



**International
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**

*Matériaux métalliques — Essais de traction — Méthode
d'évaluation des changements de propriétés dans un
environnement gazeux à haute pression en utilisant une pièce
d'essai creuse*

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols	2
5 Principle	2
6 Test piece	3
6.1 General	3
6.2 Shape and dimensions	3
6.3 Inner surface of hole	3
6.4 Number of test pieces	4
7 Measurement of original cross-sectional area	4
8 Marking the original gauge length	4
9 Test equipment	4
9.1 Principle	4
9.2 Testing machine	5
9.3 Extensometer	5
9.4 Test deviation	5
10 Test conditions	5
10.1 Test gas	5
10.2 Test temperature	6
10.3 Test pressure	6
10.4 Testing rate	6
11 Evaluation of the test results	6
11.1 Yield strength, tensile strength, percentage elongation after fracture with hollow test piece	6
11.2 Percentage reduction of area at leakage	7
11.3 Relative elongation after fracture with hollow test piece, $A_{h(rel)}$	7
11.4 Relative reduction of area at leakage with hollow test piece, $Z_{h(rel)}$	7
11.5 Relative 0,2 % proof strength with hollow test piece, $R_{p0,2 h(rel)}$	7
11.6 Relative tensile strength with hollow test piece, $R_{mh(rel)}$	8
12 Test report	8
Bibliography	9

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 1, *Uniaxial testing*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Hollow test pieces have been occasionally used for the evaluation of influence of hydrogen^[1] since the 1950's. But those hollow test pieces were mostly shaped with thin walls or in a tubular form, and the deformation behaviour was different from that of solid test piece; as a result, neither the percentage elongation after fracture (the elongation) nor the percentage reduction of area (the reduction of area) can be accurately obtained. The influence of a high-pressure hydrogen gas environment has been conventionally evaluated using a solid test piece within a gas-filled cylinder or vessel with the tensile force applied from outside the vessel to evaluate the influence of the hydrogen gas on the material under test as described in ASTM G 142^[2] or ISO 11114-4^[3].

Since 2005, the use of a hydrogen gas filled hollow test piece has been utilized as a method to evaluate changes in tensile properties of metallic materials due to concurrent exposure to gaseous hydrogen. It was found in previous studies^{[4]–[9]} that testing of a hollow test piece with a small diameter axial hole pressurized with gaseous hydrogen yielded similar trends for both the elongation and the reduction of area to testing of solid test pieces stored in a similar gaseous environment. For this reason, this method has been considered a material screening test method for evaluation of metallic materials in gaseous hydrogen and the resulting data are not suitable for design.

This document does not address the determination of entirely the same values of mechanical properties for design purposes as specified by the ISO 6892 series but is suitable as a screening or first selection method for metallic materials in a gaseous or liquid media. The hollow test piece is suitable for the evaluation of materials used for high-pressure pipe or vessels, and can be used not only for hydrogen gas, but also for other gaseous or corrosive media. However, the major concerns for the hollow test piece are the hoop stress and the roughness of inner surface. In this document, the hollow test piece method is regarded for tests filled with various media and the required preparation of the test piece, such as an inner surface finish, is also described.

The type of the pressurized gas in the hollow test piece can affect the tensile properties in the test. Also, the specific test conditions, e.g., test speed, test gas, internal pressure, temperature, and gas purity, can affect the outcome. As this document describes in general the test procedure for the hollow test piece, it does not describe the most suitable test conditions for all possible variations of the test parameters. Preliminary tests should be conducted to identify technical relevant test conditions. Test conditions in this document will be revised with the increase of test results.

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Metallic materials — Tensile testing — Method for evaluating the susceptibility of materials to the effects of high-pressure gas within hollow test pieces

1 Scope

This document specifies the geometries and proposed finishing procedures of the inner surface of hollow test piece of metallic materials, filled with a high-pressure gaseous medium. The document specifies a tensile testing procedure to evaluate the effect of high-pressure gaseous medium compared to a high-pressure inert gas or air. The document can be used for the screening of metallic materials by evaluating mechanical property changes due to the effects of various test gases, including hydrogen.

NOTE Temperature range and pressure range depend on the materials to be tested and test gas to be used.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

ISO 6892-2, *Metallic materials — Tensile testing — Part 2: Method of test at elevated temperature*

ISO 6892-3, *Metallic materials — Tensile testing — Part 3: Method of test at low temperature*

ISO 9513, *Metallic materials — Calibration of extensometer systems used in uniaxial testing*

ISO 21920-2, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

ASME B31.8, *Gas Transmission and Distribution Piping Systems for thick wall pipes*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6892-1, ISO 21920-2 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

hollow test piece

test piece in form of round bar with a hole in its central axis

3.2

relative elongation after fracture with hollow test piece

$A_{h(rel)}$

value of the elongation after fracture obtained with a high-pressure gaseous medium divided by the elongation after fracture obtained with a high-pressure inert gas or air with hollow test piece

3.3

relative reduction of area with hollow test piece

$Z_{h(rel)}$

value of the reduction of area at leakage obtained with a high-pressure gaseous medium divided by the reduction of area obtained with a high-pressure inert gas or air with hollow test piece

3.4

relative 0,2 % proof strength with hollow test piece

$R_{p0,2 h(rel)}$

value of the 0,2 % proof strength obtained with a high-pressure gaseous medium divided by the 0,2 % proof strength obtained with a high-pressure inert gas or air with hollow test piece

3.5

relative tensile strength with hollow test piece

$R_{mh(rel)}$

value of the tensile strength obtained with a high-pressure gaseous medium divided by the tensile strength obtained with a high-pressure inert gas or air with hollow test piece

4 Symbols

The symbols and corresponding designations are given in [Table 1](#).

Table 1 — Symbols and designations

Symbol	Unit	Designation
d_o	mm	original internal diameter of the parallel length
d_f	mm	internal diameter at the fracture section after fracture
D_o	mm	original outer diameter of the parallel length
D_f	mm	outer diameter at the fracture section after fracture
$\dot{\epsilon}_{L_c}$	s ⁻¹	estimated strain rate over the parallel length
L_o	mm	original gauge length
L_f	mm	final gauge length after fracture
A_h	%	percentage elongation after fracture with hollow test piece: $A_h = \frac{L_f - L_o}{L_o} \times 100$
S_o	mm ²	original cross-sectional area of the parallel length
S_f	mm ²	final cross-sectional area at leakage
Z_h	%	percentage reduction of area at leakage with hollow test piece: $Z_h = \frac{S_o - S_f}{S_o} \times 100$
$A_{h(rel)}$		relative elongation after fracture with hollow test piece
$Z_{h(rel)}$		relative reduction of area with hollow test piece
$R_{p0,2 h(rel)}$		relative 0,2 % proof strength with hollow test piece
$R_{mh(rel)}$		relative tensile strength with hollow test piece

5 Principle

A tensile test is carried out by pressurizing the hole of the hollow test piece with a test gas to determine the effects of the test gas and temperature on the mechanical properties of the material under test.

The effect of the test gas is evaluated by comparing the material properties to those obtained by filling the hole of the hollow test piece with high-pressure inert gas or air at the same test conditions.

6 Test piece

6.1 General

The shape and dimensions of inner hole of the test pieces may be adapted to the material and the gaseous medium to be tested.

6.2 Shape and dimensions

The outer shape of the test piece shall be a cylindrical test piece. ISO 6892-1 can be used as a guideline for test piece selection.

The outer diameter (D_o) of the parallel part of the test piece should be within the range of 3 mm to 12 mm, and the recommended inner diameter (d_o) of the parallel length (hole) is within the range of 1 mm to 4 mm. The d_o/D_o ratio is recommended to be less than 0,33. The d_o/D_o ratio can be agreed between the parties.

The gauge length of the specimen can be according to ISO 6892-1 and the ring-like cross section of the hollow test piece can be used as area of the cross section to calculate the gauge length.

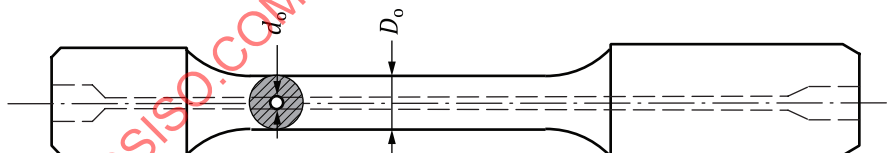
NOTE 1 The percentage elongation after fracture of hollow test pieces becomes smaller than that of solid test pieces with the increase of the d_o/D_o ratio.

NOTE 2 The d_o at the grip ends is not specified but the test piece breaks at the grip if the wall thickness is too thin.

The calculated hoop stress according to ASME B31.8, at test pressure shall not exceed two-thirds of the measured yield strength of the test material in air. Appropriate wall thickness shall be determined for each combination of test piece size, material strength, pressure and temperature.

Figure 1 shows an example of the shape and dimensions of the test piece. The hole shall be located along the entire gauge length of the test piece and should go through full length of test piece (full hollow test piece), but optionally, a blind hole test piece can be allowed according to the agreements between parties. Procedures for the inner surface finish (see 6.3) and the test gas purity (see 10.1) for the blind hole test piece should be agreed.

The total run-out tolerance of the hole should be within 0,05 mm over the parallel length. The gas fill connection can be placed at the end of the grip section(s).



Key

d_o inner diameter

D_o outer diameter

Figure 1 — Schematic illustration of a hollow test piece

6.3 Inner surface of hole

The inner surface shall be prepared so as not to affect the properties of the sample. In the case of a heat-affected layer of inner surface arising by drilling or electrical discharging machining, it shall be removed.

NOTE The effect of the surface quality of the inner side of the test piece on the tensile properties is part of the ongoing scientific discussion.

After final finishing the inner surface of the hole, any remaining machining debris and/or polishing media shall be removed from the inner surface with twisted paper, cotton thread, or any other means. Any cleaning method is acceptable provided that no influence on test results, such as hydrogen ingress is possible during

the cleaning process. The hole shall be rinsed with a solvent to remove oil and grease. The finishing procedure and the final finish of the inner surface polish may be according to the agreements between parties.

An inner surface roughness, R_a of 0,25 μm or better is recommended. Measurement or estimation shall be reported.

For the blind hole test piece, the machining and polishing processes should be examined in preliminary tests according to the agreements between parties.

6.4 Number of test pieces

More than two hollow test pieces should be tested with both inert gas and the test gas, unless agreed otherwise between the parties. The properties obtained with inert gas should be averaged for calculation of relative values in [11.3](#) to [11.6](#).

7 Measurement of original cross-sectional area

The outer diameter of parallel part shall be the average of three points measured along the parallel length of the test piece.

The inner diameter should be measured using, for example, measuring pins. If the inner diameter is not measured, a nominal inner diameter can be used. However, caution should be taken to minimize the error this introduces, and knowledge about the process to make the hole is necessary. The method for measuring the inner diameter or if the nominal diameter is used, should be noted in the test report.

8 Marking the original gauge length

Marking of the original gauge length shall be carried out in accordance with ISO 6892-1.

9 Test equipment

9.1 Principle

[Figure 2](#) shows the principle of the high-pressure gaseous environment test apparatus using hollow test pieces and an example schematic of the basic system. The apparatus consists of a material testing machine that applies tensile force to the test piece, a gas supply unit that fills the hollow part of the test piece with gas, and a temperature control unit that heats and cools the hollow test piece filled with gas.

Safety requirements are not shown in this schematic; it is the responsibility of the user to refer to relevant local safety regulations, codes and standards for deployment of pressure systems.

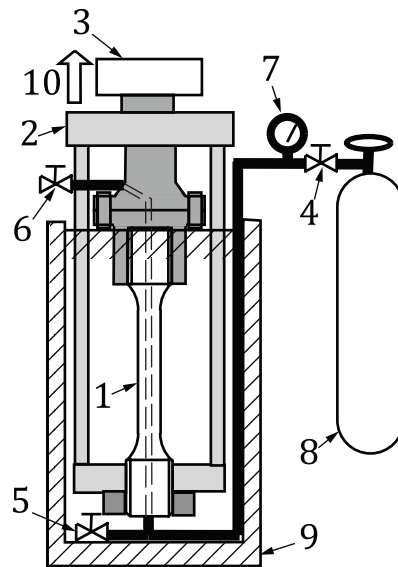
The axis of the test piece shall coincide with the axis of application of the force.

During the testing, the gas supply shall be shut (see valve A in [Figure 2](#)).

If a gas analysis is performed, gas analysis access ports should be at either valve B or valve C (see [Figure 2](#)). However, other valves may be used according to agreement between parties. The valve used for sampling shall be stated in the test report.

For tests conducted with high-pressure combustible and/or other gas, a flammable or other gas hazard exists. Although with proper system design the amount of release combustible or other gas can be minimized. The user of this document should be aware of appropriate local regulations, codes and standards for additional safety requirements (such as locating equipment in an explosion-proof room).

NOTE A small diameter high-pressure line and minimum line length from valve A (see [Figure 2](#)) to the test piece minimizes the volume of gas released upon failure of the test piece.

**Key**

1	test piece	6	valve C
2	loading Frame	7	pressure gauge
3	load cell	8	gas cylinder
4	valve A	9	temperature-control bath
5	valve B	10	load

Figure 2 — Principle of the high-pressure gaseous medium test method with hollow test pieces and schematic of basic system (non-safety related).

9.2 Testing machine

The tensile testing machine used shall conform with the requirements of ISO 6892-1 and shall be able to control slow strain rates.

9.3 Extensometer

In case extensometers are used, the equipment shall be in accordance with ISO 9513 class 1 unless otherwise agreed between parties.

9.4 Test deviation

If it is found that a deviation in specified test parameters (temperature, pressure, test rate, etc.) occurred during the conduct of the test, then the test shall be repeated, unless otherwise agreed between the parties.

10 Test conditions

10.1 Test gas

Two gases shall be used to conduct tests with the hollow test piece: a test gas and an inert gas. A test gas (with appropriate safety measures), for example, can be either hydrogen, hydrogen sulphide, a corrosive gas, ammonium gas or carbon dioxide gas. An inert gas can be either helium, argon, or nitrogen for reference in high-pressure condition and for purge.

The test piece shall be purged or vacuum-pumped with the inert gas at least three times to remove air from the system. Three purge cycles with the test gas shall be conducted to displace the inert gas. The purity, or

quality, of the test gas is an important testing parameter and should be defined by the parties and shall be reported.

NOTE A high number of purging cycles will result in high purity of the test gas and reduce the effect of gas contamination.

In a hydrogen environment, hydrogen effects are very sensitive to impurities and hydrogen of 99,999 9 % concentration or higher should be used. Other gas qualities can be used according to agreement between the parties. The gas quality shall be stated in the test report, including at least the hydrogen concentration and the content of oxygen (O₂) and water vapor (H₂O).

For tests with gaseous hydrogen, analysis of the test gas from the test piece hole can be performed using a trace oxygen meter and a dew point meter (or equivalent methods) according to the agreement between the parties.

For tests with inert gas, the pressure shall be the same as that of the test gas; however, if it is proven that there is no effect of pressure, the test may be conducted in atmospheric pressure air.

10.2 Test temperature

The test temperature shall be in accordance with ISO 6892-1 for room temperature and ISO 6892-2 or ISO 6892-3 for testing at elevated or low temperature. The soaking time (time taken to stabilize the temperature of the test piece prior to mechanical loading) should be included in the test report.

10.3 Test pressure

The test pressure is the pressure of the test gas enclosed in the hole of the test piece at the start of loading. The test pressure shall be adjusted by a gas pressure regulator, but for tests at high or low temperatures the test piece temperature may also be adjusted to achieve a change in pressure.

Prior to starting the test, the pressure shall be stable within ± 1 MPa for a minimum of 10 min if the test gas pressure is greater than or equal to 20 MPa. If the test gas pressure is less than 20 MPa, it shall be stable within ± 5 % of the test pressure for a minimum of 10 min. This is the pressure stabilization time, and it shall be included in the test report.

The test pressure shall be continuously monitored and/or recorded during the test. The test pressure shall be maintained within ± 5 % of the specified test pressure for the duration of the test until the test gas is released at the final fracture or before the fracture due to the crack propagation to the surface of the test piece. The requirement to maintain it within ± 5 % after the start of the test applies regardless of which pressure the test is being performed at.

10.4 Testing rate

The test speed shall be less than or equal to $0,000\ 05\ \text{s}^{-1}$ at the estimated strain rate over the parallel length $\dot{\epsilon}_{L_c}$.

This is the maximum recommended strain rate. To provide the maximum effect of damage for certain test gases in the hollow test piece, slower strain rates can be used.

11 Evaluation of the test results

11.1 Yield strength, tensile strength, percentage elongation after fracture with hollow test piece

In general, the evaluation of the test shall be done in accordance with ISO 6892-1. However, percentage elongation after fracture and percentage reduction of area of the hollow test piece are generally smaller than those of the solid test piece due to the hollow and should be used as comparison only.

If the test gas pressure in the hollow releases before final fracture, the displacement at which the gas released should be identified from the recorded stress-displacement curve or gas pressure-displacement curve. The difference between the displacements before and after final fracture should be subtracted from