic specimens from the base metal and weld metal were prepared according to Tecnalia's internal procedure, which was not specified.
- Specimens were then analysed using optical microscopy, and representative micrographs of the main observations (microstructure, presence of defects, etc.) were taken.
- Microhardness measurements were conducted according to EN ISO 6507-1 for the seamless specimens and according to ISO 9015-2 for the welded specimens.

✓ Compression tests (with fittings) according to UNE EN ISO 815 standard.

- Specimens were tested under a compressive force for 24 hours at 23 °C. compression factor = 25%.
- After removing the compressive load, the residual deformation exhibited by the joints was assessed. For this, the specimen thickness before and after testing was determined using the method described in ISO 23529 standard.

All steels, PE pipes, cardboard joints, elastomeric flat joints and membrane of pressure regulator did not show any sign of damage due to hydrogen exposure. Copper and ductile iron materials tested at 3 bar; did not show evidence of hydrogen embrittlement; however, they showed hydrogen embrittlement at 16bar. NRB joint also showed signs of damage.

Table 9 summaries the main results of the project.

Table 9 H2SAREA results

Material familiy	Material	Type of Test	Exposure Conditions	Results
Carbon Steel Pipe	X42	SSRT Tests	-100% H_2 at 16 bars (1000 h)	No evidence of hydrogen embrittlement observed
Ductile Iron Pipe	-	SSRT Tests	-100% H_2 at 16 bars (1000 h)	Hydrogen embrittlement observed
			-100% H_2 at 3 bars (1000 h)	No hydrogen embrittlement observed
Copper Pipe	-	Evaluation of Joints and Degradations	-100% H_2 at 16 bars (1000 h)	Hydrogen embrittlement observed
			--100% H_2 at 3 bars (1000 h)	No hydrogen embrittlement observed
Polyethylene	Grades used in the network	SSRT Tests	100% H_2 at 16 bars (1000 h)	No evidence of degradation
Elastomeric Joints	Nitrile, Neoprene, Viton, EPDM	Visual inspection, Compression test	100% H_2 and H_2+GN mixtures	Blistering in NBR joints, all other materials show no significant change.

3.1.5 SyWeSt H2

The SyWeSt H2 project "Investigation of Steel Material for Gas Pipelines and Plants for Assessment of their Suitability with Hydrogen" [37] has been conducted by the German long-distance grid operator Open Grid Europe GmbH, which is responsible for the largest long-distance grid in Germany with round 12,000 km, together with the Materials Testing Institute of the University of Stuttgart. In this project fracture mechanical investigations were performed primarily on pipeline steel grades used in Germany comparing the established fracture-mechanical parameters with the criteria specified in ASME B 31.12, drawing up a modified correlation for crack growth.

The tests considered base materials and welded joints. Compact specimens, as shown in Figure 26 were machined with a thickness of 10 mm wherever possible. However, for some specimens, this thickness was reduced (e.g. in the case of an excessively thin wall). In some cases, samples were so small that they could not be tested from a technical point of view which limited the validity of the test results. Prior to testing, an approx. 2 mm fatigue crack was made on the samples following the specifications in ASTM E1820-20.

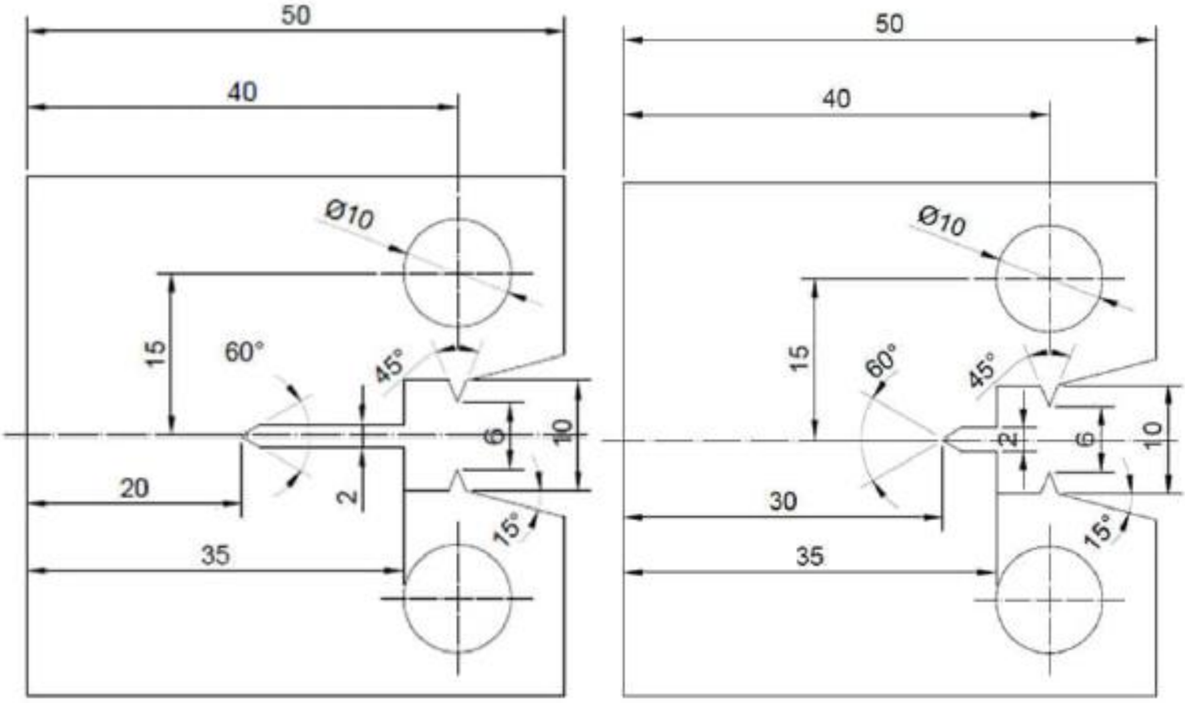


Figure 26 Sample geometry for static tests (left) and cyclical test (right)

Fracture toughness and FCG tests were performed in an autoclave filled with hydrogen at up to 100 bar pressure. Cyclical tests were performed according to ASME E647.

Testing was performed on steel materials as follow:

- L290 NE
- Grade A
- St35
- 15 k (St.35)
- X42
- RR St 43.7
- P355 NH
- L360 NE
- StE 360.7
- L360 NB
- 14 HGS
- TStE 355 N
- WSTE 420
- St53.7
- X56.7
- St60.7
- P 460 NH

- X70
- X80
- GRS550/X80
- L485 (HV high/low)
- L415 (curve)
- P355 NL1 (Valve)
- GJS 400 (Valve)
- C22.3 (Valve)
- GS C25 N (Valve)
- P460 QL1 (Valve)
- StE 320.7
- StE 480.7 TM
- L485
- L360

FCG tests were performed on the majority of samples at a constant hydrogen pressure of pH2 = 100 bar. The test frequency was set to f = 1 Hz and the stress ratio to R = 0.5. FCG tests at R ratio of 0.1 and 0.7 were performed on L360 and L485 steels. During testing, the crack growth rates were established in the range of stress intensities K of approx. 10 to approx. 40 MPa $m^{1/2}$.

The tests carried out confirm the results from the literature and show that the influence of the material structure and the associated different ductility properties have only a very minor effect on crack growth within the scope of the usual variations. The static fracture mechanics tests also performed show that the minimum fracture toughness required by ASME B 31.12 (KIc > 55 MPa m½) is clearly met by both current and older piping materials.

The findings from the study indicate that existing pipeline steel is fundamentally suitable to transport Hydrogen up to 100%. Both fatigue crack growth rate and fracture toughness criteria specified in ASME B31.12 were well met. A small sample of valves tested also indicated their suitability for hydrogen use.

The study also indicates that no impact of hydrogen on crack growth behaviour for a partial pressure pH2 less than equal to 1 bar could be established in the measurement performed.

3.1.6 NATURALHy

NATURALHY was an Integrated Project funded by the European Commission's Sixth Framework Programme for research, technological development and demonstration preparing for the hydrogen economy by using the existing natural gas system.

In this project the mechanical behaviour of the steel grade API 5L X52 and X70 in hydrogen gas was studied. Specifically, fatigue crack growth tests were performed in a mixture of H_2 and CH_4 at a pressure of 65 bar to establish the effect of hydrogen on the fatigue crack behaviour of these steels. The FCG of X52 and X70 in 100% H_2 is shown in Figure 27 and the effect of H_2 obtained is described as follows:

- Hydrogen can have a significant effect on fatigue performance. This includes both the fatigue threshold value and the fatigue crack growth rate. It is strongly dependent on the actual test conditions and the amount of H_2.
- Under simulated in-field conditions (pressurized gas and in-field ΔK values) the performance is far better than under (not realistic) laboratory conditions.
- Under simulated in-field conditions a blend of 75% vol. natural gas and 25% vol. H_2 is allowed for the X70 material, without a degradation of the fatigue performance.
- Under simulated in-field conditions a blend of 50% vol. natural gas and 50% vol. H_2 is allowed for the X52 material, without a degradation of the fatigue performance.
- If 100ppm (or better still 250 to 500 ppm) of oxygen is added in 100% vol. pressurized H_2, the fatigue performance is comparable to that in 100 % vol. natural gas.

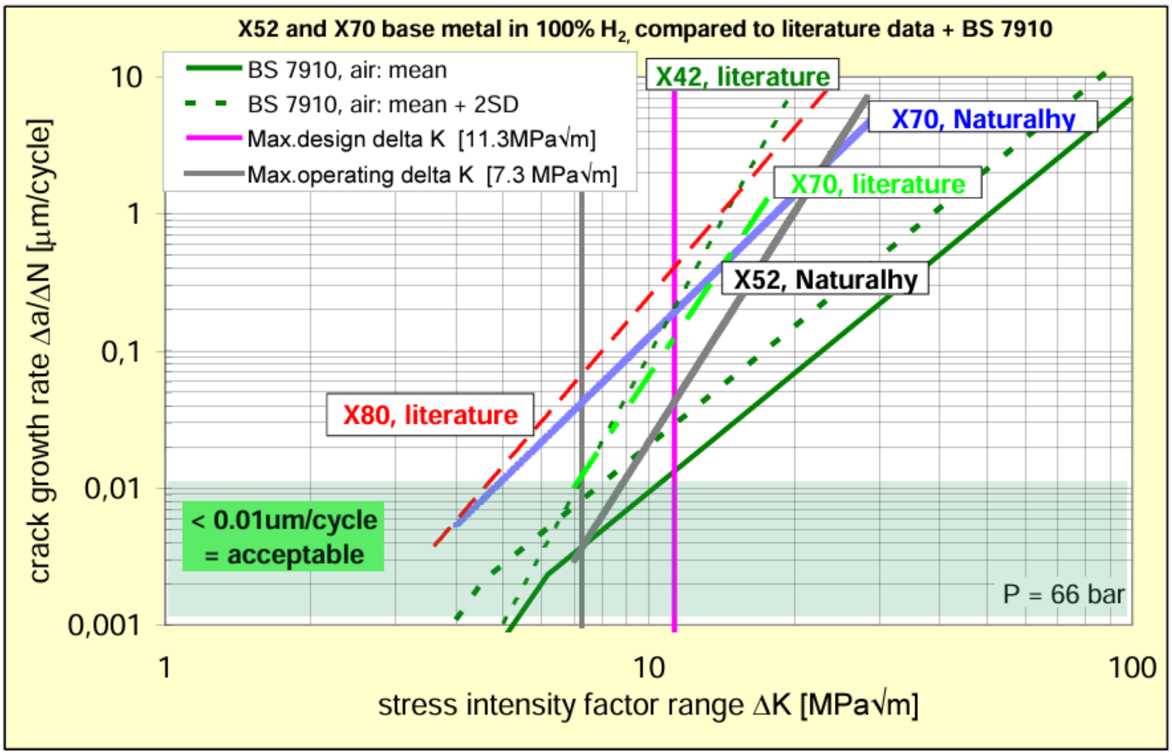


Figure 27 Fatigue crack growth of X52 and X70 base materials in 100% H_2. [38] Reference

3.1.7 HIGGS

HIGGS project received funding from the Fuel Cells and Hydrogen 2 and was a Europe Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No. 875091. The Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research. The main goal of the HIGGS project "Hydrogen in Gas Grids - A systematic validation approach at various admixture levels into high-pressure grids" was to facilitate the way to decarbonization of the gas grid and its usage, by covering the gaps of knowledge of the impact that high levels of hydrogen could have on the gas infrastructure at transmission level, its component and its management. The initiative, funded by the European Union, seeks to advance the integration of hydrogen into gas networks, thus contributing to the climate objectives of the European Green Pact. Gas tightness tests on valves nodes, assessment of the hydrogen sensitivity on carbon steels (API 5L X42, API 5L X52, API 5LX60 and API 5LX70) and embrittlement/degradation of critical components and equipments of the transmission gas infrastructure were evaluated for several testing campaigns from low H_2 content (20% mol H_2) to 100% H_2 and in presence of impurities (CO_2 and H_2S) at a pressure of 80 bar (see Table 10).

Table 10 HIGGS experimental campaigns performed in the R&D facility

Campaign No.	Gas Composition	Type of test	Pressure (bar)	Test duration (h)
1	20 mol % H_2 80 mol % CH_4	• Gas tightness • Gas separation • Hydrogen embrittlement: constant displacement tests • Inspection of equipment & valves	80	2200-3000
2	20 mol % H_2 4 mol % CO_2 11 ppmv H_2S 76 mol % CH_4	• Gas tightness • Hydrogen embrittlement: constant displacement tests • Inspection of equipment & valves		
3	20 mol % H_2 4 mol % CO_2 11 ppmv H_2S 66 mol % CH_4	• Gas tightness • Hydrogen embrittlement: constant displacement tests • Inspection of equipment & valves		
4	100 mol % H_2	• Gas tightness • Hydrogen embrittlement: constant displacement tests and SSRT tests • Inspection of equipment, valves and welded sections of API 5L pipes		

To evaluate the compatibility with hydrogen of the API 5L carbon steel pipes, the following constant displacement tests were conducted

- C-ring tests on smooth and notch base metal specimens according to ASTM G38;
- CT-WOL tests on base metal and weld specimens according to ASTM E1681;
- 4 point bend tests on base metal and weld specimens according to ASTM G39

In combination with the static load tests, the SSR tensile test method was used as a valuable screening method. SSRT were carried out on notched specimens in 100mol% H2, at 80 bar, according to ISO7539-7 and ASTM G142 standards.

The results showed that:

- Constant displacement C-ring and 4 point bend tests on steel specimen grades X42, X52, X60 and X70 did not show cracking when exposed to the different hydrogen gas compositions, at 80 bar pressure and test duration up to 3000 hours. No crack propagation was also noticed for the CT-WOL specimens, under the same experimental conditions
- SSR tests carried out for X60 and X70 steels in 100 mol% H_2, at 80 bar pressure showed that the yield values of the notched tensile strength ratio indicate a low sensitivity to hydrogen embrittlement. However, a significant loss of ductility in the notch specimens, mainly manifested in the loss in the RA values as well as in the morphology of the fracture surface was observed.
- It was also observed that depending on the loading conditions -static or rising load- and the presence or absence of notches in the material, the degree of embrittlement was different. In general, rising load tests produced higher embrittlement indexes in the material.
- Besides and for the different gas compositions, the gas tightness tests of representative valves of the natural gas (NG) grid were evaluated to identify possible leakages when operated with hydrogen. It was observed that all the testing valves remained tight for the duration of the test regardless the hydrogen content in the pipe, with just minor hydrogen losses due to hydrogen leakage through the body of the valves. The results of the inspection carried out in the valves, pressure regulator, cartridge filter and turbine gas meter components after their exposure to the different hydrogen mixtures, showed no apparent damage on the different parts examined.

It was concluded that the results obtained both in the constant displacement tests carried out in the R&D testing platform and in the SSRT, were indicative of low susceptibility to hydrogen embrittlement for the four API 5L steels grades in the tests conditions established in HIGGS, suggesting they are suitable for high-pressure hydrogen service [39]

3.1.8 20HyGrid

20HyGrid is a Romanian project, carried out by the DSO Delgaz Grid, which aim was to analyze the compatibility and behaviour of the materials and elements present in the current Romanian natural gas distribution grid and in its installations, under a mixture of natural gas and 20 % volume of hydrogen.

Testing was conducted on more than 260 devices, presented in the natural gas distribution network and in home installations. The results were encouraging and showed that the use of mixture (20 % vol. hydrogen and 80 % vol. natural gas), at pressures below 16 bar, in existing customer distribution networks, installations, and appliances, without requiring any modification, is technically possible and without additional risks compared to the use of natural gas.

3.1.9 HYREADY

This project was supported by GERG and HIPS-NET, the overall goal was to formulate a set of practical guidelines that enable grid managers to assess the consequences of and mitigating measures for adding hydrogen up to a 30% blend or pure hydrogen. The project focused on all aspects of the gas infrastructure, starting from high-pressure gas networks and low-pressure distribution networks to user equipment and compressors, storage and injection stations. The consortium consists of over 20 international partners from both Europe, Asia, Canada and the US.

No further information about testing and test condition were available.

3.1.10 MultiHy

MultiHy "Multimegawatt high-temperature electrolyser to generate green hydrogen for production of high-quality biofuels" project (Grant agreement No. 875123) is currently active and aims to install, integrate and operate the world's first high-temperature electrolyser (HTE) system in multi-megawatt-scale (~2.4 MW), at a biofuels refinery in Rotterdam (NL) to produce hydrogen (≥ 60 kg/h) for the refinery's processes. The goal is to demonstrate the technological and industrial leadership of the EU in Solid Oxide Electrolyser Cell (SOEC) technology. With its rated electrical connection of ~3.5 MWel,AC,BOL, electrical rated nominal power of ~2.6 MWel,AC and a hydrogen production rate ≥ 670 Nm^3/h. This leads to GHG emission reductions of ~8,000 tonnes during the planned minimum HTE operation time (16,000 h). MULTIPLHY's electrical efficiency (85 %el,LHV) will be at least 20 % higher than efficiencies of low temperature electrolysers, enabling the cutting of operational costs and the reduction of the connected load at the refinery and hence the impact on the local power grid.

3.1.11 H2GAR

H2GAR (H_2 Gas Assets Readiness) project has for aim to contributes to the development of new standards and technologies on H_2 gas readiness for the transport of H_2. This will be performed by identifying any technological and regulatory gaps for both existing and new pipelines, defining a common approach among members regarding two scenarios of H_2 levels (10% and 100%) assessing the impact on gas turbines and compressors, identifying the most cost effective solutions to separate hydrogen from H_2/NG mixtures but also the issues concerning quantity/quality metering of H_2/NG mixtures. The work will also assess every aspect of the safety issues relating to the transport of hydrogen.

3.1.12 SHIMMER

SHIMMER (Safe Hydrogen Injection Modelling and Management for European gas network Resilience) is a project supported by the Clean Hydrogen Partnership and its members, under Grant Agreement No. 101111888, which runs parallel in time to CANDHy.

SHIMMER will evaluate the materials and components present in the current natural gas transmission and distribution grid, by reviewing the existing procedures on qualification and testing to assess their hydrogen readiness. This includes conducting a comprehensive mapping of gas infrastructure across Europe to identify strategies for effectively managing a multi-gas network and ensuring the safe integration of hydrogen into natural gas infrastructure. The project will also focus on defining methods, tools, and technologies for multi-gas network management and quality tracking, including simulation, prediction, and safe management of transients in the context of widespread hydrogen injection. Additionally, SHIMMER aims to propose best practice guidelines for handling the safety of hydrogen in the natural gas infrastructure while managing associated risks.

This project started recently, and no results have been obtained or published yet. The project will be completed in 2025.

3.1.13 PilgrHYm

PilgrHYm project is supported by the Clean Hydrogen Partnerhsip and its members under Grant Agreement No. 101137592. This project, currently active, is a pre-normative research on integrity assessment protocols of gas pipes repurposed to hydrogen and mitigation guidelines. The main objectives of the PilgrHYm are to provide quantified data on more than 70% of the European transmission steel grid, to refine exiting standards and codes by reducing over conservatism and to provide a common understanding on compatibility of networks within the EU for hydrogen transmission. This will be obtained by conducting an extensive testing program on small-scale laboratory specimens (base metal and welds), which will include 8 base materials, 2 types of welds, and 2 heat-affected zones representative of the EU gas grids. These specimens will be selected after a thorough review by Transmission System Operators (TSOs) to address safety concerns, the lack of regulations, codes, and standards, as well as research gaps related to the compatibility of current pipelines with hydrogen. PilgrHYm aims to develop a pre-normative framework to support the creation of a European standard for hydrogen transportation through pipelines, significantly contributing to the decarbonization of European industry and the reduction of carbon emissions. This project is funded by EU contributions.

3.1.14 FutureGrid

FutureGrid is a National Gas initiative designed to support the UK's transition from natural gas to hydrogen, with the aim of achieving net-zero emissions. The project involves a high-pressure test facility at DNV Spadeadam, where a microtransmission network simulates the National Transmission System (NTS) to test the effects of hydrogen on gas pipelines and assets. Phase 1 focuses on testing various hydrogen blends (from 2% to 100%) with gas transmission assets to ensure the NTS can safely transport hydrogen. These tests are essential to develop safety standards and provide design guidance for future national hydrogen transmission networks.

In addition to technical advances, FutureGrid emphasises the importance of tracking and reporting the value generated by innovation projects. During the RIIO-1 period (2013-2021), National Gas identified

£89m in value from innovation projects, which included financial, environmental and operational benefits. This success is being built upon in the current RIIO-2 period (ending March 2026), where efforts are focused on demonstrating the NTS's ability to transport hydrogen and other net-zero emission gases.

Collaboration and knowledge sharing are integral to FutureGrid's strategy, with National Gas participating in working groups, conferences and stakeholder sessions to exchange ideas and access further research and development. A strong culture of innovation is fostered within the organisation, ensuring that innovations are effectively integrated into core operations. FutureGrid also includes phases to test demixing technologies and gas compressors, for a wider transition to a hydrogen-based energy system in the UK.

3.1.15 Summary of completed project results

Most of the studies have been performed on steel materials and the following tests were conducted:

- ✓ FT (ASTM E1820)
- ✓ K_{IH} (CT-WOL – ASTM E1681)
- ✓ SSR (ASTM G142/ISO 7539-7)
- ✓ FCG (ASTM E647)
- ✓ C-ring (ASTM G38)
- ✓ 4 point-bend (ASTM G39).

The project results reported on non-steel metallic materials are summarized in Table 11.

Table 11 Non-steel metallic materials findings

Projects	Materials	Tests	Specimen	H2 content (%)	Pressure	Results
Sedigas	Ductile cast iron (vintage)	3-point bend	Tube (150-170mm long)	5, 10, 50 and 100	150 mmbar	Resistance to bending and elastic moment higher than the minimum in UNE ISO 696.
H2VorOrt	Steels, gray cast iron, ductile cast iron					Steels compatible for H2 Ductile cast iron – initial trials appear to

						be yielding positive results Gray cast iron – Not ready for H2
HyDeploy	Brass, copper, lead and cast iron Brass and cast iron	Tensile Tensile		20-100 100	2 barg 8 barg	No effect of H2 on brass, copper and lead. For cast iron, no effect with 20% but with 100% No effect of H2 No difference between soaked and unsoaked specimens
H2SEARA	Ductile iron and Copper	Absorption SSRT (EN ISO 6892-1) at 0.0003s-1	Tensile	100	3 and 16 bar	Both materials showed effect of hydrogen embrittlement at 16 bar. No hydrogen embrittlement observed at 3 bar

The study performed in cast iron by Sedigas showed that mechanical properties comply with manufacturing standard. Tensile tests of cast iron material (after exposure in H_2 at 2 bar) performed in the HyDeploy project reported that test in 20% H_2 has no effect while test in 100% H_2 has potential effect. Furthermore, findings showed that exposed cast iron specimens (after soaking in pure hydrogen at 8 bar), revealed no difference compared to the unsoaked specimens. HyDeploy work also showed that brass, copper and lead materials have no significant change in mechanical properties when tested in hydrogen. H2VorOrt project states that gray cast iron is not ready for H_2 but ductile cast iron is in working progress. SSRT on cast iron and copper materials were conducted in the H2SAREA project, prior testing the specimens were soaked in 100% hydrogen and then tested at 3 and 16 bar. Both materials revealed hydrogen embrittlement at 16 bar but nothing at 3 bar.

Tests performed on non-steel metallic materials are limited to qualitative tests (i.e. screening tests) providing information on the behaviour of materials in H_2 gas but not providing data that allow for a quantitative evaluation of the performance of structural metals in service. For this, fracture mechanics and fatigue tests would be needed.

3.2 Literature research on testing of non-steel metallic materials

Birkitt et al [40] study the effect of hydrogen on cast iron, copper, yellow brass, copper specimens soldered with lead free solder and copper specimens soldered with lead solder within the UK gas distribution network. After soaking the materials for 6 weeks in a blended gas composed of 23 vol% H_2 in CH4 (UK standard G222) at 2 bar, tensile testing to BS EN ISO 6892-1 [41] method A2 at the crosshead speed of 0.01mms-1 (corresponding to a strain rate of 0.000253 s-1) were performed. For all tested materials no detrimental effect on their mechanical properties was observed and the materials were therefore found to be acceptable for use within a blended H_2/NG gas feed up to 20vol% H_2 and 2 bar operating pressure. Tensile test data for lead-free and lead based solder samples are shown in Figure 29.

Material (as-manufactured)	Lap Shear Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation at Break (mm)
Lead-free solder control	–	240 SD 1	13 SD 0.7
Lead-free solder in H_2	19.4 SD 0.6	238 SD 0.9	8 SD 5.1
Lead-free solder in CH_4	19.8 SD 0.4	237 SD 3	10 SD 4.2
Lead-free solder in G222	15.9 SD 3.9	237 SD 4	8 SD 5.6

Material (as-manufactured)	Lap Shear Strength (MPa)[a]	Elongation at Break (mm)[a]
Lead solder control	10.4 SD 3.0	1.1 SD 0.2
Lead solder in H_2	8.4 SD 3.8	0.9 SD 0.5
Lead solder in CH_4	8.7 SD 6.1	1.2 SD 0.5
Lead solder in G222	12.9 SD 4.7	1.2 SD 0.5

[a] *Failure in the lead soldered joint rather than the parent metal.*

Figure 28 Tensile test data for lead-free (left) and lead based (right) solder samples after

Tensile test on cast iron materials was performed by Matsuo [42] and Matsuno [43]. Specimens as shown in Figure 29 were first charged in H_2 between 24h and 168h prior to be tested in air at ambient temperature at cross head speeds of 0.02 mm/min, 1 mm/min and 50 mm/min. The stress-strain curves obtained are shown in Figure 30a and revealed that the strength properties were slightly affected by hydrogen; however, the ductility was severely deteriorated by hydrogen. Similar results were observed on tensile tests (SSRT) performed at a strain rate of 10^{-4} s^{-1} (Figure 30b) [44].

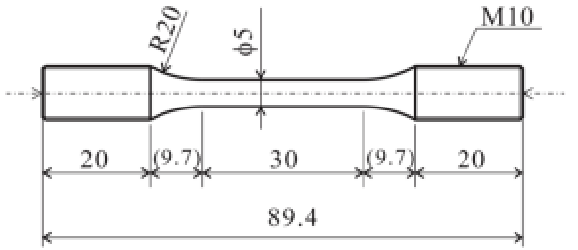


Figure 29 Shape and dimension of tensile specimen in mm [42]

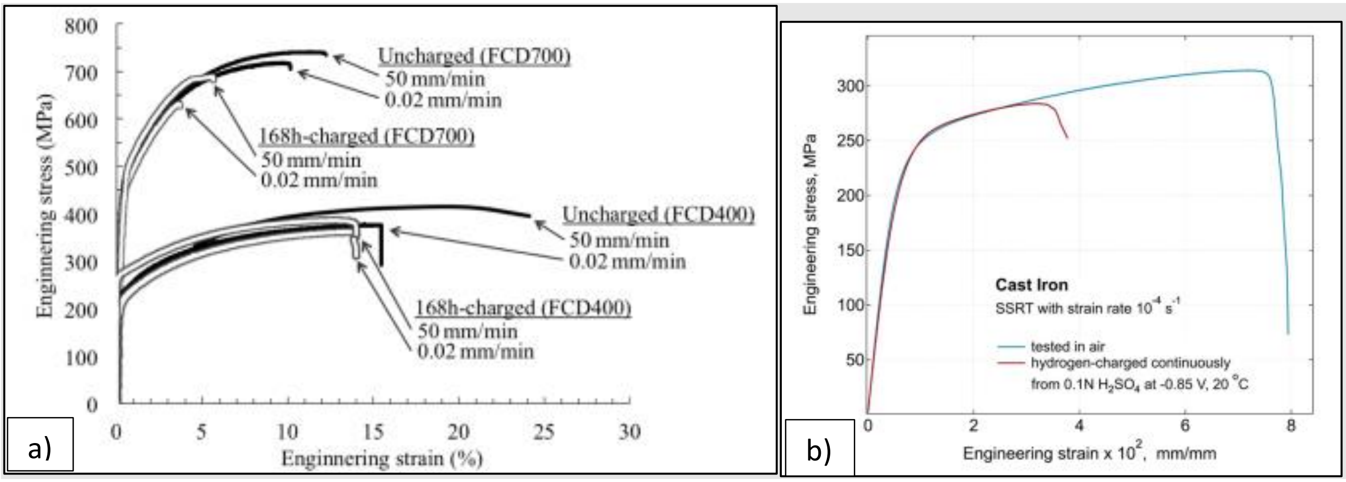


Figure 30 Stress-strain curve of cast iron materials: a) [42] and b)-

Matsunaga et al. [45] investigate the effect of external and internal hydrogen on ductile cast iron. Fatigue crack growth test in 100% hydrogen according to ASTM E647 [13] were performed on test specimens as shown in Figure 31. Specimens were tested in 70 bar H_2 using ΔK-increasing at frequency, f = 0.01 up to 5 Hz. Tests were also performed using the same condition on hydrogen-charged specimens in 100 MPa H_2 at 85˚C for 200h. FCG da/dN curves obtained are showed in Figure 32 and revealed an acceleration of the FCG due to hydrogen.

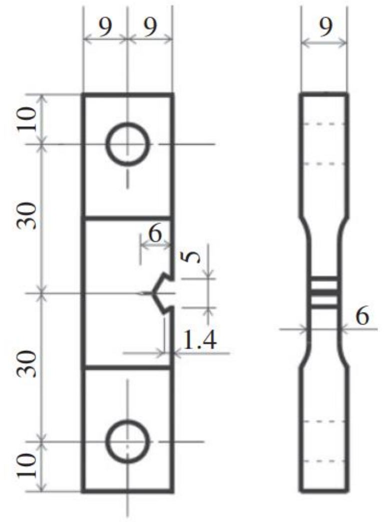


Figure 31 Specimen shape and dimensions (in mm) [45]

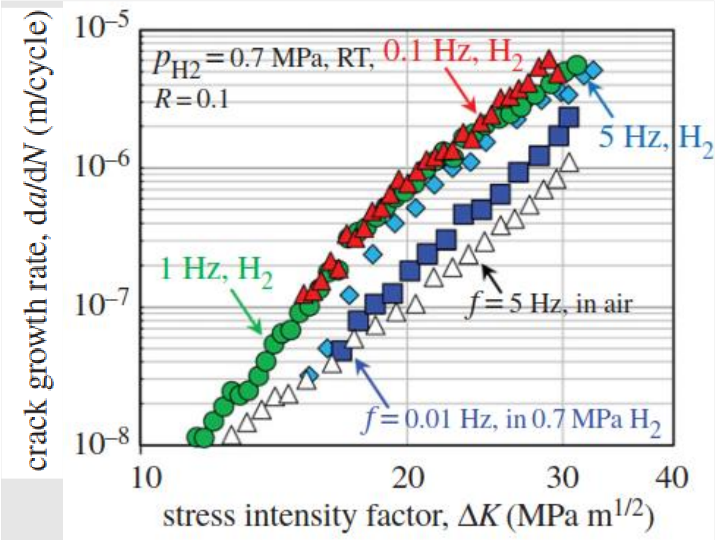


Figure 32 Relationship between crack growth rate, da/dN, and stress intensity factor range, ΔK, obtained by ΔK increasing tests [45]

There are opposite opinions on the suitability of cast iron in H_2 with standards and studies reporting that this material is not suitable for 100% H_2 gas [2], [35], [36] and [46] while others studies reported that H_2 gas (up to 20 %), is suitable [35] [40]. A study performed on a natural gas network in Holland even revealed that nodular cast iron is suitable for 100% H_2 [47].

The reported hydrogen effect on copper properties during tensile tests vary and are not entirely consistent. Vennett and Ansell [48] reported as much as 16% loss in ultimate strength in material tested (smooth and notched specimens) at 69 MPa hydrogen gas at constant crosshead displacement of 8.5 x 10-3 mm/s. However, the study conducted by Barthélémy H. [49] reported that several sources presented results from a common set of tests in which smooth and notched specimens of oxygen-free high conductivity copper experienced no losses of strength or ductility in hydrogen pressure up to 69 MPa. In the former study, Vennett and Ansell [48] observed inclusions in the oxygen free copper used in their study, perhaps indicating that these hydrogen effects could be attributed to oxides or other second phase inclusions. Walter and Chandler [50] attempted to measure threshold stress intensity factors for oxygen free copper at room temperature and -129˚C in 34.5 MPa H_2 gas. They estimated K_{IC} (plane strain fracture toughness) to be in the range of 16 to 20 MPa $m^{1/2}$. Barthélémy H. also reported that hydrogen had no effect in disk rupture testing and no fatigue data for copper are available in the literature. The latter was also stated in the SANDIA report [33].For copper and its alloys it appears that more testing is necessary to completely understand the effects hydrogen gas, particularly with regard to pressure.

Regarding Aluminium and its alloys, Barthélémy H. reported that these materials have demonstrated high resistance to HE in gaseous environments. Tensile test on smooth and notched specimens performed on pure and high purity aluminium in high-pressure (up to 52 MPa) gaseous H_2 by Vennett and Ansell [48] showed no H2 effects. San Marchi C. and Somerday B. [51] stated that tensile tests of aluminium specimens (pure and highly purity aluminium) tested at 34.5 MPa and 52 MPa hydrogen gas were unaffected by hydrogen. Other tensile tests performed by Ordin [52]at 68.9 MPa H_2 also showed that alumimium alloys have good resistance to H_2. In the literature, the results of tensile tests are available but mainly for high pressure (> 16 bar) and no results from fracture mechanics and fatigue tests are reported.

The H_2 effect on nickel materials can be significant. Tensile testing on smooth nickel specimens, with or without hydrogen-precharged showed reduction of tensile ductility [33]. Chene and Brass [53]explored the effect of strain rate for Inconel 600 using cathodic precharging and observed little effect of strain rate at room temperature for strain rate less than about 10^{-3} s^{-1}, in contrast to another study where tensile elongation was found to be decrease with strain rate to lower strain rates [54]. Reduction of strength and ductility of notched specimens was also observed and Chene and Brass found that the decrease of tensile elongation for notched Inconel 600 approximately double compared to smooth tensile specimens. Regarding fracture mechanics, SANDIA [33] reported that the fracture toughness is affected by H_2. The same is reported by SANDIA for the fatigue tests of nickel materials at 34.5 MPa showing a significant reduction in number of cycles to failure. Disk rupture test also show that nickel-based alloys are extremely sensitive to hydrogen-assisted fracture in gaseous environments [33].

Regarding brass alloys, Panagopoulos, et al [55] studied the effect of hydrogen charging on the mechanical properties of 70%Cu-30%Zn α-brass alloy. It was observed that hydrogen charging reduced significantly the ductility and slightly the ultimate tensile strength of the brass alloy. This ductility reduction increased with increasing the duration of the charging time (Figure 33). A brittle intergranular fracture was observed in the outer periphery of charged α -brass fracture surface.

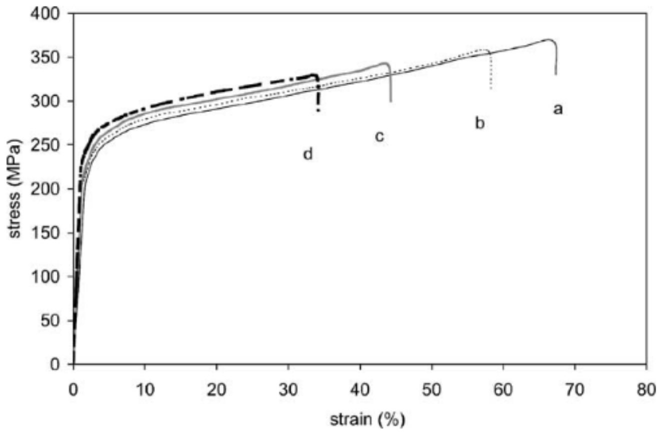


Figure 33 Stress-strain curves for uncharged brass specimen (a) and for brass specimens charged with 25 mA cm-2 for 6 h(b), 20h (c) and 40 h (d) charging time

Briottet L. and Riccetti B. [56] evaluated the susceptibility of two brass alloys CW617N (58%Cu, 2%Pb, balance Zn) and CW614N (58%Cu, 3%Pb, balance Zn) in NG+H_2 mixtures up to 5 bar pressure, under SSR tensile loading on smooth and notched specimens. Obtained results for the CW617N alloy tested at 20%H_2 indicated no significant hydrogen effect, see Figure 34.

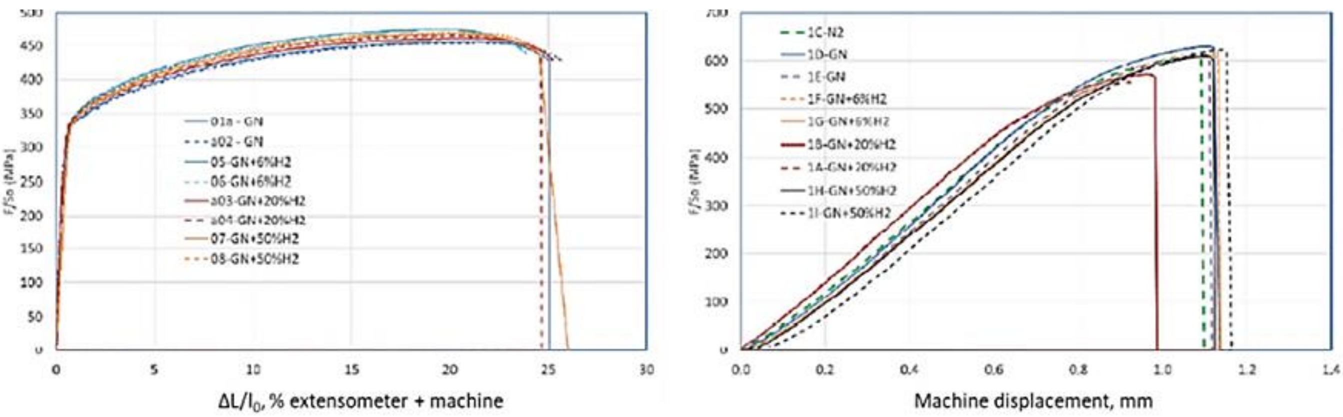


Figure 34 Tensile curves results for the CW617N brass alloy on smooth specimens (left) and notched specimens (right)

Hardly any references have been found concerning the compatibility of lead with hydrogen. Only the studied performed by Birkitt et al [40], see above was found.

4. CONCLUSION

There are currently numerous standards that address at least in part the problem of hydrogen gas under pressure in contact with metallic materials. The standards AMSE B31.12, ASME VIII Div. 3, ISO 11114-4, ANSI/CSA CHMC 1 and EIGA IGC Doc 121/14 and the EPRG report address this problem by proposing standardized tests for the characterization of the susceptibility of metals to an environment rich in hydrogen gas. Metals that comply with the parameters imposed by these standards are therefore suitable for use in contact with hydrogen gas.

The most common tests to evaluate the behaviour of materials are K_{IH} test, Fracture toughness, SSR, FCGR, fatigue life, disk rupture and C-ring tests. SSR, disk rupture and C-ring tests are screening tests allowing to evaluate the susceptibility to hydrogen embrittlement by comparing tests performed in hydrogen to tests performed in inert environments while the other tests provide quantitative measurements which could be used in the structural life assessment of materials.

Current standards mainly cover steel materials, most commonly used in hydrogen gas distribution pipelines and well studied, and little information regarding testing requirements is provided on non-steel metallic materials. Standards should be updated to provide information to assess the compatibility of non-steel metallic materials. Regarding K_{IH} test, a stress intensity value and test duration should be provided for non-steel metallic materials and therefore further research are needed.

Standards are applicable to machined specimens and not to components. Components of the distribution grid are usually of small size with small thickness which limit the specimen size and testing method. Furthermore, pipe material of the distribution gas grid also have small thicknesses which make the extraction of specimens difficult even impossible for some test methods. So, carrying out mechanical tests will present significant experimental difficulties such as the machining of specimens but also for the measurement of physical and mechanical parameters.

Tests on non-steel metallic materials are limited and therefore limited information on their compatibility with hydrogen gas is available. In the literature, mainly tensile and SSR tests have been performed on non-steel metallic materials. Studies showed that aluminium and its alloys as well as copper and its alloys (oxygen free) have demonstrated good resistance to H_2 gas. Tensile tests performed on smooth and notched specimens in high pressure were unaffected by H_2. Tensile tests performed on cast iron showed that the ductility was deteriorated by H_2; however, test conducted in 20% H_2 at low pressure and room temperature showed that its behaviour does not change compared to same tests carried out in air. FCG tests have been performed on ductile cast iron revealing an acceleration of the crack growth due to the presence of hydrogen and fracture toughness tests were carried out on copper materials.

Further tensile testing but also fracture and fatigue testing should be carried out to improve the understanding of non-steel metallic materials, particularly with regard to pressure. Screening tests (tensile/SSR, disc pressure and C-ring tests), provide qualitative methods for evaluating the relative behavior of metals and alloys in hydrogen gas environments, but these tests do not provide data that allow for a quantitative evaluation of the performance of structural metals in service. For the latter purpose, tests that explicitly address crack initiation and growth under quasi-static or cyclic loading are more relevant. Specifically, fracture mechanics test methods provide quantitative measurements of hydrogen-assisted crack propagation, and similarity concepts allow this data to be used in structural life assessments.

Following this work, fracture tests such as K_{IH} test, fracture toughness, SSR and C-ring and fatigue test such FCGR should be conducted on materials such as ductile and gray cast iron, copper, aluminium, brass and lead. Due to the small material thicknesses used in the distribution gas grid, testing like K_{IH} test, fracture toughness and FCGR might not be possible; therefore, C-ring and SSR on notched specimens could be performed to study the material behavior in presence of a crack in H_2.

New test methods could also be developed for small specimens' size. To evaluate the influence of hydrogen the ISO committee are currently working on a new standard: ISO 7039 "Metallic materials - Tensile testing - Method for evaluating the susceptibility of materials to the effects of high-pressure gas within hollow test pieces".

5. ACKNOWLEDGEMENTS

The project is supported by the Clean Hydrogen Partnership and its members.

Clean Hydrogen Partnership

Co-funded by the European Union

REFERENCES

[1] WP2 - Task 2.1, «Inventory of non-steel metallic materials of the natural gas distribution grid,» 2024.

[2] ASME B31.12, «Hydrogen Piping and Pipelines,» *The American Society of Mechanical Engineers,* 2023.

[3] ASME VIII Div. 3, «Rules for construction of pressure vessels, Article KD10 "Special requirements for vessels in hydrogen service,» *The American Society of Mechanical Engineers,* 2023.

[4] ISO 11114-4, «Transportable gas cylinder - Compatibility of cylinder and valves materials with gas content. Part 4 Test methods for selecting steels resistant to hydrogen embrittlement,» *International Organisation for Standardization,* 2017.

[5] ANSI/CSA CHMC 1, «Test methods for evaluating materials compatibility in compressed hydrogen applications – Metals,» *American National Standards Institute/Canadian Standards Association,* 2014.

[6] EIGA IGC Doc 121/14, «Hydrogen Pipeline Systems,» *European Industrial Gases Association ,* 2014.

[7] EPRG, «Hydrogen Pipelines Integrity management and repurposing Guideline White Paper,» *European Pipeline Research Group,* 2023.

[8] IGEM/TD/1, «Steel pipelines for high pressure gas transmission,» *The Institution of Gas Engineers and Managers,* 2022.

[9] IGEM/H/1, «Reference Standard for low pressure hydrogen utilization,» *Institution of Gas Engineers and Managers,* 2023.

[10] CEN/TR 17797, «Gas infrastructure – Consequence of hydrogen in the gas infrastructure and identification of related standardization need in the scope of CEN/TC234,» *European Committee for Standardization,* 2022.

[11] UNI 9860, «Gas infrastructure - Pipelines with maximum operating pressure no greater than 0.5 MPa (5 bar) - Gas user derivation systems - Design, construction, testing, operation, maintenance and rehabilitation,» *Italian National Standards,* 2022.

[12] ASTM E1681, «Standard Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials,» *American Society for Testing and Materials,* 2023.

[13] ASTM E647, «Standard Test Method for Measruement of Fatigue Crack Growth Rates,» *American Society for Testing and Materials,* 2015.

[14] ASTM F1459, «Standard Test Method for Determination of the Susceptibility of Metallic Materials to Hydrogen Gas Embrittlement,» *American Society for Testing and Materials,* 2017.

[15] ISO 7539-6, «Corrosion of metals and alloys - Stress corrosion testing - Part 6: Preparation and use of precracked specimens or tests under constant load or constant displacement,» *Internationel Standardization for Standardization,* 2018.

[16] ASTM G142, «Standard Test Method for Determination of Susceptibility of Metals to Embrittlement in Hydrgen Containing Environments at High Pressure, High Teperature, or Both,» *American Society for Testing and Materials,* 2022.

[17] ASTM E8/E8M, «Standard Test Methods for Tension Testing of Metallic Materials,» *American Society for Testing and Materials,* 2022.

[18] ASTM E1820, «Standard Test Method for Measurement of Fracture Toughness,» *American Society for testing and Materials,* 2021.

[19] ASTM E466, «Standard Practice for Conducting Force Constant Amplitude Axial Fatigue Tests of Metallic Materials,» *American Society for Testing and Materials,* 2015.

[20] ASTM E606, «Standard Test Method for Strain-Controlled Fatigue Testing,» *American Society for Testing and Materials,* 2021.

[21] ASTM G129, «Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking,» *American Society for Testing and Materials,* 2000.

[22] C. San Marchi and J. Ronevich, «PVP2022-84757: Fatigue and Fracture of Pipeline Steels in High-Pressure Hydrogen Gas,» in *ASME 2022 Pressure Vessel and Piping Conference*, Las Vegas, 2022.

[23] ISO 12135, «Metallic Materials - Unified method of test for the determination of quasistatic fracture toughness,» *International Organization for Standardization,* 2021.

[24] ISO 15653, «Metallic materials - Method of test for the determination of quasistatic fracture toughness of welds,» *International Organisation for Standardization,* 2018.

[25] TWI website, «https://www.twi-global.com/technical-knowledge/faqs/faq-what-is-a-tearing-resistance-curve,» [Online].

[26] ASTM G142, «Standard Test Method for Determination of Susceptibility of Metals to Embrittlement in Hydrogen Containing Environments at High Pressure, High Temperature, or Both,» *American Society for Testing and Materials,* 2022.

[27] ASTM G129, «Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking,» *American Society for Testing and Materials,* 2021.

[28] ISO 7539-7, «Corrosion of metals and alloys - Stress corrosion testing - Part 7: Method for slow strain rate testing,» *Internationel Standardization for Standardization,* 2005.

[29] ASTM E399-22, «Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials,» *American Society for Testing and Materials,* 2022.

[30] ASTM E647, «Standard Test Method for Measurement of Fatigue Crack Growth Rates,» *American Society for Testing and Materials,* 2023.

[31] ASTM G38, «Standard Practice for Making and Using C-Ring Stress Corrosion Test Specimens,» *American Society for Testing and Materials,* 2021.

[32] ISO 7539-5, «Corrosion of metals and alloys - Stress corrosion testing - Part 5: Preparation and use of C-ring specimens,» *International Organization for Standardization,* 1989.

[33] SANDIA Report, «Technical Reference for Hydrogen Compatibility of Materials,» 2012.

[34] SDG22-01, «Estudio del posible efecto de la conducción conjunta de gas natural/hidrógeno en la resistencia mecánica de las conducciones de canalización de gas fabricadas en fundición dúctil,» 2022.

[35] Dr. Navdeep Singh Khalon, «HyDeploy Project - Winlaton Trial Report,» 2022.

[36] H2SAREA, «Informe de evaluacion de pruebas realizadas en H2SAREA,» Nortegas, May 2023.

[37] M. Steiner, U. Marewski, H. Silcher, «Investigation of Steel Material for Gas Pipelines and Plants for Assessment of their Suitability with Hydrogen,» DVGW Project SyWeSt H2, 2023.

[38] NATURALHy, «Preparing for the hydrogen economy by using the existing natural gas system as a catalyst,» *GERG,* 2010.

[39] HIGGS, «Hydrogen in Gas GridS: a systematic validation approach at various admixture levels into high-pressure grids, Project Results,» *H2020, CORDIS, European Commission.*

[40] Birkitt K., Loo-Morrey M., Sanchez C., O'Sullivan L., «Materials aspects associated with the addition of up to 20 mol% hydrogen into an existing natural gas distribution network,» *Internal journal of hydrogen energy,* vol. 46, pp. 12290-12299, 2021.

[41] ISO 6892, «Metallic materials. Tensile testing - Method of test at room temperature,» *International Organization for Standardization,* 2016.

[42] Matsuo T., «the effect of pearlite on the hydrogen-induced ductility loss in ductile cast irons,» *Journal of Physics,* 2017.

[43] Matsuno k. et al., «Effect of hydrohen of uniaxial tensile behaviors of ductile cast iron,» *International Journal of Modern Physics,* 2012.

[44] Sahiluoma P. et al., «Hydrogen Embrottlement of nodular cast iron,» *Materials and Corrosion,* 2020.

[45] Matsunaga H., Takakuwa O., Yamabe J. and Matsuoka S., «Hydrogen-enhanced fatigue crack growth in steels and its frequency dependence,» *Philosophical Transaction A,* 2016.

[46] AIGA 087/14, «Standard for Hydrogen Piping System at User Locations,» *Asia Industrial Gases Association,* 2012.

[47] KIWA, «Approval Requirement 214 - Fitness for admixtures up to and including 100% hydrogen,» 2021.

[48] Vennett RM and Ansell GS, *A Study of Gaseous Hydrogen Damage in Certain FCC Metals,* 1969.

[49] Barthélémy H., «Effect of purity and pressure on the hydrogen embrittlment of steels and other metallic materials».

[50] Walter RJ and Chandler WT, «Influence of Gaseous Hydrogen on Metals: Final Report,» *National Aeronautics and Space Admiistration,* 1973.

[51] San Marchi C. and Somerday B., «Technical Reference on Hydrogen Compatibility of Materials,» 2008.

[52] Ordin P.M., «Safety Standard for Hydrogen and Hydrogen Systems. Guidelines for hydrogen system design, materials selections, operations, storage and transportation,» 1997.

[53] Chene J. and Brass AM., «Role of temperature and strain rate on the hydrogen-induced intergranular rupture in alloy 600,» *Metallurgical and Materials Transactions A,* 2004.

[54] Hasegawa M. and Osawa M., «Hydrogen danage of nickel-base heat resistant alloys,» 1981.

[55] Panagopoulos C.N, El-Amoush A.S., Georgarakis K.G., «The effect of hydrogen charging on the mechanical behaviour of α-brass,» *Journal of Alloys and Compounds,* 2005.

[56] Briottet L. and Riccetti B., «Permeation de l'hydrogene dans une canalisation en polyethylene, Tache WP5-Matérial de réseau et compresseurs,» *Rapport technique No. DTBH(RT/2016/065),* 2016.

[57] API 6D, «Specification for Pipeline and Piping Valves,» *American Petroleum Institute,* 2014.

[58] ISO 14313, «Petroleum and natural gas industried - Pipeline transportation systems - Pipeline valves,» *International Organization for Standardization,* 2007.

[59] ASTM E1681, «Standard Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials,» *American Society for Testing and Materials,* 2023.

[60] [Online]. Available: https://www.twi-global.com/technical-knowledge/faqs/faq-what-is-a-tearing-resistance-curve#.

[61] ISO 12135, «Metallic materials - Unified method of test for the determination of quasistatic fracture toughness,» *International Organization for Standardization,* 2016.

[62] NACE TM0198, «Slow Strain Rate Test Method for Screening Corrosion-Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Service,» *National Association of Corrsion Engineers,* 2020.

[63] ASTM E399-97, «Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials,» *American Society for Testing and Materials ,* 1997.

[64] ISO 12108, «Metallic materials - Fatigue testing - Fatigue crack growth method,» *The International Organization for Standardization,* 2018.

[65] A. F1459-06, Standard Test Method For Determination Of The Susceptibility Of Metallic Materials To Hydrogen Gas Embrittlement (HGE), 2017.