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Electrochemical Performance Test of Lithium Manganese Oxide - Test Method for the Initial Specific Discharge Capacity and the Initial Efficiency

锰酸锂电化学性能测试 首次放电比容量及首次充放电效率测试方法

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Electrochemical Performance Test of Lithium Manganese Oxide - Test Method for the Initial Specific Discharge Capacity and the Initial Efficiency

1 Scope

This Standard specifies the test method for the initial specific discharge capacity and the initial charge and discharge efficiency of lithium manganese oxide as the cathode material of lithium ion batteries.

This Standard is applicable to the test of the initial specific discharge capacity and the initial charge and discharge efficiency of lithium manganese oxide as the cathode material of lithium ion batteries.

2 Normative References

This document does not have normative references.

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Test Conditions

When there are no special instructions, the various test procedures specified in this document should be carried out in a drying room (the environmental dew-point temperature is ≤ -20 °C). For the situation where there is no drying room, the various test procedures shall be carried out under the following environmental conditions: the relative humidity is ≤ 40.0% and the temperature is 20 °C ~ 30 °C.

5 Reagents or Materials

5.1 Lithium manganese oxide.

5.2 Conductive agent: acetylene black or carbon black; D_{50} is 1.0 μm ~ 3.0 μm.

5.3 Polyvinylidene fluoride: abbreviated as PVDF; battery grade; molecular weight ≥ 5×10^5; rotational viscosity ≥ 6,000 mPa • s; moisture content ≤ 0.10%.

6.11 Inert atmosphere (argon) glove box: water vapor and oxygen contents (mass fraction) are not greater than 0.0005%.

6.12 Insulating tweezers.

6.13 Liquid injector: 1 mL.

6.14 Button battery sealing and packaging machine.

6.15 Lithium-ion battery electrochemical performance testing machine: 5 V/10 mA, with an accuracy of 0.1%.

6.16 Thermostat: 0 °C ~ 60 °C, with a temperature control accuracy of 0.5 °C.

6.17 Desiccator.

7 Test Procedures

7.1 Pre-treatment of Reagents and Materials

7.1.1 Lithium manganese oxide (5.1), conductive agent (5.2), polyvinylidene fluoride (5.3): place it into the vacuum oven (6.10); at the temperature of 100 °C, bake for 15 h. Then, place it into the desiccator (6.17) to cool down to room temperature.

7.1.2 Lithium ion battery separator (5.7): place it in the vacuum oven (6.10); at the temperature of 75 °C ~ 95 °C, bake for 4 h. Then, take it out and transfer it to the inert atmosphere (argon) glove box (6.11) for storage.

7.1.3 Standard structural components of CR2032 battery (5.9): use ethanol (5.6) to conduct ultrasonic cleaning on the standard structural components of CR2032 battery for 3 times, 30 min each time. After the operation is completed, take out the structural components (5.9) and place them in the vacuum oven (6.10). At 90 °C, dry them for 15 h. Then, transfer them to the inert atmosphere (argon) glove box (6.11) for storage.

7.1.4 Aluminum foil (5.5): use ethanol (5.6) to wipe and clean the aluminum foil.

7.2 Preparation of Cathode Sheet

7.2.1 Glue preparation

In accordance with the mass fraction ratio of 90 : 4 : 6, calculate NMP (5.4), conductive agent (5.2) and polyvinylidene fluoride (PVDF) (5.3); use the electronic balance (6.2) for weighing. The operation procedures are as follows:

a) Add the weighed NMP (5.4) to the beaker under the centrifugal diffuser (6.4); gradually add the weighed polyvinylidene fluoride (5.3); perform dispersive stirring, until it is completely dissolved;

Where,

ρ_c---the compaction density of the cathode sheet, expressed in (g/cm^3);

m_c---the mass of the cathode sheet, expressed in (g);

m_{Al}---the mass of the aluminum foil substrate, expressed in (g);

φ---the diameter of the cathode sheet, expressed in (mm);

d_c---the thickness of the cathode sheet, expressed in (μm);

d_{Al}---the thickness of the aluminum foil substrate, expressed in (μm).

Use the roller machine (6.9) to roll the baked sheet in 7.2.3. When the compaction density of the cathode sheet reaches the design value (generally, 2.2 g/cm^3 ~ 2.8 g/cm^3), stop rolling. Use the sheet-punching machine (6.7) to punch out a sufficient number of cathode sheets with a diameter of 14 mm; place them in the vacuum oven (6.10). At 90 °C, bake for 15 h. Then, use the electronic balance (6.3) for weighing. Calculate the mass (m) of the active substance lithium manganese oxide in the cathode sheet; number and record it. Transfer to the inert atmosphere (argon) glove box (6.11) for storage.

7.3 Battery Assembly

Battery assembly shall be carried out in the inert atmosphere (argon) glove box (6.11). The sequence of the assembly of button batteries, from bottom to top, is: anode shell, lithium metal sheet, lithium ion battery separator, cathode sheet, gasket, spring support sheet and anode shell. The specific operations may take the following steps as a reference:

---Place the anode shell (with the opening facing upwards) flatwise on the surface of the horizontal platform;

---Use the insulating tweezers (6.12) to pick up the lithium metal sheet (5.8) and place it into the anode shell; contact the plane of the anode shell and lay flatwise at the center of the anode shell;

---Use the insulating tweezers (6.12) to pick up the lithium ion battery separator (5.7), so that it completely covers the lithium metal sheet and is centered;

---Use the liquid injector (6.13) to inject 200 μL of the electrolyte (5.10) into the anode shell;

---Use the insulating tweezers (6.12) to pick up the cathode sheet prepared in 7.2.4; place it right in the middle of the lithium ion battery separator (5.7);

---Use the insulating tweezers (6.12) to respectively pick up the gasket and the

7.5 Data Record

After one week of the charge and discharge cycle of the test battery, record the charge and discharge capacity and the mass of the active substance lithium manganese oxide in the corresponding test battery. Calculate the initial specific discharge capacity and the initial charge and discharge efficiency of lithium manganese oxide.

8 Test Data Processing

8.1 Initial Specific Discharge Capacity

The initial specific discharge capacity of lithium manganese oxide is calculated in accordance with Formula (3):

$$C = \frac{Q_{ID}}{m} \qquad \cdots\cdots (3)$$

Where,

C---the initial specific discharge capacity, expressed in (mA • h/g);

Q_{ID}---the initial discharge capacity, expressed in (mA • h);

m---the mass of the active substance lithium manganese oxide in the test battery, expressed in (g).

The calculation result shall retain one decimal place.

8.2 Initial Charge and Discharge Efficiency

The initial charge and discharge efficiency of lithium manganese oxide is calculated in accordance with Formula (4):

$$\eta = \frac{Q_{ID}}{Q_{IC}} \times 100\% \qquad \cdots\cdots (4)$$

Where,

η---the initial charge and discharge efficiency;

Q_{ID}---the initial discharge capacity, expressed in (mA • h);

Q_{IC}---the initial charge capacity, expressed in (mA • h).

The calculation result shall retain one decimal place.

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