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Determination of powder resistivity for cathode materials of lithium ion battery

锂离子电池正极材料粉末电阻率的测定

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1 Scope

This document describes the methods for determining the powder resistivity of cathode materials of lithium ion batteries by the four probe method and the two probe method.

This document applies to the determination of the powder resistivity of cathode materials of lithium ion batteries.

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 8170 Rules of rounding off for numerical values and expression and judgement of limiting values.

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 resistivity, rho: The ratio of the electric field strength of the current inside a material to the steady state current density, that is, the volume resistance per unit volume. [SOURCE:

GB/T 40007-2021, 3.2]

3.2 four probe method: A method in which the powder material is pre-pressed to shape and, during measurement with a four probe resistivity meter, the compacted density of the sample either changes continuously or stays constant because pressure is applied continuously or held constant, so that the resistivity of the sample is measured at different compacted densities or at the same compacted density. [SOURCE: GB/T 40007-2021, 3.3, modified]

4 Principle

4.1 Principle of the four probe method. As shown in Figure 1, the cathode powder material is charged into a die with an insulating liner. The upper end face of the sample in the die shall be in close contact with the four probes arranged in a straight line, and the four probes shall press perpendicularly on the upper end face of the sample. A current is passed between the outer probes 1 and 4, and a voltage loop is formed between the inner probes 2 and 3. Where the thickness h of the sample is greater than four times the probe coefficient, the resistivity is calculated by formula (1); where the thickness h is not greater than four times the probe coefficient, the resistivity is calculated by formula (2). In the formulas, rho is the resistivity of the sample, in ohm centimetres; pi is the circular constant, 3.14; l is the probe coefficient, in centimetres; U1 is the voltage measured between the two inner probes, probe 2 and probe 3, in volts; I1 is the direct current applied between the two outer probes, probe 1 and probe 4, in amperes; and F is a correction factor, read from the correction factor table of the instrument according to the sample thickness h, the inner diameter D of the die and the probe coefficient l.

4.2 Principle of the two probe method. As shown in Figure 2, the cathode powder material is charged into a die with an insulating liner. The upper and lower standard electrodes are in close contact with the sample and are connected to the pressing device. Under a given pressure a current I is passed through the two ends of the sample by way of the standard electrodes, a voltmeter measures the voltage U between the upper and lower standard electrodes, and the resistivity is calculated from Ohm's law by formula (3). In the formula, rho is the resistivity of the sample, in ohm centimetres; U2 is the voltage between the upper and lower standard electrodes, in volts; I2 is the direct current between the upper and lower standard electrodes, in amperes; S is the cross-sectional area of the material, in square millimetres; and h is the thickness of the material, in millimetres.

5 Reagents and materials

5.1 Absolute ethanol: analytical grade.

5.2 Dust-free paper.

6 Apparatus

6.1 Electronic balance, with a scale division of 0.0001 g.

6.2.1 Four probe measuring equipment shall consist of the sample die, the pressing system, the thickness measuring system and the resistance measuring system; the layout is shown in Figure 3, and the requirements for its parts are as follows. a) Sample die: a split cylinder whose liner shall be of insulating material; inner diameters of 11.28 mm, 13.00 mm, 15.00 mm, 16.00 mm and 20.00 mm and other sizes are available; the inner diameter of the die shall be not less than ten times the probe coefficient, and the die shall allow the powder sample to be pressed into a thin disc whose thickness is not more than four times the probe coefficient. b) Pressing system: the pressing system shall keep the force or the pressure stable during the test, and the rate at which pressure is applied in repeat tests shall be stable and consistent. c) Thickness measuring system: it shall be able to read the thickness accurately, with a measurement deviation of plus or minus 0.001 mm. d) Resistance measuring system: the four probes are pre-embedded in the insulating material of the pressing column, the probe tips lie in the same plane as the pressing column or disc on the four probe resistance measuring system, and the insulation resistance between the probes, including that between a probe and the housing, is greater than 1 000 megohms.

6.2.2 Two probe measuring equipment shall consist of the sample die, the pressing system, the thickness measuring system and the resistance measuring system; the layout is shown in Figure 4, and the requirements for its parts are as follows. a) Sample die: a split cylinder with an insulating liner containing a metal spacer of hard, well conducting material; inner diameters of 11.28 mm, 13 mm, 15 mm, 16 mm and 20 mm and other sizes are available. b) Pressing system: the pressing system shall keep the force or the pressure stable during the test, and the rate at which pressure is applied in repeat tests shall be stable and consistent. c) Thickness measuring system: it shall be able to read the thickness accurately, with a measurement deviation of plus or minus 0.001 mm. d) Resistance measuring system: the measuring system shall contain a cylindrical or disc-shaped upper and lower electrode, which also serve as the pressing columns or discs; the electrodes shall fit the metal spacer well and their surfaces shall be flat.

6.2.3 An integrated automatic test and data acquisition system may be selected; it contains modules for automatic pressing, testing, data acquisition and calculation, and can output the data automatically.

7 Test sample

The test sample shall be vacuum packed and free from visible caking, agglomeration and moisture pick-up, and the test shall be completed within 2 h of opening the bag.

8 Test conditions

The temperature and humidity of the surroundings shall be kept stable during the test; the temperature should be 25 degrees Celsius plus or minus 5 degrees Celsius and the humidity shall not fluctuate by more than plus or minus 5 %. Where the sample is sensitive to humidity, the test may be carried out in surroundings with a humidity of not more than 10 %.

9 Test procedure

9.1 Preparation of the instrument. 9.1.1 Clean the inner cavity of the die and the fittings of the measuring system with dust-free paper wetted with absolute ethanol (5.1); the cleaning shall be complete and no stain shall be visible to the naked eye on the paper after wiping. 9.1.2 Before the test, inspect the inner cavity of the die and the fittings of the measuring system visually; there shall be no scratches or pits visible to the naked eye, the spacer shall enter the cavity smoothly, and no obvious material shall escape during the test.

9.2 Setting of the parameters. Set the parameters for a single-point or multi-point test in accordance with Table 1 and save the test parameters. Table 1 gives the recommended settings for the classes of sample lithium iron phosphate, lithium cobalt oxide and lithium nickel cobalt manganese oxide, covering the amount of sample, which should be enough to fill between one third and two thirds of the volume of the die cavity, the test pressure in megapascals and the dwell time in seconds. Note 1: the test pressure P is F divided by S, where F is the test force applied to the sample during the test and S is the loaded area of the sample during the test. Note 2: for other classes of sample, refer to the parameters above; to avoid introducing error and to improve stability, test at a high pressure.

9.3 Parallel tests. Carry out three parallel tests and take their mean.

9.4 Determination. 9.4.1 Switch on the equipment before the test and carry out thickness zeroing or baseline verification, or proceed in the way recommended. 9.4.2 Following Table 1, weigh out a given mass of sample on the electronic balance (6.1), load the spacer, the sample and the upper pressing column or disc into the cavity of the die in that order, distribute the sample evenly in the die, and place the die together with the lower pressing column or disc in the pressing system; or proceed in the way recommended for the instrument. 9.4.3 Apply pressure up to the test pressure and hold it for the time given in Table 1. 9.4.4 Apply a current to the material and read the voltage between the two terminals (two probe method) or between the two inner probes (four probe method), or carry out the test in accordance with the operating instructions.

10 Processing of the test data

10.1 Where an integrated automatic test and data acquisition system is used, the powder