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Standard Test Method for Determination of the Susceptibility of Metallic Materials to Hydrogen Gas Embrittlement (HGE)[1]

This standard is issued under the fixed designation F1459; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the quantitative determination of the susceptibility of metallic materials to hydrogen embrittlement, when exposed to high pressure gaseous hydrogen.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E384 Test Method for Knoop and Vickers Hardness of Materials

3. Summary of Test Method

3.1 A thin disk metallic specimen is subjected to an increasing gas pressure at constant rate until failure (bursting or cracking of the disk). The embrittlement of the material can be evaluated by comparing the rupture pressures of identical disk specimens in hydrogen (P_{H2}) and in a reference inert gas such as helium (P_{He}) (**1, 2**).[2]

3.2 The ratio P_{He}/P_{H2} can be used to evaluate the susceptibility of the metallic material to gaseous hydrogen embrittlement. The ratio is dependent on the pressurization rate. A ratio of 1 or less indicates the material is not susceptible to hydrogen embrittlement. A ratio greater than 1 indicates that the material is susceptible to hydrogen embrittlement and the susceptibility increases as the ratio increases.

4. Significance and Use

4.1 This test method will provide a guide for the choice of metallic materials for applications in high pressure hydrogen gas.

4.2 The value of the P_{He}/P_{H2} ratio will be a relative indication of the severity of degradation of the mechanical properties to be expected in hydrogen.

5. Apparatus

5.1 A basic test system shall consist of the following items:

5.1.1 *Test Cell,* consists of two flanges as shown schematically in Fig. 1.

5.1.1.1 The test cell shall befabricated from materials such as 316 stainless steel in the annealed condition that are not susceptible to HGE (**3, 4**).

5.1.1.2 The seals shall be elastomer O-rings for helium testing and hydrogen testing at rates of 10 bar/min (145 psig/min) or higher. For hydrogen tests at a lower rate, indium O-rings shall be used.

5.1.1.3 An evaluation port (Item 1 in Fig. 1) on the lower flange is used to check gas purity and adjust pressurization rate.

5.1.2 The test cell is pressurized with hydrogen or helium through a pneumatic system. Fig. 2 schematically illustrates the pneumatic system.

5.1.2.1 The pressurization rate shall be adjustable in the system. A throttle valve is used to adjust the pressurization rate in Fig. 2.

6. Gases

6.1 *Helium,* purity 99.995 minimum, 6000-psig (41 400-kPa) or higher pressure source.

6.2 *Hydrogen,* purity 99.995 minimum, 6000-psig (41 400-kPa) or higher pressure source.

7. Specimen Preparation

7.1 Fifteen (15) specimens with identical dimensions and temper conditions shall be prepared for each test program. Six (6) specimens are to be tested in helium and nine (9) specimens are to be tested in hydrogen. One specimen is to be tested at the predetermined pressurization rate in helium or hydrogen as prescribed in 8.2.3.

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[1] This test method is under the jurisdiction of ASTM Committee F07 on Aerospace and Aircraft and is the direct responsibility of Subcommittee F07.04 on Hydrogen Embrittlement.

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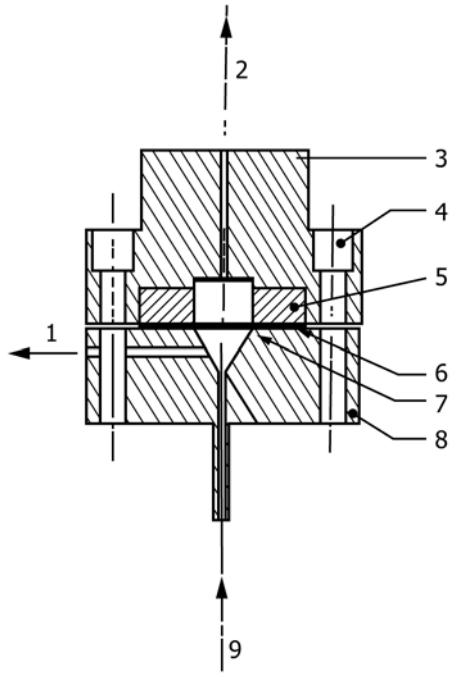
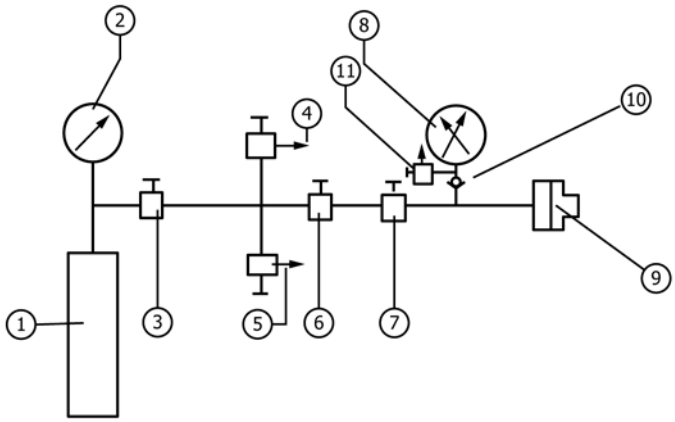
[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.



1. Port for evacuation and flow adjustment
2. Discharge port
3. Upper flange
4. Bolt
5. High strength steel ring
6. Disk
7. O-ring
8. Lower flange
9. Gas inlet

FIG. 1 Test Cell



1. High-pressure tank
2. Pressure gage
3. High-pressure valve
4. To vacuum pump
5. To pressure intensifier
6. High-pressure valve
7. Throttle valve
8. Slave hand pressure gage
9. Test cell
10. Check valve
11. Pressure bleed valve

FIG. 2 Schematic of Disk Pressure Test

7.2 The specimens for the test cell, illustrated in Fig. 1, have a diameter of 58 mm (2.28 in.) and a thickness of 0.75 mm (0.030 in). If not available, other thickness between 0.25 and 1 mm (0.010 and 0.040 in.) are also acceptable.

7.3 The disk specimen shall have a flatness of less than 1/10 mm (1/254 in.) deflection.

7.4 The surface of the disk specimen shall be free of oxides. The surface roughness *Ra* shall be 0.001 mm (40 µin.) or less.

7.5 The disk specimen shall be prepared by a method that does not alter mechanical properties of the material at the edge of the specimen. Microhardness testing should be conducted per Test Method E384 at the outer edge of the specimen (outside the tested area) to ensure it is as a means of confirming that the mechanical properties were not altered.

7.6 The specimens shall be cleaned, free of grease and dried before test.

8. Procedure

8.1 *Pressurization Procedure:*

8.1.1 Install a disk specimen in the test cell.

8.1.2 Evacuate the system to 10^{-2} to 10^{-3} torr for 3 min to eliminate air, moisture, and residual test gases from the system.

8.1.3 Purge the system with the gas to be tested, check gas purity from the evacuation port on a per batch basis.

8.1.4 Repeat 8.1.2 and 8.1.3 if necessary.

8.1.5 Adjust the pressurization rate to the desired level.

8.1.6 Pressurize the system. The pressure versus time data shall be recorded.

8.1.7 Stop the test when the disk has burst. Record the burst or crack pressure.

8.2 *Test Procedure:*

8.2.1 Perform hydrogen and helium tests according to the pressurization procedure in 8.1.

8.2.2 Six (6) specimens shall be tested in helium. Nine (9) specimens shall be tested in hydrogen.

8.2.3 The pressurization rates shall be between 0.1 and 1000 bar/min (1.5 to 14 500 psi/min). Suggested pressurization rates are 0.1, 1, 10, 100, 500, and 1000 bar/min (1.5, 15, 145, 1450, 7250, and 14 500 psi/min). Additional tests shall be conducted in hydrogen at or near the rates that yield the lowest burst pressure.

9. Calculation

9.1 Plot the burst pressure versus pressurization rate (logarithmic scale) for the hydrogen and helium test data.

9.2 Perform a linear regression on the helium data to obtain a linear relation between the rupture pressure and the pressurization rate.

9.3 Calculate the apparent helium burst pressure based on the linear regression at the pressurization rates in the hydrogen pressure.

9.4 Calculate the ratio P_{He}/P_{H2} at the pressurization rate tested in hydrogen, where P_{He} is the apparent helium burst pressure calculated from 9.3.

9.5 Plot the ratio P_{He}/P_{H2} versus pressurization rate.

10. Interpretation of Results

10.1 The maximum value of the P_{He}/P_{H2} ratio is used to evaluate the susceptibility of the test material to hydrogen gas embrittlement.

10.2 If the maximum ratio P_{He}/P_{H2} is equal to 1, the material is considered to be not susceptible to hydrogen

embrittlement. If the ratio is higher than 2, the material is considered to be sensitive to hydrogen, and provisions must be taken to avoid exposure to hydrogen. If the ratio is between 1 and 2, embrittlement may be expected after long exposure to hydrogen in any form.

11. Report

11.1 Report material information with alloy identification, hardness, chemistry, heat treatment, and so forth.

11.2 Report specimen geometry including diameter and thickness.

11.3 Report the test conditions including gas purity and pressurization rates.

11.4 Report the ratio P_{He}/P_{H2} at each pressurization rate and the maximum value of the ratio.

12. Precision

12.1 *Reproducibility*—The results of the test for each material and condition obtained by the same operator usually differ by the following percentages: normally processed and machined specimens, 2 to 3 %; ultra high-strength materials 5 to 10 %.

12.2 Results differing by more than the indicated percentages should be considered suspect and unacceptable.

13. Keywords

13.1 gaseous disk pressure test; hydrogen gas embrittlement; relative susceptibility

REFERENCES

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