NB

ENERGY INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

NB/T 42082-2016

Vanadium flow battery - Test method for electrode

全钒液流电池 电极测试方法

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Issued by: National Energy Administration of the PRC

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1 Scope

NB/T 42082-2016 is the Chinese energy sector standard that fixes how the electrode of a vanadium flow battery is tested. It exists because the flow battery is the technology the Chinese grid is betting on for storage measured in hours rather than minutes, and because in that battery the electrode is not where the energy is kept but where the reaction happens. The energy sits in two tanks of vanadium electrolyte outside the stack, which is why capacity and power can be sized independently; what the electrode has to do is give the vanadium ions a large, wettable, conductive surface on which to change oxidation state, hold that performance for the twenty years the asset is financed over, and do it while

electrolyte is pumped through it continuously. An electrode that is slightly less porous, slightly less conductive or slightly less uniform in thickness than the datasheet claims turns into a round-trip efficiency the project never reaches, and the loss is invisible until the plant is built. This standard is the measuring stick that makes electrode specifications comparable between suppliers. It sets the general requirements and the sample preparation, then defines the test method for each property that matters: thickness uniformity, resistivity, mechanical properties, electrochemical activity, apparent density, surface density and flow resistance, closing with the requirements for the test report. Three informative annexes carry the detail of the flow resistance test and of the test preparation. It was issued on 16 August 2016 by the National Energy Administration and took effect on 1 December 2016.

This standard specifies the general requirements and test methods for electrodes for vanadium flow batteries, mainly including thickness uniformity test, resistivity test, mechanical property test, electrochemical activity test, apparent density test, surface density test, flow resistance test. This standard applies to the test methods for vanadium flow battery electrodes.

1.0 V ~

0.2 V (relative to the voltage value of the saturated calomel electrode).

Note 1: The test process must be carried out under the protection of an inert atmosphere.

Note 2: The recommended amount of electrolyte is not less than 100 mL.

2 Normative references

The following documents are essential for the application of this document. For all dated references, only the dated version applies to this document. For all undated references, the latest version (including all amendments) applies to this document.

GB/T 29840-2013 Vanadium flow battery - Terminology

3 Terms, definitions, symbols

3.1 Terms and definitions The terms and definitions defined in GB/T 29840-2013, as well as the following terms and definitions, apply to this document. For ease of use, some terms and definitions in GB/T 29840 are repeated below.

3.1.1 Electrode The place where electrochemical reaction occurs. [GB/T 29840-2013 Definition 2.6]

3.1.2 Electrochemical activity The ability of the electrode surface to undergo electrochemical reaction. 3.1.3 - Temperature: 25 °C ± 5 °C; - Air humidity: 5% ~ 95%. For tests with special requirements for environmental conditions, the test environment conditions shall be determined by negotiation between the supplier and the buyer.

5 Test preparation

5.1 Sample preparation Sample preparation is as follows: - Samples are randomly selected from the same batch or different batches; - Samples shall be flat and undamaged; - Soak the samples in acetone for at least 1 hour; then dry them in an oven at 120 °C for more than 2 hours and set aside.

5.2 Test instruments and apparatus The instruments and apparatus used in this standard and their accuracy requirements are as follows: - Thickness gauge: Used to measure the thickness of the sample, which has an accuracy of not less than 10 μm; - Caliper: Used to test the length and width of the sample, which has an accuracy of not less than

0.1 mm; - Electronic balance: Used to test the mass of the sample, which has an accuracy of not less than

0.1 mg; - Low resistance tester: Used to test the vertical resistance of the sample, which has an accuracy of not less than

0.01 mOmega; - Mechanical properties testing machine: Used to test the mechanical strength of the sample, which has an accuracy of ±0.5% of its full scale; - Electrochemical tester: Used to test the activity of the sample, which has an accuracy of not less than 1 mA and a voltage accuracy of not less than 1 mV; - Precision pressure controller: Pressure accuracy is ±2% of its full scale; - Stainless steel fixture; - Flat plate forming machine; Where: - The average thickness of the sample under a certain pressure, in millimeters (mm); di - The thickness measurement value of a certain point of the sample under a certain pressure, in millimeters (mm); n - The number of measurement data points.

6.3.2 The thickness standard deviation is calculated by formula (2). Where: sigma - The thickness standard deviation of the sample under a certain pressure, in millimeters (mm); di - The thickness measurement value of a certain point of the sample under a certain pressure, in millimeters (mm); - The average thickness of the sample under a certain pressure, in millimeters (mm); n - The number of measurement data points.

6.3.3 The thickness dispersion coefficient is calculated by formula (3). Where: delta - Coefficient of dispersion, reflecting the degree of dispersion on the unit mean; sigma - Standard deviation of the thickness of the sample under a certain pressure, in millimeters (mm); The sample shall not be larger than the cross-sectional size of the flat fixture of the mechanical properties tester. The recommended sample is round, which has a diameter of 50 mm. The number of samples shall not be less than 3 (to ensure that 3 valid values are obtained).

8.2 Compression rate test

8.2.1 Measure the initial average thickness d0 of the sample when the pressure is close to zero according to the method in Chapter 6.

8.2.2 Install the sample between two smooth flat fixtures; fix the position of the lower clamp; calibrate the corresponding position of the upper clamp when the pressure is close to zero as the displacement origin; the positive direction of the displacement movement is calibrated as vertically downward.

8.2.3 During the test, apply pressure to the outside of the two fixtures. For every increase of

0.01 MPa, record the displacement value lp corresponding to the downward movement of the upper fixture, until the measured displacement value is less than or equal to 5% of the displacement value measured at the previous pressure, it is considered to have reached the minimum value and the test is stopped.

Note: The recommended measurement pressure range is less than

8.3 Data processing Calculate the compression rate of the sample under different pressures according to formula (5). Where: gamma - Compression rate of the sample under a certain pressure, in percentage (%); lp - Thickness of the sample reduced by compression under a certain pressure, in millimeters (mm); d0 - Initial thickness of the sample, i.e. thickness when the pressure is close to zero, in millimeters (mm). Take 3 valid samples as a group and calculate the average value as the test result. Plot the results calculated under different pressures into a compression rate-pressure curve.

9.1 Sample preparation

9.1.1 The sample size (effective area) is 1 cm2 (1 cm x 1 cm); the number of samples is not less than 3 (to ensure 3 valid values). The sample shape and size can also be determined by negotiation between the supplier and the buyer.

9.1.2 Place the sample in two hollow frames of the same size; the size of the hollow part of the frame is 1 cm x 1 cm. Connect the sample to the gold-plated copper sheet, to make a test electrode with airtight edges.

9.2.1 Positive electrode activity test process

9.2.1.1 Test the battery assembled with the sample using the test electrode as the working electrode, the saturated calomel electrode (SCE) as the reference electrode, the platinum sheet or platinum wire as the auxiliary electrode.

9.2.1.2 Use an electrochemical tester to scan the cyclic voltammetry curve of the sample. The electrolyte is a solution containing

0.05 mol/L VO2+,

0.05 mol/L VO2+, 3 mol/L H2SO4. The scan rate is 20 mV/s; the scan range is

0.2 V ~

1.6 V (relative to the voltage value of the saturated calomel electrode).