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Proton exchange membrane fuel cell - Part 3: Test method for proton exchange membrane

质子交换膜燃料电池 第 3 部分:质子交换膜测试方法

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Proton exchange membrane fuel cell - Part 3: Test method for proton exchange membrane

1 Scope

This document describes the thickness uniformity test, proton conductivity test, equivalent weight test, air permeability test, tensile property test, swelling rate test, and water uptake test for proton exchange membranes used in proton exchange membrane fuel cells.

This document applies to all types of proton exchange membranes.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 1040.3-2006, Plastics - Determination of Tensile Properties - Part 3: Test Conditions for Films and Sheets (ISO 527-3:1995, IDT)

GB/T 1446-2005, Fiber-reinforced plastics composites - The generals

GB/T 20042.1-2017, Proton exchange membrane fuel cell - Part 1: Terminology

3 Terms and definitions

Terms and definitions determined by GB/T 20042.1-2017 and the following ones are applicable to this document.

3.1

proton conductivity

The ability of a membrane to conduct protons, which is the reciprocal of resistivity.

Note 1: Proton conductivity is an electrochemical indicator to measure the proton conduction capability of the membrane, which reflects the size of the proton mobility in the membrane.

Note 2: The unit of proton conductivity is Siemens per centimeter (S/cm).

3.2

equivalent weight; EW

Dry membrane mass containing 1 mol of protons.

Note 1: It has a reciprocal relationship with IEC (Ion Exchange Capacity) which represents the size of the ion exchange capacity, and reflects the acid concentration in the proton exchange membrane.

Note 2: The unit of equivalent weight is grams per mole (g/mol).

3.3

tensile strength

At given temperature, humidity and tensile speed, when a tensile force is applied to a standard membrane sample, the ratio OF the maximum tensile force that the sample withstands before breaking TO the cross-sectional area of the sample.

Note 1: Transverse tensile strength: indicates the tensile strength of the membrane parallel to the membrane roll axial direction, expressed by σ_{TD}.

Note 2: Longitudinal tensile strength: represents the tensile strength of the membrane perpendicular to the direction of principal axis of the membrane roll, expressed by σ_{MD}.

3.4

modulus of elasticity in tension

The slope of the initial straight-line portion of the stress-strain curve in a proton exchange membrane.

Note 1: Transverse modulus of elasticity in tension: represents the modulus of elasticity in tension of the membrane parallel to the membrane roll axial direction, expressed by E_{TD}.

Note 2: Longitudinal modulus of elasticity in tension: represents the modulus of elasticity in tension of the membrane perpendicular to the direction of principal axis of the membrane roll, expressed by E_{MD}.

Note 3: The slope of the two points on the recommended stress-strain curve where the strains are $\varepsilon_1 = 0.5\%$ and $\varepsilon_2 = 2.5\%$ is the modulus of elasticity in tension.

Note 4: The modulus of elasticity in tension is represented by E, in megapascals (MPa).

3.5

tensile strain at break

The increment per unit length of the original gauge length when the sample breaks.

Note 1: Transverse tensile strain at break: indicates the tensile strain at break of the membrane parallel to the membrane roll axial direction, expressed by ε_{TD}.

Note 2: Longitudinal tensile strain at break: represents the tensile strain at break of the membrane perpendicular to the direction of principal axis of the membrane roll, expressed by ε_{MD}.

Note 3: The tensile strain at break is represented by ε, in a dimensionless ratio or percentage (%).

3.6

peeling force at 180° angle

Under the peeling condition where the peeling angle is 180°, the load required for continuous peeling of a test strip of certain width at a certain speed.

Note: The unit of peeling force at 180° angle is Newtons per millimeter (N/mm).

3.7

gas permeation rate

Under constant temperature and unit pressure difference, during steady permeation, the volume of gas permeating the unit area of the sample per unit time.

Note: The gas permeation rate is expressed by the volume value under standard temperature and pressure, and the unit is cubic centimeters per square meter day Pa [$cm^3/(m^2 \cdot d \cdot Pa)$].

3.8

gas permeation coefficient

Under constant temperature and unit pressure difference, during steady permeation, the volume of gas permeating the unit thickness and unit area of the sample per unit time.

Note: The gas permeation coefficient is expressed by the volume value under standard temperature and pressure, and the unit is cubic centimeter per square meter second Pa [$cm^3 \cdot cm/(cm^2 \cdot s \cdot Pa)$].

3.9

water uptake

The amount of water absorbed per unit mass of dry membrane at a given temperature.

Note: Water uptake is expressed in %.

3.10

swelling rate

Dimensional change in the transverse, longitudinal and thickness directions relative to the dry membrane at a given temperature.

Note 1: The dimensional changes in the transverse, longitudinal and thickness directions are recorded as TD, MD and Z axis, respectively.

Note 2: Swelling rate is expressed in %.

4 Thickness uniformity test

4.1 Test equipment

4.1.1 Thickness gauge: The accuracy is not less than 0.1 μm.

Note: See Appendix A for detailed content of test preparation.

4.1.2 Caliper: The accuracy is not less than 0.02 mm; it is used to test the length and width of the membrane.

4.2 Sample preparation and conditioning

4.2.1 Sample preparation

The sample can be square or circular; the effective area is at least 100 cm^2.

The sample shall be free of wrinkles, defects and breakage.

4.2.2 Sample conditioning

Place the samples under the condition where the temperature is 23 °C ± 2 °C and the relative humidity is 50% ± 5% for more than 12 h.

4.3 Test method

4.3.1 The zero point of the thickness gauge shall be calibrated before each measurement, and the zero point shall be rechecked after each sample measurement.

4.3.2 During the measurement, lower the measuring head gently to avoid deformation of the sample. During the test, the strength of the test head applied to the surface of the sample shall be selected between 0.7 N/cm^2 and 2 N/cm^2.

σ – the in-plane proton conductivity of the sample, in Siemens per centimeter (S/cm);

a – the distance between two electrodes, in centimeters (cm);

R – measured impedance of the sample, in ohms (Ω);

b – the effective length of the membrane in the direction perpendicular to the electrode, in centimeters (cm);

d – the thickness of the sample, in centimeters (cm).

Note: Take 3 samples as a group, and calculate the average value as the test result.

6 Equivalent weight (EW) test

Note: See Appendix A for detailed content of test preparation, and Appendix B for content of test report.

6.1 Instruments and equipment

6.1.1 Analytical balance: The division value is not less than 0.1 mg.

6.1.2 Automatic potentiometric titrator: The pH value accuracy is not less than 0.1.

6.2 Sample preparation

Take a sample whose mass is not less than 0.5 g; cut it into pieces, and place it in a vacuum oven. Under the vacuum conditions where the absolute pressure is not higher than 20 kPa and the temperature is 80 °C, vacuum dry it for 8 hours; move it to a desiccator and cool it to room temperature; then, quickly weigh (complete within 30 s) to constant weight. The difference between the two weights shall be less than 0.2 mg.

6.3 Test method

6.3.1 After taking it out of the oven, move it to a desiccator and cool it to room temperature; immediately use an analytical balance to weigh the mass m of the dry membrane.

6.3.2 Put the sample into a sealed reagent bottle that is filled with saturated sodium chloride solution, and stir for 24 h.

6.3.3 Use NaOH standard solution, and use an automatic potentiometric titrator to titrate to neutrality; record the volume V_{NaOH} of the consumed NaOH solution.

6.4 Data processing

Calculate the EW value of the membrane according to Formula (5):

7.1.3 Water bath circulation temperature control device: The temperature control accuracy is ±0.05 °C.

7.2 Sample preparation

7.2.1 The sample shall be representative and free of wrinkles or visible defects. The sample is generally circular; its diameter depends on the instrument used; the number of samples shall meet the requirements of 3 effective parallel tests.

7.2.2 Before the test, the sample shall be dried for at least 4 h at a temperature of 80 °C.

7.3 Test method

7.3.1 Separate the high-pressure chamber and the low-pressure chamber of the differential-pressure gas permeameter, and apply vacuum grease evenly to the area outside the test marking line of the low-pressure chamber test bench.

7.3.2 Place a piece of medium-speed qualitative filter paper, which is cut as required, just above the central cavity of the low-pressure chamber test bench.

7.3.3 Flatly attach the prepared samples to the low-pressure chamber test bench that is coated with grease; ensure that no air bubbles are generated in the contact area between the sample and the grease.

7.3.4 Close the high-pressure chamber and the low-pressure chamber tightly; open the water bath circulation; set the temperature of the temperature control device to 23 °C.

7.3.5 Turn on the power switch of the gas permeameter; open the computer operating software of the instrument; run the replacement process of safe gas (nitrogen or other inert gas) for a time of not less than 600 s.

7.3.6 After the replacement of the safe gas, switch the valve, and pass in high-purity hydrogen; at the same time, turn on the vacuum pump; simultaneously evacuate and degas the high-pressure chamber and the low-pressure chamber to below 10 Pa.

7.3.7 Close the isolation valve, open the test gas cylinder and the gas source switch, and charge the test gas into the high-pressure chamber. The gas pressure in the high-pressure chamber shall be within the range of 1.0×10^5 Pa ~ 1.1×10^5 Pa. When the pressure is too high, the isolation valve shall be opened to discharge.

7.3.8 After the degassing is completed, the instrument automatically closes the exhaust valves of the high- and low-pressure chambers and starts the air permeability test.

7.3.9 Exclude the nonlinear permeation stage at the beginning of the test, and record the pressure change value ΔP of the low-pressure chamber and the test time t.

7.3.10 Continue the test until the pressure change of the low-pressure chamber remains constant within the same time interval, and a stable permeation is achieved. Take at

7.4.3 For a given test apparatus, the low-pressure chamber volume V and the penetration area S of the sample are constants.

7.4.4 The test results are expressed as the arithmetic mean of each group of samples.

8 Tensile property test

Note: See Appendix A for detailed content of test preparation, and Appendix B for content of test report.

8.1 Instruments and equipment

8.1.1 Testing machine

Any testing machine that can meet the test requirements of this chapter is acceptable.

8.1.2 Test fixture

The test fixture shall not cause the sample to break at the fixture. When a load is applied, the longitudinal axis of the sample shall coincide with the tensile direction passing through the centerline of the fixture.

8.1.3 Thickness gauge and caliper

8.1.3.1 Thickness gauge: The accuracy is not less than 0.1 μm.

8.1.3.2 Caliper: The accuracy is not less than 0.02 mm; it is used to test the length and width of the membrane.

8.2 Sample preparation and conditioning

8.2.1 The samples shall be cut at equal intervals along the longitudinal and transverse directions of the material to be tested, and cut into dumbbells or strips of a certain size according to the method specified in GB/T 1040.3-2006. The edge of the sample shall be smooth without gaps. Use a low-power magnifying glass to check the gaps, and discard samples with defective edges.

8.2.2 The samples are grouped according to each test direction, and the number of samples in each group shall meet the requirements of 3 valid tests.

8.2.3 Accurately print or draw a marking according to the sample size requirements. This marking shall have no effect on the sample.

8.2.4 Sample conditioning: The sample shall be placed for at least 4 h under constant temperature and humidity conditions where the temperature is 23 °C ± 2 °C and the relative humidity is 50% ± 5%.

9.1.2 Tensile testing machine

The tensile testing machine shall make the failure load of the sample between 15% and 85% of the full standard load. The force value indication error shall not be greater than 1%. The testing machine shall continuously peel at a rising speed of 300 mm/min ± 10 mm/min or a suitable speed, and shall be able to automatically record the relevant displacement and load.

9.2 Sample preparation and conditioning

9.2.1 Sample preparation

The test sample is in the shape of a long strip; the longitudinal sides are parallel; the width of the sample is 15mm ± 0.1mm, and the length is not less than 250 mm; each group of test strips is not less than 5. Peel off the composite layer and the base material in advance for 50 mm along the direction of the sample; there shall be no obvious damage to the peeled part.

9.2.2 Sample conditioning

The test sample shall be placed for at least 4 h under constant temperature and humidity conditions where the temperature is 23 °C ± 2 °C and the relative humidity is 50% ± 5%.

9.3 Test method

9.3.1 Measure the width of the test sample under constant temperature and humidity conditions where the temperature is 23 °C ± 2 °C and the relative humidity is 50% ± 5%. The width of each sample shall be measured at 3 points within the gauge length, and the average value shall be taken. The width measurement accuracy is ±0.5%.

9.3.2 Clamp the peeled part of the test sample on the upper and lower fixtures of the testing machine; make the longitudinal axis of the peeled part of the test sample coincide with the center line of the upper and lower fixtures, and clamp it. The pressure value of the pneumatic clamp shall be selected in the range of 0.3 MPa ~ 0.7 MPa. During the test, the unpeeled part is T-shaped with the tensile direction, as shown in Figure 4. Record the stress-strain curve of the peeling process.

d_1 – the thickness size of the sample after immersion in a constant temperature water bath, in micrometers (μm);

d_0 – the initial thickness size of the sample, in micrometers (μm).

Note 2: Take 3 samples as a group, and calculate the average value as the test result.

11 Water uptake test

Note: See Appendix A for detailed content of test preparation, and Appendix B for content of test report.

11.1 General

This chapter specifies the method for determining the water uptake of proton exchange membranes for fuel cells under specified dimensions, temperatures and water immersion conditions.

The immersion temperature for the two methods specified in this chapter is 23 °C ± 2 °C and boiling water temperature is 100°C ± 2°C.

11.2 Test equipment

11.2.1 Analytical balance: The division value is 0.1 mg.

11.2.2 Oven: It can be controlled at 80 °C ± 0.2 °C.

11.2.3 Constant temperature water bath: The temperature control accuracy is ±0.2 °C.

11.3 Sample preparation

11.3.1 According to the provisions of GB/T 1446-2005, intercept a square sample whose side length is 50 mm ± 1 mm or a circular sample whose diameter is 50 mm ± 1 mm as the sample to be tested.

11.3.2 The number of samples shall be at least 3, and there shall be no wrinkles, defects and damage.

11.4 Test method

Place the sample in an oven at 80 °C ± 2 °C to dry for 24 h; move to a desiccator and cool to room temperature; use an analytical balance to weigh the initial mass m_0 of the sample.

The test methods for water uptake at 23 °C and 100 °C are as follows:

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