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Test Method for Evaluating Hydrogen Compatibility of Plastic Liner of High Pressure Gaseous Hydrogen Cylinders

(ISO 11114-5:2022, Gas Cylinders – Compatibility of Cylinder and Valve Materials with Gas Contents – Part 5: Test Methods for Evaluating Plastic, NEQ)

高压氢气瓶塑料内胆和氢气相容性试验方法

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Test Method for Evaluating Hydrogen Compatibility of Plastic Liner of High Pressure Gaseous Hydrogen Cylinders

This Document specifies the general requirements, test conditions, test methods and test reports for hydrogen compatibility testing of plastic liner of high-pressure gaseous hydrogen cylinders.

This Document is applicable to the compatibility test between hydrogen and plastic liner of high-pressure gaseous hydrogen cylinders whose storage medium is hydrogen and whose operating temperature is no lower than -40°C and no higher than 85°C.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 1033.1 Plastics - Methods for determining the density of non-cellular plastics - Part 1: Immersion method liquid pycnometer method and titration method

GB/T 1040.1 Plastics - Determination of tensile properties - Part 1: General principles

GB/T 1040.2 Plastics - Determination of tensile properties - Part 2: Test conditions for molding and extrusion plastics

GB/T 5163 Sintered metal materials，excluding hardmetals - Permeable sintered metal materials - Determination of density，oil content，and open porosity

GB/T 5330.1 Industrial wire screens and woven wire cloth - Guide to the choice of aperture size and wire diameter combinations - Part 1：Generalities

GB/T 5832.1 Gas analysis - Determination of moisture - Part 1: Electrolytic method

GB/T 5832.2 Gas analysis - Determination of moisture - Part 2: Dew point method

GB/T 6285 Determination of trace oxygen in gases - Electrochemical method

GB/T 13005 Terminology of gas cylinders

GB/T 28726 Gas analysis - Gas chromatograph with helium ionization detector

Δp: the pressure difference between the high pressure side and the low pressure side of the specimen during the permeability test, in Pa;

$\frac{\Delta Q}{\Delta t}$: gas transmission rate during stable permeability, in mol/s.

4 General Requirements

4.1 Hydrogen compatibility test of plastic liner of high-pressure gaseous hydrogen cylinder includes plastic liner material test and cylinder liner test. Plastic liner material test includes hydrogen permeability test, physical property test, tensile property test, hydrogen cycle test and hydrogen aging test. Cylinder liner test includes extreme temperature permeability test, liner buckling and bubbling test. The test process is shown in Figure 1.

4.2 The hydrogen compatibility test device shall be overall designed, installed, debugged and managed in accordance with the provisions of GB/T 29729 and GB/T 34542.1.

4.3 There shall be no impurity components in the hydrogen compatibility test device that can interact with the tested sample or pollute the testing hydrogen environment.

5 Test Conditions

5.1 Gas

5.1.1 Hydrogen: it shall meet the requirements of GB/T 37244.

5.1.2 Nitrogen or inert gas: purity ≥99.999%.

5.2 Replacement

5.2.1 Before the test, the test system and hydrogen supply pipeline system shall be replaced with nitrogen or inert gas, and then replaced with hydrogen. Before introducing hydrogen, the volume fraction of oxygen in the test system shall be detected, and its value shall be less than or equal to 1%. At the end of the replacement, the volume fraction of oxygen in the test system shall be less than or equal to 1×10^{-6}; and the volume fraction of water shall be less than or equal to 5×10^{-6}.

5.2.2 The oxygen content shall be tested according to the provisions of GB/T 28726 or GB/T 6285; and the water content shall be tested according to the provisions of GB/T 5832.1 or GB/T 5832.2.

5.2.3 The hydrogen gas, replacement gas and replacement procedure used in the test shall comply with the provisions of 5.1.1, 5.1.2 and 5.2.1 respectively; and when the oxygen and water contents in the test system meets the requirements of 5.2.1 in three consecutive tests,

thickness of the specimen shall be determined by the thickness of the liner.

6.1.2.1.2 For the same liner material, 12 hydrogen permeability test specimens shall be prepared, divided into three groups, 4 in each group.

6.1.2.1.3 Before the test, the specimen shall be placed in a vacuum drying oven with an absolute pressure of 1000Pa~5000Pa and a temperature of 65°C until the mass loss of the specimen within 24 h is less than 0.1%.

6.1.2.2 Test device

6.1.2.2.1 Please refer to Appendix A for the basic structure of the main body of the hydrogen permeability test device.

6.1.2.2.2 The mesh size of the wire screen shall be less than or equal to 0.15mm; the screening area percentage shall be greater than or equal to 35%; and the wire diameter shall be selected according to the provisions of GB/T 5330.1.

6.1.2.2.3 The porosity of sintered metal supports shall be greater than or equal to 40%. Before the test, the porosity of the sintered metal support shall be calibrated according to the provisions of GB/T 5163.

6.1.2.2.4 The sealing ring should be replaced every time a hydrogen permeability test is performed.

6.1.2.2.5 The control accuracy of the temperature control device shall be no less than ±1°C; and the control accuracy of the pressure control device shall be no less than ±1% of the test pressure.

6.1.2.3 Test procedures

6.1.2.3.1 Before the test, the high-pressure side chamber of the specimen shall be replaced according to the provisions of 5.2.1.

6.1.2.3.2 Each group of specimens shall be subjected to hydrogen permeability test under the following temperature and pressure conditions:

a) (55±1)℃, 1.15 times the nominal working pressure of the gas cylinder;

b) (15±1)℃, 1.0 times the nominal working pressure of the gas cylinder;

c) (55±1)℃, 0.1 times the nominal working pressure of the gas cylinder;

d) (15±1)℃, 0.1 times the nominal working pressure of the gas cylinder.

6.1.2.3.3 At the specified test temperature, pressurize the high-pressure side of the specimen to the specified test pressure. After the temperature and pressure on the high-pressure side are stable, measure the hydrogen transmission rate on the low-pressure side of the specimen.

6.1.3 Physical property test

6.1.3.1 Sampling

6.1.3.1.1 A long strip specimen with a length of (40±0.2) mm and a width of (10±0.2) mm should be used. The thickness of the specimen shall be taken as the thickness of the liner.

6.1.3.1.2 At least three physical property specimens shall be prepared for the same liner material.

6.1.3.2 Test device

6.1.3.2.1 Density measuring instruments and impregnation fluids shall comply with the relevant provisions of Method A in GB/T 1033.1.

6.1.3.2.2 If the mass of the specimen is 1g~5g, the measurement value shall be accurate to 0.1mg; if the mass of the specimen is greater than 5g, the measurement value shall be accurate to 1mg.

6.1.3.3 Test procedures

6.1.3.3.1 Follow the following procedures to measure sample mass, density and volume:

a) Use a digital camera to record the appearance image of the specimen;

b) Measure the dry mass of the sample by an analytical balance;

c) Use an analytical balance to weigh the total mass of the impregnation container and the impregnation liquid, and separately weigh the masses of the hanging wires and settling blocks;

d) Immerse the specimen in the impregnation liquid, remove the bubbles on the surface of the specimen and weigh it. The specimen shall not be in contact with the wall of the impregnation container;

e) Perform surface drying treatment on the removed specimens;

f) Calculate the sample density and volume according to the provisions of GB/T1033.1.

6.1.3.3.2 Use a tool microscope or other suitable measuring instrument to measure the dimensions of the specimen.

6.1.3.3.3 Measure the size, volume, mass and density of the specimen before the start of the hydrogen cycle test, within 1 h after the end of the test, 24 h after the end of the test and 48 h after the end of the test; and record the apparent image of the specimen.

6.1.3.3.4 Calculate the volume change rate, mass change rate and density change rate of the specimen before and after the hydrogen cycle test.

6.1.4 Tensile property test

6.1.4.1 Sampling

6.1.4.1.1 The specimen shall be Type-B specimen specified in GB/T 1040.2, and the thickness of the specimen shall be taken as the thickness of the liner.

6.1.4.1.2 For the same liner material, 18 base metal specimens shall be prepared, divided them into two groups, 9 in each group. For welded plastic liner, take 18 specimens at each welding joint per bead and divide them into two groups, 9 in each group. For blow molded liner containing parting line, take 6 specimens at each parting line, divided them into two groups, 3 in each group.

6.1.4.2 Test device

The test device shall comply with the provisions of GB/T 1040.1, GB/T 1040.2 and related standards.

6.1.4.3 Test procedures

The tensile property test of plastic liner materials shall comply with the provisions of GB/T 1040.1, GB/T 1040.2 and related standards. The test shall be carried out according to the following procedures:

a) Before carrying out the hydrogen cycle test, the first group of specimens shall be subjected to tensile tests at (23±2)℃, (-50±2)℃ and (90±2)℃; and the tensile curves shall be recorded;

b) Use the second group of specimens to complete the hydrogen cycle test. 48 h after the hydrogen cycle test is completed, conduct tensile tests at (23±2)℃, (-50±2)℃ and (90±2)℃; and record the tensile curve;

c) Record the change rate of average tensile fracture stress and the change rate of average tensile fracture nominal strain for the material before and after the hydrogen cycle test; and evaluate the mechanical properties of the specimen.

6.1.5 Hydrogen cycle test

6.1.5.1 Sampling

Specimens for hydrogen cycle test shall include:

a) The specimen that completes the first hydrogen permeability test in 6.1.2;

b) The specimen that completes the first physical property test in 6.1.3;

c) The second set of specimens in 6.1.4.

The test device shall be a closed container with controllable temperature and pressure. The temperature control accuracy shall be no less than ±1°C, and the pressure control accuracy shall be no less than ±1MPa.

6.1.6.3 Test procedures

6.1.6.3.1 The hydrogen aging test shall be carried out according to the following procedures:

a) Before test, the specimen shall be dried according to the provisions of 6.1.2.1.3;

b) Take 3 specimens and place them in hydrogen gas with a pressure greater than or equal to 1.25 times the nominal working pressure of the cylinder and a temperature greater than or equal to 85°C for 1000 h. After cooling the temperature to room temperature, slowly reduce the pressure to atmospheric pressure;

c) Take another 3 specimens and store them at ambient temperature and normal pressure for 1000 h, it shall be carried out at the same time as procedure a);

d) Conduct a tensile test on the above specimen according to the test methods in GB/T 1040.1 and GB/T 1040.2; and record the tensile curve.

6.1.6.3.2 During the test, the specimen shall not contact the non-clamping parts of the test chamber.

6.1.6.3.3 Record the change rate of average tensile fracture stress and the change rate of average tensile fracture nominal strain for the material.

6.2 Gaseous cylinder liner test

6.2.1 Extreme temperature permeability test

6.2.1.1 Test device

The test device shall be a closed temperature-controllable container.

6.2.1.2 Test procedures

The hydrogen permeability rate of the gaseous cylinder is measured by connecting an external mass spectrometer, gas chromatograph, hydrogen concentration detection device and other effective methods to the test device. The test shall be carried out according to the following procedures:

a) Place the gaseous cylinder and its accessories in a closed container with a temperature of 55℃~60℃;

b) Use hydrogen to slowly pressurize the gaseous cylinder and its accessories to 1.15 times the nominal working pressure, and let it stand at this pressure for at least 12 h;

c) Measure the hydrogen permeability amount once every 12 h or more, and measure it for at least 3 consecutive times, until the difference between the two measurement values is less than or equal to ±10% of the previous measurement value; then confirm that the permeability has reached a stable state; and record the time for the gaseous cylinder permeability reaches a steady state;

d) Record the change curve of hydrogen permeability in the gaseous cylinder with time.

6.2.2 Liner curvature and bubbling test

After completing the extreme temperature permeability test described in 6.2.1, conduct the liner curvature and bubbling test on the same gaseous cylinder. The test shall be carried out according to the following procedures:

a) Reduce the pressure of the gaseous cylinder to atmospheric pressure at the maximum hydrogen release rate of the gaseous cylinder during actual use. During the deflation process, the surface temperature of the gaseous cylinder shall be greater than or equal to -40°C;

b) Let it stand for 5 h in an environment of (20±5)℃, pressurize the gaseous cylinder to the nominal working pressure and maintain the pressure. The pressure maintaining time shall be greater than or equal to the time for the permeability to reach a stable state in 6.2.1.2;

c) Repeat procedures a) and b) until 9 cycles are completed;

d) Cut the cylinder, inspect the inner surface, and cut surface;

e) Observe whether the liner is buckled and whether there are bubbles, pores and other defects on the surface and cutting surface of the liner.

7 Test Report

The test report shall contain at least the following contents.

a) Test standards and methods:

1) This document number;

2) Other reference documents (including their year of publication);

3) Description of specimen method.

b) Specimen parameters:

1) Gaseous cylinder model;

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