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ePTFE membrane for reinforcement of proton exchange membrane

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1 Scope

This document specifies the technical requirements, the test methods, the inspection rules and the marking, packaging, transport and storage of the polytetrafluoroethylene film used to reinforce proton exchange membranes (referred to below as the PTFE film).

This document applies to the research, production, use and management of polytetrafluoroethylene film used to reinforce proton exchange membranes; other kinds of reinforcing material for ion exchange membranes may be dealt with by reference to it.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through the normative references made to them in the text. For dated references, only the edition corresponding to that date applies to this document; for undated references, the latest edition (including all amendments) applies to this document.

GB/T 191 Packaging - Pictorial marking for handling of goods.

GB/T 20042.3-2022 Proton exchange membrane fuel cell - Part 3: Test method for proton exchange membrane.

GB/T 32361-2015 Test methods for pore size of separation membranes - Bubble point and mean flow method.

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 ePTFE membrane for reinforcement of proton exchange membrane: A film produced from polytetrafluoroethylene resin by a biaxial stretching process, which can be combined

with a proton conducting polymer and reinforces its mechanical strength.

3.2 porosity: The percentage of the volume of the pores of the membrane in the whole volume of the membrane. [SOURCE: GB/T 20103-2006, 2.1.32]

3.3 most probable pore size: The pore size on the pore size distribution curve that corresponds to the point where the percentage is highest. [SOURCE: QB/T 5002-2016, 3.2]

3.4 mass per unit area: The mass per unit area of the film in its natural state. Note 1: this includes the solid part of the film and its open and closed pores. Note 2: the concept is the same as grammage, and the unit is grams per square metre.

3.5 apparent density: The mass per unit volume of the film in its natural state. Note 1: this includes the solid part of the film and its open and closed pores. Note 2: the unit is grams per cubic centimetre.

4 Technical requirements

4.1 Appearance. The surface of the PTFE film shall be flat and even in colour, and shall be free from cracks, indentations, scratches, foreign matter, pinholes and other defects visible to the naked eye.

4.2 Physical properties. The physical properties of the PTFE film shall conform to Table 1. The relative average deviation of the thickness shall be not more than 20.0 %. The mass per unit area shall be from 2.5 to 4.5 grams per square metre. The porosity shall be not less than 75.0 %. The most probable pore size shall be not less than 0.15 micrometres. The tensile strength shall be not less than 15.0 MPa in both the transverse direction (TD) and the machine direction (MD). The elastic modulus shall be not less than 20.0 MPa in both the transverse direction and the machine direction. The tensile strain at break shall be not less than 70.0 % in both the transverse direction and the machine direction.

5 Test methods

5.1 Conditioning of the specimens. The specimens shall be kept for not less than 4 h at a temperature of 23 degrees Celsius plus or minus 2 degrees Celsius and a relative humidity of 50 % plus or minus 5 %.

5.2 Appearance. Remove the outer 0.5 m to 1.0 m of each roll of PTFE film, take a specimen 1 m long in the lengthwise direction and examine it visually under daylight lamps at an illuminance of not less than 400 lx.

5.3.1 Test instrument for the relative average deviation of the thickness. A thickness gauge with an accuracy of not less than 1 micrometre; the pressure the measuring head applies to the surface of the sample shall be not less than 0.7 N per square centimetre and not more than 2.0 N per square centimetre.

5.3.2 Preparation of the sample. Remove the outer 0.5 m to 1.0 m of each roll of PTFE film and cut a full-width piece not less than 0.2 m long in the lengthwise direction; keeping 1 cm clear of the edge of the film, cut the full-width piece lengthwise from left to right into six equal strips to serve as specimens.

5.3.3 Test procedure. Measure the thickness as follows: a) inspect the measuring faces of the thickness gauge and confirm that they are free from oil, dust and other contamination; b) after zeroing the thickness gauge, lower the measuring head gently and measure the thickness evenly at five points; c) after each strip has been measured, check the zero of the thickness gauge again before measuring the next strip; d) complete the measurement of the six strips in this way, giving a total of thirty measuring points.

5.3.4 Processing of the data. The thickness of the specimen is the mean thickness, expressed as the arithmetic mean of all the thickness readings, calculated by formula (1). The relative average deviation of the thickness is calculated by formula (2). In the formulas, Rd is the relative average deviation of the thickness, in percent; xi is the thickness at the i-th measuring point, in micrometres; x with a bar is the mean thickness, in micrometres; and n is the number of measuring points. Round the calculated result to one decimal place.

5.4.1 Test instruments for the mass per unit area and the porosity. An analytical balance with an accuracy of not less than 1 mg; and a thickness gauge with an accuracy of not less than 1 micrometre, the pressure applied by the measuring head to the surface of the sample being not less than 0.7 N per square centimetre and not more than 2.0 N per square centimetre.

5.4.2 Preparation of the sample. Remove the outer 0.5 m to 1.0 m of each roll of PTFE film and take a full-width piece 0.5 m long in the lengthwise direction, the sampling position being not less than 5 cm from the edge of the roll of PTFE film. Place above and below the full-width piece one sheet of A4 paper printed with a grid of 5.0 cm by 5.0 cm squares, and cut ten specimens along the grid, each specimen measuring 5.0 cm by 5.0 cm.

5.4.3 Test method. Pick up the specimens one by one with tweezers and measure the thickness x of each with the thickness gauge, taking three points on each specimen and averaging them, then calculate the mean thickness of the ten specimens. Weigh the specimens one after another on the analytical balance, record the masses and calculate the mean mass of the ten specimens.

5.4.4.1 Mass per unit area. The mass per unit area of the specimen is calculated by formula (3). In the formula, sigma is the mass per unit area, in grams per square metre; m is the mean mass of the specimens, in grams; and A is the area of the specimen, in square centimetres. Round the calculated result to one decimal place.

5.4.4.2 Porosity. The apparent density of the specimen is calculated by formula (4). In the formula, rho is the apparent density, in grams per cubic centimetre; sigma is the mass per unit area, in grams per square metre; and x is the mean thickness of the specimens, in

micrometres. Round the calculated result to one decimal place. The porosity of the specimen is calculated by formula (5). In the formula, epsilon is the porosity, in percent; rho zero is the density of the polytetrafluoroethylene resin, in grams per cubic centimetre; and rho is the apparent density, in grams per cubic centimetre. Round the calculated result to one decimal place.

5.5 Most probable pore size. Test in accordance with the method given in 5.4.1 of GB/T 32361-2015; the equivalent diameter corresponding to the pore size at the point of highest percentage on the pore size distribution curve is the most probable pore size. Round the calculated result to two decimal places.

5.6 Tensile strength, elastic modulus and tensile strain at break. Test in accordance with the method given in Clause 8 of GB/T 20042.3-2022, with the initial separation of the test grips set at 60 mm and a tensile speed of 6 mm/min. Round the calculated results to one decimal place.

6 Inspection rules

6.1 Classification of inspection. Inspection of the product is divided into factory inspection and type inspection.

6.2 Rules for making up a batch. Products of the same specification produced continuously from the same raw material, under the same process conditions and on the same equipment form one batch.

6.3.1 Inspection items and sampling scheme for factory inspection. The inspection items and the sampling scheme for factory inspection shall follow Table 2. Table 2 lists eight inspection items, all of which are required for both factory inspection and type inspection: appearance, tested to 5.2 against the requirement of 4.1; the relative average deviation of the thickness, tested to 5.3; the mass per unit area and the porosity, tested to 5.4; the most probable pore size, tested to 5.5; and the tensile strength, the elastic modulus and the tensile strain at break, tested to 5.6, all seven of these against the requirement of 4.2. For every item the sampling scheme is one roll per batch.

6.3.2 Rules for judgement. Where all the inspection items conform, the batch is judged to conform. Where one item does not meet the requirements of this document, twice the number of samples shall be taken again from the remaining product of that batch and the failing item retested. Where the retest results meet the requirements of this document, the batch is judged to conform; where the retest results still do not meet the requirements of this document, the batch is judged not to conform.

6.4.1 Conditions for type inspection. Type inspection shall be carried out, on at least three consecutive batches, in any of the following cases: a) when a new product goes into production or an old product is being type approved; b) periodically in normal production, or after a certain output has accumulated, once a year; c) when there is a major change in the