
**Transportable gas cylinders —
Compatibility of cylinder and valve
materials with gas contents —**

**Part 4:
Test methods for selecting metallic
materials resistant to hydrogen
embrittlement**

*Bouteilles à gaz transportables — Compatibilité des matériaux des
bouteilles et des robinets avec les contenus gazeux —*

*Partie 4: Méthodes d'essai pour le choix de matériaux métalliques
résistants à la fragilisation par l'hydrogène*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11114-4 was prepared by Technical Committee ISO/TC 58, *Gas cylinders*.

ISO 11114 consists of the following parts, under the general title *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents*:

- *Part 1: Metallic materials*
- *Part 2: Non-metallic materials*
- *Part 3: Autogenous ignition test in oxygen atmosphere*
- *Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement*

Introduction

It is widely recognised that compressed hydrogen and some hydrogen bearing gases can have an embrittling effect on metallic materials, especially steels. This embrittling effect has resulted in the failure of hydrogen cylinders (including some bursts) that has led gas cylinder users and manufacturers to adopt specific measures.

The adoption of these measures has eliminated all known failures of hydrogen cylinders from this embrittlement phenomenon.

The basic recommendation was to minimise the stresses in the cylinder wall (see ISO 11114-1) and eliminate harmful defects.

This tensile strength limit of 950 MPa was developed for quenched and tempered gas cylinders of 34 Cr Mo 4 type steels using steelmaking practices, chemistry and manufacturing techniques typical of those used during the early 1980s and successfully used for filling pressures up to 300 bar. This practice has been in widescale use up to the current time. Other higher pressures, although at lower tensile strength limits, have also been used.

In recent years, improvements in steelmaking, e.g. by reducing the sulphur and phosphorus contents, have indicated the possibility of increasing the tensile strength limit of 950 MPa for embrittling gas service, as a result of improvements in the fracture toughness of the material.

Experimental work has shown that the relevant parameters affecting hydrogen embrittlement are the following:

- a) microstructure resulting from the combination of the chemistry and the heat treatment;
- b) mechanical properties of the material;
- c) applied wall stress;
- d) internal surface imperfections resulting in local stress concentrations;
- e) characteristics of the gas contained (composition, quality, pressure, etc.).

When developing this part of ISO 11114 only the material aspects, a) and b) above, were considered. Other essential features, c) and d), are covered by the relevant parts of ISO 9809.

This part of ISO 11114 specifies test methods to identify steels which, when combined with the cylinder manufacturing requirements specified in ISO 9809 (all parts), will result in cylinders suitable for use in embrittling gas service.

However, some low alloy steels other than 34 Cr Mo 4 may require tensile strength lower than 950 MPa to be suitable for the manufacture of gas cylinders for embrittling gas service.

These tests have been developed following an extensive world-wide programme which incorporated laboratory and full scale tests.

Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents —

Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement

1 Scope

This part of ISO 11114 specifies test methods and the evaluation of results from these tests in order to qualify steels suitable for use in the manufacture of gas cylinders (up to 3 000 l) for hydrogen and other embrittling gases.

This part of ISO 11114 only applies to seamless steel gas cylinders.

The requirements of this part of ISO 11114 are not applicable if at least one of the following conditions for the intended gas service is fulfilled ¹⁾:

- the working pressure of the filled embrittling gas is less than 20 % of the test pressure of the cylinder;
- the partial pressure of the filled embrittling gas of a gas mixture is less than 5 MPa (50 bar) in the case of hydrogen and other embrittling gases, with the exception of hydrogen sulphide and methyl mercaptan in which cases the partial pressure shall not exceed 0,25 MPa (2,5 bar).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4287, *Geometrical product specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 7539-1:1987, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-6:2003, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*

ISO 9809-1, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 1: Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa*

ISO 9809-2, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 2: Quenched and tempered steel cylinders with tensile strength greater than or equal to 1 100 MPa*

1) In such cases the cylinders may be designed as for ordinary (non embrittling) gases.

ISO 9809-3, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 3: Normalized steel cylinders*

ISO 11114-1:1997, *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents — Part 1: Metallic materials*

ISO 11120, *Gas cylinders — Refillable seamless steel tubes for compressed gas transport, of water capacity between 150 l and 3 000 l — Design, construction and testing*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply. Some of the definitions used are based upon those in ISO 7539-1:1987.

3.1.1 embrittling gases

gases listed in ISO 11114-1:1997, Table A.3, group 2 and Table A.12, group 11

3.1.2 hydrogen rupture pressure, P_{H_2}

maximum pressure recorded during the hydrogen rupture pressure test

3.1.3 helium rupture pressure, P_{He}

maximum pressure recorded during the helium rupture pressure test

3.1.4 hydrogen embrittlement index

maximum value of the ratio P_{He}/P_{H_2} as a function of the pressure rise rate

3.1.5 environmentally-assisted cracking

synergistic effect on a metal caused by the simultaneous action of a particular environment and a nominally static tensile stress, which results in the formation of cracking

3.1.6 threshold stress

stress above which a crack will initiate and grow, for the specified test conditions

3.1.7 plane strain stress intensity factor, K_1

function of applied load, crack length and specimen geometry having dimensions of stress $\times$ length

NOTE K_1 uniquely defines the elastic stress field intensification at the tip of a crack subjected to opening mode displacements.

3.1.8 threshold stress intensity factor for susceptibility to environmentally-assisted cracking, K_{1H}

stress intensity factor above which an environmentally-assisted crack will initiate and grow, for the specified test conditions under conditions of high constraint to plastic deformation, i.e. under essentially plane strain conditions

3.1.9 HAC hydrogen assisted cracking

3.2 Symbols

For the purposes of this document, the following symbols apply.

a	effective crack length measured from the crack tip to the loading plane
a_0	average value of a
B	specimen thickness
e_m	the mean disc thickness
E	modulus of elasticity
K_{IAPP}	applied elastic stress-intensity
K_{1H}	threshold stress intensity factor
m	elastic displacement per unit load
P	applied load
P_r	actual rupture pressure
P_r'	corrected rupture pressure
P_{rH2}'	corrected hydrogen rupture pressure
P_{rHe}'	theoretical helium rupture pressure corresponding to the same pressure rise rate as for the hydrogen test, calculated by regression from the corrected helium rupture pressure
R_m	actual value of tensile strength
$R_{P0,2}$	average of measured yield stress of three specimens from the test cylinder representing the HAC test specimen's location at room temperature
V	crack-mouth opening displacement (CMOD) defined as the mode 1 (also called opening-mode) component of crack displacement due to elastic and plastic deformation, measured at the location on a crack surface that has the greatest elastic displacement per unit load, m
W	effective width of a compact specimen, measured from the back face to the loading plane
Y	stress intensity factor coefficient derived from the stress analysis for a particular specimen geometry, which relates the stress intensity factor for a given crack length to the load and specimen dimensions

4 General requirements

The test methods as described in Clause 5 are valid for working pressure up to 300 bar. For higher working pressures additional verification shall be undertaken.

The tests shall be performed for selecting steels for hydrogen cylinders. Chromium–molybdenum steels, quenched and tempered with a guaranteed maximum actual ultimate tensile strength of 950 MPa, do not need to be tested and can safely be used for the construction of hydrogen cylinders. For carbon–manganese steels, different limits may apply (see ISO 9809-1).

The tests described in Clause 5 are “qualification tests” for a given steel. This means that the tests need not be repeated for each “type” of cylinder once a steel has been qualified for a specific design strength level.

The test samples shall be taken from a representative cylinder or from a piece of tube (for long cylinders according to ISO 11120), representative of the relevant manufacturing process including heat treatment.

The test samples shall have a mechanical strength not lower than the maximum intended tensile strength to be used for the cylinders to be manufactured. If it is intended later to increase the maximum strength of the steel, a new qualification test shall be performed.

With respect to the possible variation of the chemical composition, the chemistry of the steel tested shall be recorded in the qualification test report and the difference in chemistry for the steels actually used for the cylinders shall not exceed the "permissible difference" according to ISO 9809-2. In addition, for sulphur and phosphorus, these permissible differences are limited to 0,005 % and 0,010 %, respectively. In no case shall the phosphorus content of either the qualification or the production cylinders exceed 0,015 %.

With respect to the heat treatment, the manufacturer shall specify the relevant temperatures and times, and the quenching conditions (if relevant). Any modification to the heat treatment needing a new type approval according to ISO 9809-2 requires a new qualification test.

For the qualification of a given steel for the manufacturing of gas cylinders, method A, B, or C can be used (see 5.1, 5.2 and 5.3, respectively). Additionally, tensile tests shall be carried out (see 5.4).

5 Test methods

5.1 Disc test (method A)

5.1.1 Principle of test

A mounted test piece in the shape of a disc is subjected to an increasing gas pressure at constant rate to burst or to crack. The embrittling effect of hydrogen is evidenced by comparing the hydrogen rupture pressures P_{H_2} with the helium rupture pressures P_{He} , helium being chosen as a reference gas.

The ratio P_{He}/P_{H_2} shall be determined.

The lower this ratio, the better will the steel type behave in the presence of hydrogen. This ratio is dependent on the pressure rise rate, which shall remain constant during the whole test.

NOTE 1 Hydrogen rupture pressures also depend on the hydrogen purity. Oxygen or traces of water vapour can partially inhibit the hydrogen embrittlement effect.

NOTE 2 The test can be carried out with any other embrittling gas or gas mixture (e.g. H_2S , hydrides). The embrittlement index of the considered gas will then be defined similarly.

5.1.2 Test conditions and procedure

5.1.2.1 Sample disc

The sample disc shall be flat and ground (or machined to an equivalent surface finish), and shall have the following characteristics.

Dimensions:

- Diameter: $58 \begin{smallmatrix} 0 \\ -0,5 \end{smallmatrix}$ mm.
- Thickness: $0,75 \text{ mm} \pm 0,005 \text{ mm}$.
- Flatness: less than 1/10 mm deflection.

Surface condition (both sides):

- Roughness: R_a value (see ISO 4287) less than 0,001 mm. The roughness of the samples used for both H_2 and He tests shall be equivalent.
- No trace of oxides.

The following operations shall be performed to verify the sample quality:

- Immediately after the final preparation and prior to the testing, store the samples in a dry atmosphere, e.g. a desiccator.
- Degrease the sample and check thickness at 4 points taken 90° apart to define a mean thickness.
- Determine the disc's hardness (e.g. Vickers) over its outer circumference to verify that machining has not altered the original material properties.

5.1.2.2 Cell and other apparatus

The cell (see Figure 1) is composed of 2 stainless steel flanges embedding the disc. A volume of approximately 5 cm³ is provided below the disc. Above the disc a high-strength steel ring is mounted ($R_m \geq 1\,100$ MPa). The internal diameter is 25,5 mm and the ring curvature radius at the mounting diameter level is 0,5 mm.

Gas discharges can occur after disc bursting either through the lower flange or upper flange discharge port to the atmosphere. This permits the evacuation of the installation and a check of the hydrogen purity and freedom from either oxygen ($O_2 < 1\ \mu\text{l/l}$) or water vapour ($H_2O < 3\ \mu\text{l/l}$)²⁾. It also permits regulation of the gas flow and adjustment of the pressure rise rate.

The sealing device shall be an elastomer O-ring for helium testing, and for hydrogen testing at rates of more than 10 bar/min. For hydrogen testing at rates not exceeding 10 bar/min, indium O-rings shall be used.

For the flange assembly, ten high-strength steel bolts should be used, size typically M10 or equivalent. The tightening torque shall be 30 N·m for elastomer O-rings and 100 N·m for indium O-rings.

Hydrogen and helium shall be stored in high-pressure containers connected to the test cells. A flow control valve between the high-pressure container and the cell shall be used to adjust the pressure rise rate.

5.1.2.3 Test procedure

For a satisfactory outcome of the test performances, the following operations shall be carried out.

- Evacuation of the cell by pumping to eliminate any traces of air or moisture absorbed by the walls. Purging with the gas to be used, followed by vacuum pumping, can also be used to improve the cleaning efficiency.
- Check of the gas purity before testing.
- Adjustment of the gas flow to achieve the appropriate pressure rise rate.
- Isolation of the cell (at the start of pressure rise).

The rate of pressure rise shall be monitored for the duration of the entire test. It shall be regular and kept as constant as possible, neglecting the gas compressibility factor with pressure.

2) 1 $\mu\text{l/l}$ = 1 ppm. The use of ppm is deprecated.

The rupture pressures shall be noted from the recorded readings at the end of the test. The pressure gauge shall be accurate to $\pm 2\%$ for the intended measured rupture pressure.

The tests shall be carried out with hydrogen (for quality, see 5.2.2.3) and with helium ($H_2O < 3 \mu\text{l/l}$) for a range of pressure rise rates evenly distributed between 0,1 bar/min and 1 000 bar/min. When using these latter pressure rates, the minimum value for the rupture pressure shall be established (see Figure 2). This minimum value shall be used to run the rest of the tests. It is generally considered that 6 helium tests and 9 hydrogen tests (i.e 15 tests in total) are enough for a thorough material evaluation.

5.1.3 Treatment and interpretation of test results

5.1.3.1 Disc thickness

Rupture pressure P_r shall be systematically corrected for deviation from the "ideal" value corresponding to the test of a standard thickness disc.

The corrected rupture pressure P'_r , shall be given by the formula:

$$P'_r = \frac{P_r \times 0,75}{e_m}$$

where e_m is the mean disc thickness.

5.1.3.2 Presentation and interpretation of test results

Rupture pressures, corrected as indicated in 5.1.3.1, shall be plotted against the mean pressure rise rate (actual rupture pressure divided by the test duration) expressed in bar/min (see Figure 2).

For each hydrogen test, calculate the ratio

$$P'_{r\text{He}}/P'_{r\text{H}_2}$$

where

$P'_{r\text{He}}$ is the theoretical helium rupture pressure corresponding to the same pressure rise rate as for the hydrogen test, calculated by regression from the corrected helium rupture pressure;

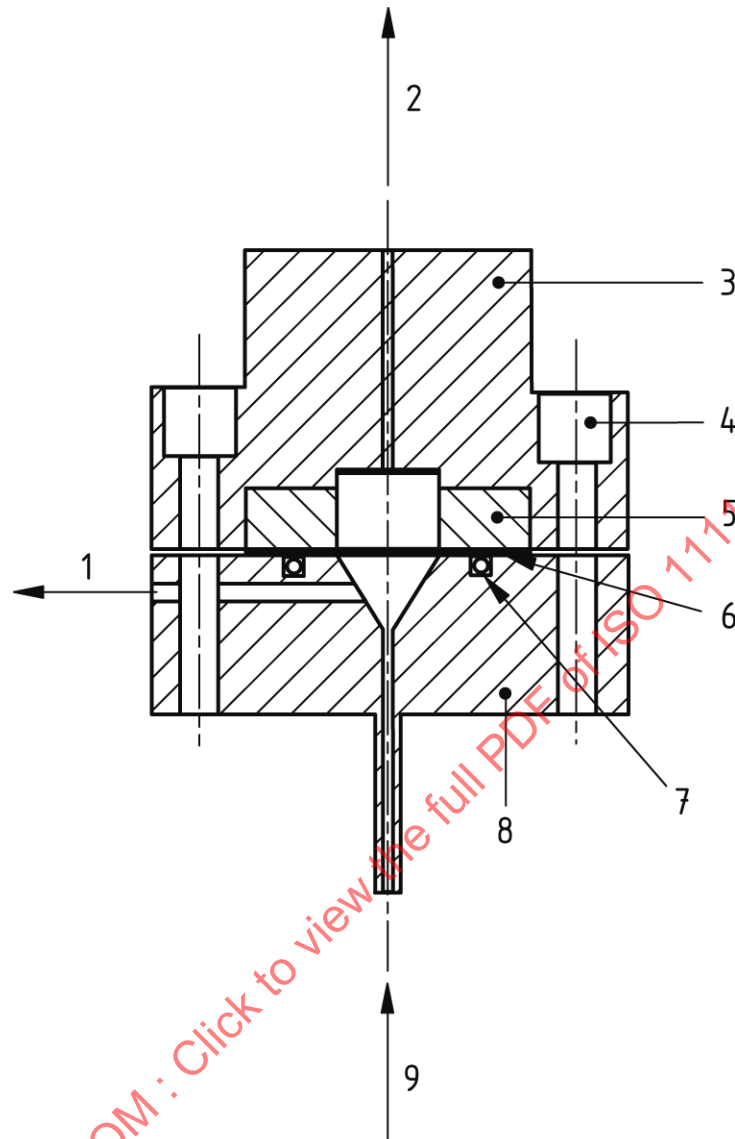
$P'_{r\text{H}_2}$ is the corrected hydrogen rupture pressure.

Ratios of $P'_{r\text{He}}/P'_{r\text{H}_2}$ shall be plotted against the pressure rise rate (see Figure 3).

Test results shall be interpreted bearing in mind that the embrittlement index of a material is the maximum value of the above-mentioned ratio and that the material shall be considered as suitable for compressed hydrogen cylinders if the index is less than or equal to 2.

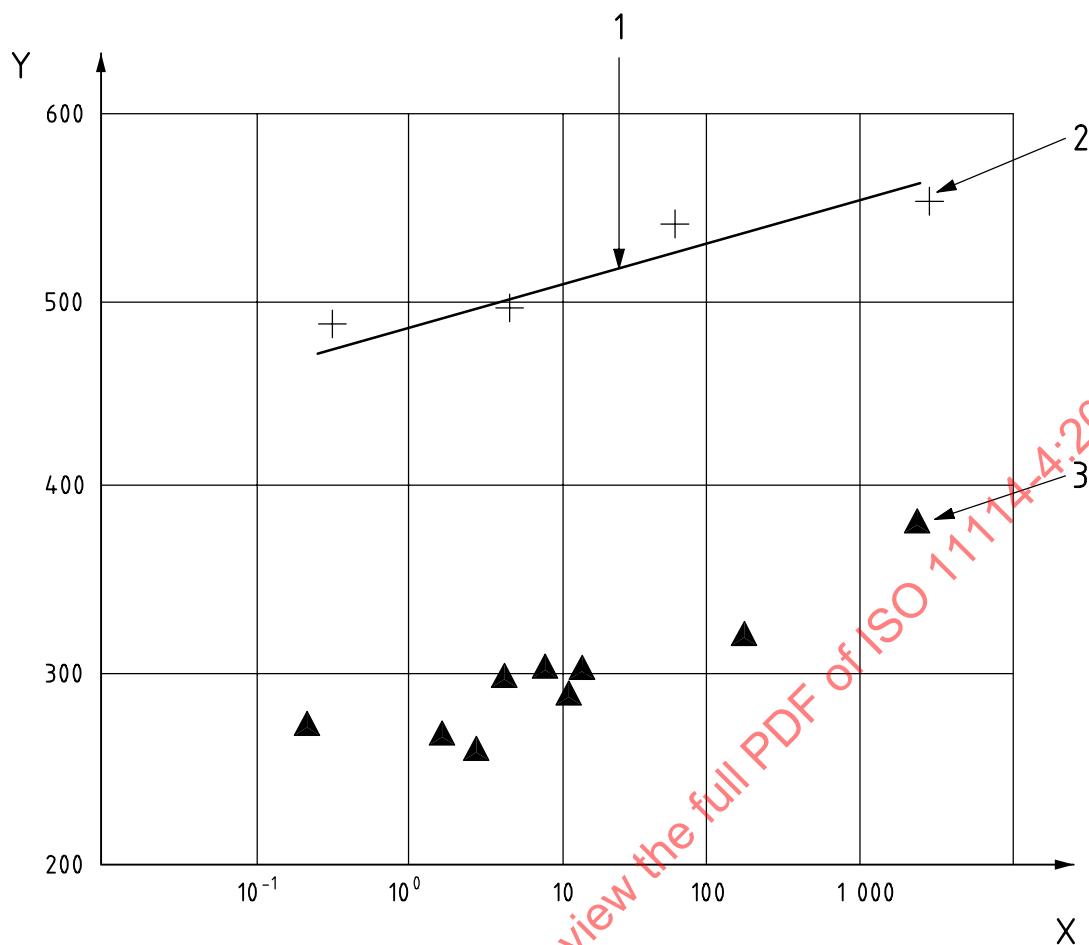
5.1.4 Test report

The detailed test conditions (gas purity, rupture pressure, hardness of the specimen, ratio $P'_{r\text{He}}/P'_{r\text{H}_2}$, embrittlement index) and the cylinder design drawing including the material properties, chemical composition and heat treatment conditions of the tested cylinder shall be reported.

**Key**

- 1 port for evacuation and flow adjustment
- 2 discharge port
- 3 upper flange
- 4 bolt hole
- 5 high-strength steel ring
- 6 disc
- 7 O-ring
- 8 lower flange
- 9 gas inlet

Figure 1 — Test installation (test cell)



X pressure rise rate (bar/min)
Y corrected rupture pressure (bar)

Key

- 1 regression curve
- 2 helium corrected rupture pressures ($P_{r,He}$)
- 3 hydrogen corrected rupture pressures (P_{r,H_2})

Figure 2 —Examples of hydrogen and helium corrected rupture pressures as a function of the pressure rise rate

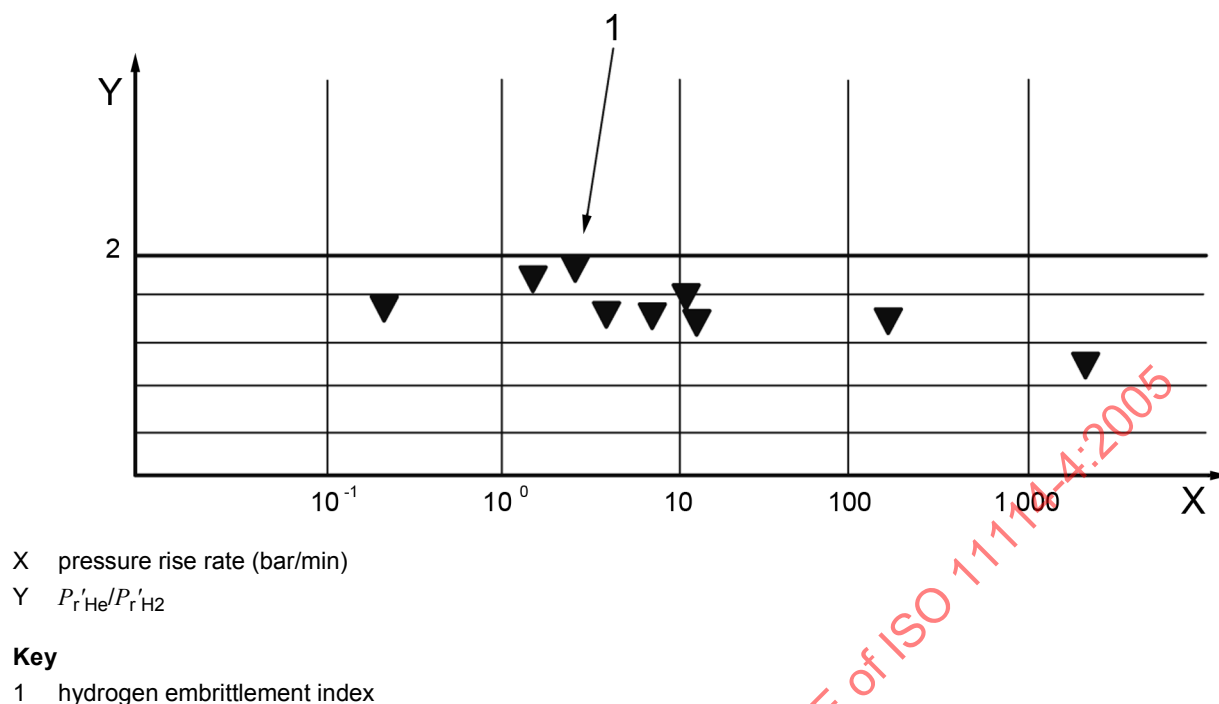


Figure 3 — Examples of the ratio P'_{rHe}/P'_{rH2} as a function of the pressure rise rate

5.2 Fracture mechanic test (method B)

5.2.1 Principle of the test method

This specifies a method for the determination of the threshold stress intensity factor (K_{1H}) for susceptibility to cracking of metallic materials in gaseous hydrogen.

The procedure covers the use of machined compact tension test pieces, as described in ISO 7539-6, for the determination of the threshold stress intensity factor, as described in ISO 7539-1.

The test involves a specimen containing a machined notch. This notch is extended by fatigue cracking to an incrementally increasing tensile load during exposure to a pressurised, gaseous hydrogen environment. The objective is to quantify the conditions under which environmentally-assisted crack propagation can occur.

If the specimens pass the test requirements, then the material is characterized as suitable for gas cylinders filled with compressed hydrogen.

5.2.2 Test procedure

5.2.2.1 Specimen type

The test involves the use of a compact tension (CT) type specimen, according to the geometry given in Figure 4 (Figure 3 of ISO 7539-6:2003), with a W dimension of 26 mm and B equal to the greatest thickness allowed by the cylinder wall curvature and thickness, but not less than 85 % of the design thickness of the cylinder being qualified. The specimen orientation (Y-X) relative to the cylinder axis is shown in Figure 5.

At least 2 specimens diametrically opposed from the cylindrical wall shall be tested. Flattening of the specimen is not allowed. If the test specimen thickness cannot meet the validity requirements of ISO 7539-6, then the thickest possible specimen as specified above shall be tested.

Dimensions in millimetres
Surface roughness values in micrometres

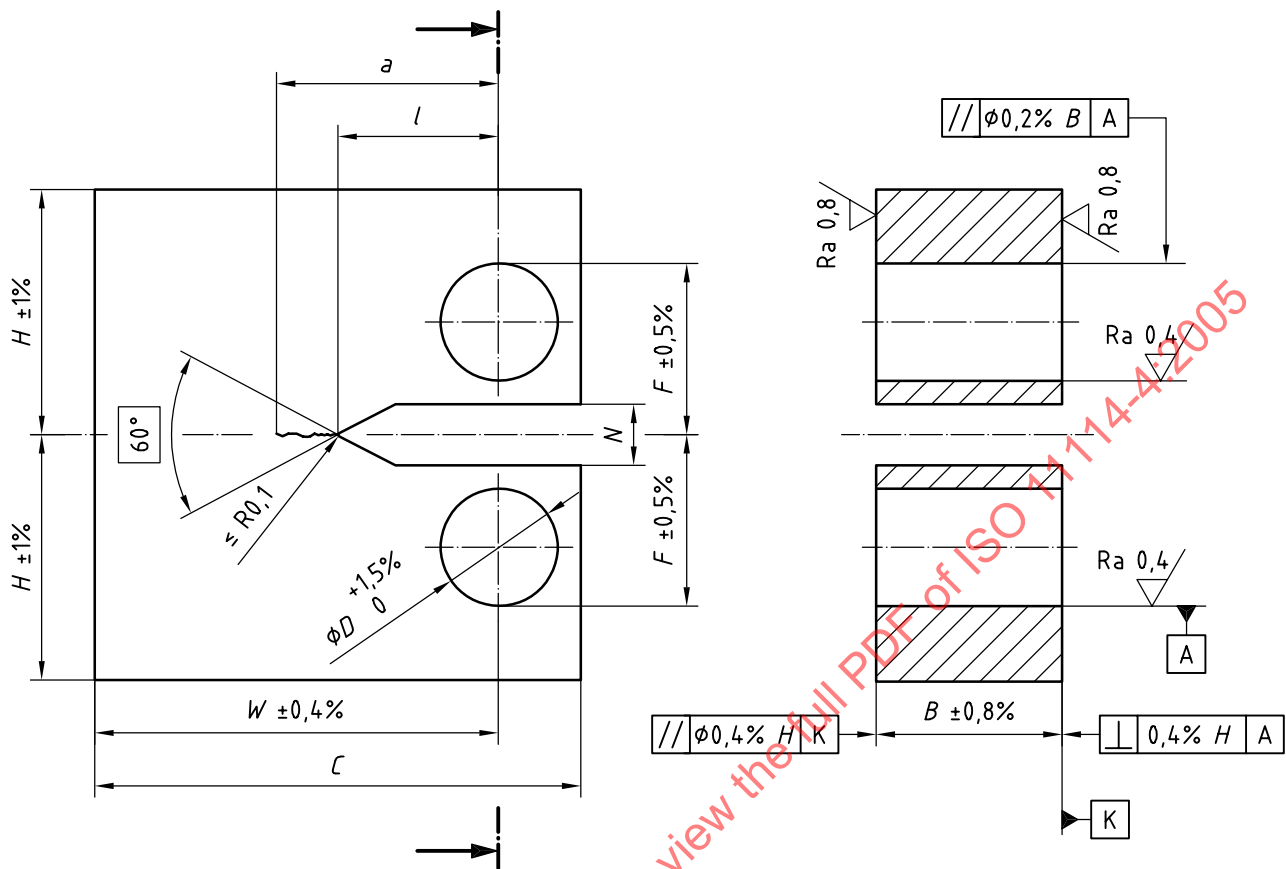


Figure 4 — Proportional dimensions and tolerances for compact tension test pieces

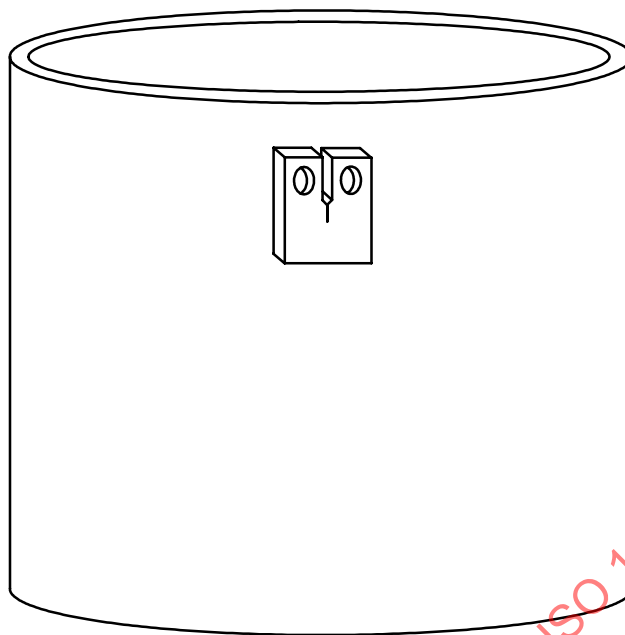


Figure 5 — Test piece: type and orientation

5.2.2.2 Specimen preparation

Prior to fatigue pre-cracking all necessary electrical contacts and wires required for crack monitoring shall be attached to the specimen. The specimen shall be thoroughly degreased, with particular attention paid to the notch tip region.

During all subsequent operations, especially the fatigue pre-crack, the specimen shall not be contaminated in order to ensure a clean crack front.

Fatigue pre-cracking shall be conducted in the test chamber in an atmosphere of hydrogen and meet the general requirements detailed in Clause 6 of ISO 7539-6:2003 for the initiation and propagation of fatigue cracks. The final maximum pre-cracking load shall give a final maximum stress intensity lower than the initial stress intensity required for the test.

On completion of fatigue pre-cracking, the specimen shall be measured as detailed in Clause 7 of ISO 7539-6:2003 to determine the thickness B , width W and average length of the fatigue pre-crack on the specimen surfaces. The last of these values shall be used in assessing the load necessary to produce the required initial stress intensity.

To prevent oxidation of the crack tip, the specimen shall now be put on test. If, for any reason, this is not possible, the specimen shall be stored in a clean, desiccated environment until ready for testing.

5.2.2.3 Gas purity and pressure

The hydrogen pressure in the test chamber shall be 150 bar, with a hydrogen purity of at least 99,999 5 %, and $O_2 \leq 1 \mu\text{l/l}$ and $H_2O \leq 3 \mu\text{l/l}$. This gas shall be covered by a batch certificate of conformity.

As an alternative, gas with a 99,999 9 % purity ($O_2 \leq 0,1 \mu\text{l/l}$ and $H_2O \leq 0,5 \mu\text{l/l}$) and covered by an individual certificate of conformity may be used.

5.2.2.4 Test apparatus

The tests shall be conducted in a stainless steel chamber, shown together with the loading bars and specimen in Figure 6, and installed on a servo-hydraulic test machine (Figure 7).

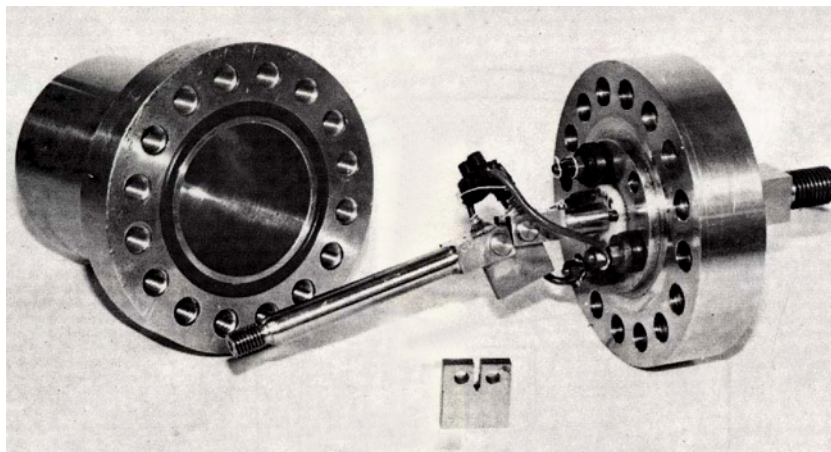


Figure 6 — Stainless steel chamber, showing loading bars and specimen

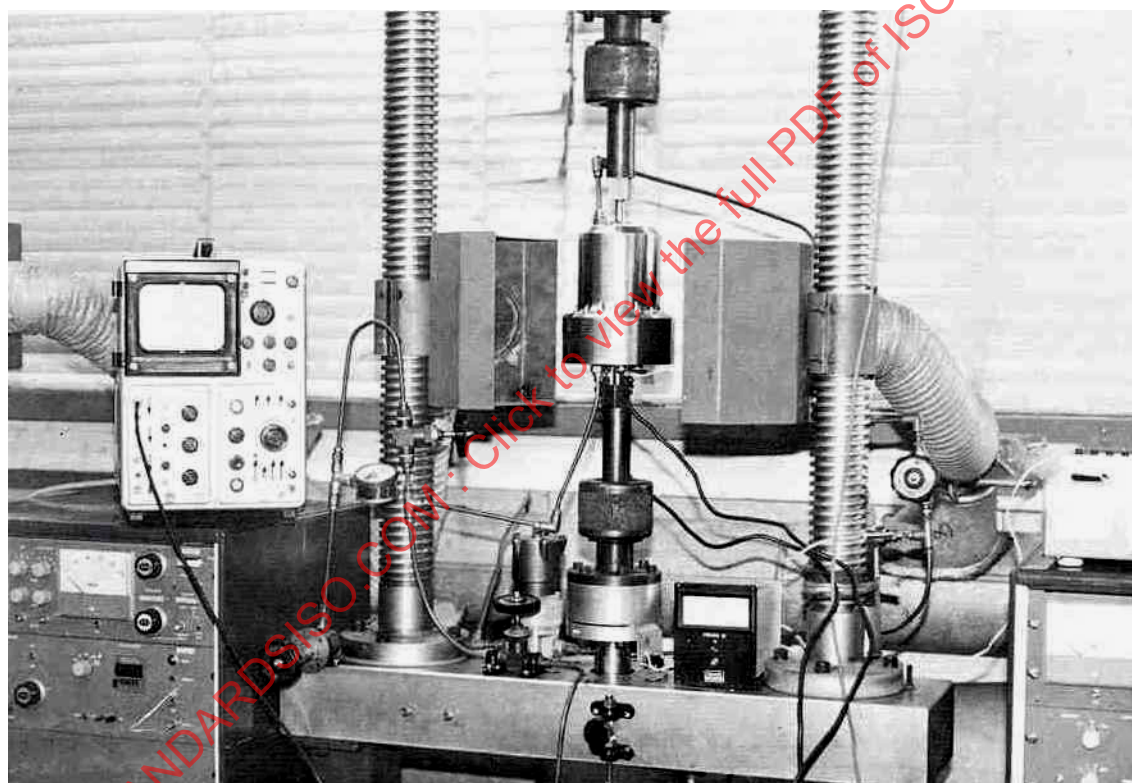


Figure 7 — Servo-hydraulic test machine

After installation of the specimen, the test chamber shall be evacuated to a pressure of < 130 Pa prior to the introduction of gaseous hydrogen. The purity of the gaseous environment in the test chamber shall be as specified in 5.2.2.3.

Crack initiation, and any subsequent growth, shall be detected and monitored using a direct current potential drop (DCPD) method.

5.2.2.5 Test method

If there is no prior knowledge of the material's threshold stress intensity factor, then the test shall be started using a load which produces a stress intensity factor of $1 \text{ MPa}\cdot\text{m}^{0.5}$ at the crack tip. Otherwise, the test may be started at a stress intensity factor equivalent to, but not exceeding, 50 % of the stress intensity factor derived from previous work.

The applied load shall be recorded continuously. The specimen shall be held at the initial load for a period of at least 20 min to allow for crack growth at room temperature. If no crack growth is detected by the end of the hold period, as evidenced by a plateau in the potential drop curve, the load shall be increased at a controlled rate in a step equivalent to a crack tip stress intensity factor increase of $1 \text{ MPa}\cdot\text{m}^{0.5}$, and held for a further 20 min. This procedure shall be continued until failure occurs, with the appropriate load being recorded on the test data sheet.

The load shall be increased at the end of a hold period, by means of a servo-controller, at a constant rate of $2 \times 10^{-3} \text{ kN}\cdot\text{s}^{-1}$, until attainment of the crack tip stress intensity required for the subsequent hold period. This would result in a controlled crack tip stress intensity factor increase totalling $1 \text{ MPa}\cdot\text{m}^{0.5}$ over a time interval of approximately 1 min, assuming a test piece of thickness 7 mm and with an a/W of 0,5.

5.2.2.6 Specimen measurement

The specimen shall again be inspected in accordance with ISO 7539-6, with the appropriate measurements to 0,01 mm being made using a travelling microscope.

5.2.3 Test results

5.2.3.1 Analysis and acceptance criteria

Calculation of the test result shall be as stated in ISO 7539-6, through a relationship of the form

$$K_{1H} = YP/BW^{0.5}$$

The load P used in the calculation is that applied to the specimen before the final increment that caused failure.

Provided the K_{1H} values of both CT specimens are greater than or equal to $(60/950) \times R_m$ ($\text{MPa}\cdot\text{m}^{0.5}$), the material is qualified for a maximum tensile strength (R_m) equal to the average of the two tensile strength values obtained in 5.4. If the constraint validity requirement of 5.2.2.1 is not met, then the material is suitable up to the design thickness of the cylinder tested.

5.2.3.2 Test report

The detailed test conditions [gas purity and pressure, record of the direct current potential drop (DCPD) measurements, location and dimension of the specimen, K_{1H} and R_m (see 5.4) and the cylinder design drawing], including the material properties and chemical composition and the heat treatment conditions of the tested cylinder, shall be reported.

5.3 Test method to determine the resistance to hydrogen assisted cracking of steel cylinders (method C)

5.3.1 General

A fatigue-precracked specimen is loaded by a constant load or constant displacement method to a stress-intensity K_{IAPP} equal to a defined value. The specimen is kept in the loaded condition for a specified time in pressurized hydrogen gas at room temperature. After the test period, the specimen is examined to assess whether the initial fatigue crack did or did not grow.

If the test specimen exhibits less than or equal to a specified amount of crack growth, then the material is characterized as suitable for gas cylinders with respect to the hydrogen assisted cracking (HAC) resistance requirement.