



Designation: F1459 – 06

## Standard Test Method for Determination of the Susceptibility of Metallic Materials to Hydrogen Gas Embrittlement (HGE)<sup>1</sup>

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 Microindentation 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 ( $P_{H_2}$ ) and in a reference inert gas such as helium ( $P_{He}$ ) (**1**, **2**).<sup>2</sup>

3.2 The ratio  $P_{He}/P_{H_2}$  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.

<sup>1</sup> 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.

Current edition approved April 1, 2006. Published May 2006. Originally approved in 1993. Last previous edition approved in 1998 as F1459 – 93 (1998)<sup>ε1</sup>. DOI: 10.1520/F1459-06.

<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

### 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  $P_{He}/P_{H_2}$  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 be fabricated 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**.

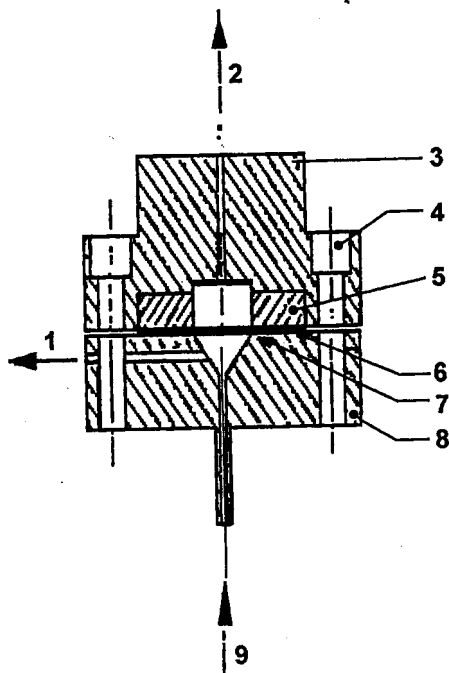
### 6. Gases

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.

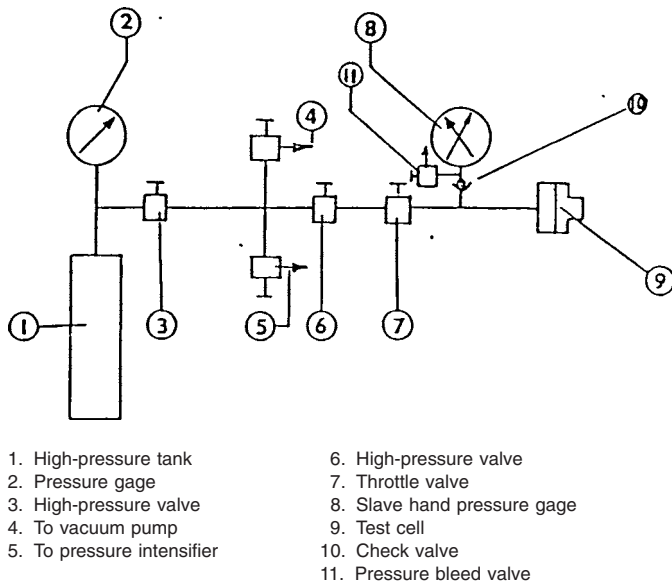
### 7. Specimen Preparation

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



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

FIG. 1 Test Cell



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

FIG. 2 Schematic of Disk Pressure Test

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.

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  $\frac{1}{10}$  mm ( $\frac{1}{254}$  in.) deflection.

7.4 The surface of the disk specimen shall be free of oxides. The surface roughness  $R_a$  shall be 0.001 mm (40  $\mu$ in.) 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  $P_{He}/P_{H_2}$  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  $P_{He}/P_{H_2}$  versus pressurization rate.

## 10. Interpretation of Results

10.1 The maximum value of the  $P_{He}/P_{H_2}$  ratio is used to evaluate the susceptibility of the test material to hydrogen gas embrittlement.

10.2 If the maximum ratio  $P_{He}/P_{H_2}$  is equal to 1, the material is considered to be not susceptible to hydrogen