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**Electrochemical performance test of lithium nickel
cobalt manganese oxide - Test method for discharge
specific capacity and charge-discharge coulombic
efficiency of the first cycle**

镍钴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 nickel cobalt manganese oxide - Test method for discharge specific capacity and charge-discharge coulombic efficiency of the first cycle

1 Scope

This Standard specifies the test method for discharge specific capacity and charge-discharge coulombic efficiency of the first cycle for lithium nickel cobalt manganese oxide of lithium-ion battery cathode material.

This Standard applies to the testing of discharge specific capacity and charge-discharge coulombic efficiency of the first cycle for lithium nickel cobalt manganese oxide of lithium-ion battery cathode material.

2 Reagents and materials

2.1 Lithium-ion battery electrolyte: Lithium-ion battery electrolyte consisting of lithium hexafluorophosphate (LiPF_6) and mixed carbonate-based organic solvent (volume ratio 1:1:1 of ethylene carbonate EC, dimethyl carbonate DMC, methyl ethyl carbonate EMC); water content $\leq 0.002\%$, free acid (HF) $\leq 0.005\%$, conductivity (25 °C) $\geq 7.0 \text{ mS/cm}$.

2.2 Ethanol: Industrial grade.

2.3 Isopropanol: Analytically pure.

2.4 Polytetrafluoroethylene emulsion binder: PTFE for short, battery grade; solid content $\geq 60\%$.

2.5 Polyvinylidene fluoride: PVDF for short, molecular formula: $-(\text{CF}_2-\text{CH}_2)_n-$, battery grade; weight-average molecular weight $\geq 5 \times 10^5$, rotational viscosity $\geq 6000 \text{ mPa} \cdot \text{s}$, water content $\leq 0.10\%$.

2.6 N-methylpyrrolidone: NMP for short, battery grade; purity $\geq 99.9\%$, water content $\leq 0.02\%$.

2.7 Conductive agent: Acetylene black or carbon black, D_{50} : $1.0 \mu\text{m} \sim 3.0 \mu\text{m}$.

3.11 Lithium-ion battery electrochemical performance tester: 5 V/5 mA.

3.12 Dryer: It contains a suitable desiccant (such as allochroic silica gel, phosphorus pentoxide, etc.).

4 Test procedures

4.1 Raw material pretreatment

4.1.1 Lithium nickel cobalt manganese oxide, conductive agent (2.7): PLACE in an oven (3.2); BAKE at 250 °C ~ 300 °C for 6 h ~ 8 h for drying; PLACE in a dryer (3.12) and COOL to room temperature.

4.1.2 PVDF (2.5): PLACE in an oven (3.2); BAKE at 80 °C ~ 90 °C for 4 h ~ 6 h for drying.

4.1.3 Glass fiber diaphragm (2.11), spring support piece (2.13), gasket (2.14), button battery positive electrode case (2.15): PLACE in oven (3.2); BAKE at 110 °C ~ 120 °C for 6 h ~ 8 h for drying.

4.1.4 Aluminum mesh (2.9): After using ethanol (2.2) to wash repeatedly for 3 times, PLACE in an oven (3.2); BAKE at 110 °C ~ 120 °C for 4 h ~ 6 h for drying.

4.1.5 The button battery negative electrode case is placed in an oven (3.2); and baked at 50 °C ~ 60 °C for 2 h ~ 3 h for drying.

4.2 Positive plate preparation

4.2.1 Preparation of alcohol system positive plate

The instruments and equipment used in the preparation of positive plate shall be clean. The pretreated lithium nickel cobalt manganese oxide, conductive agent (2.7), and PTFE (2.4) (converted to solid content), at a mass ratio of 7:2:1, are weighed using an electronic balance (3.1) and placed in a beaker. Then ADD isopropanol (2.3) (Since isopropanol is volatile, the amount of addition shall be such that it can be prepared to a uniform paste state). USE a stainless-steel spoon to stir and mix well. Then repeatedly ROLL on the roller machine (3.6) to volatilize the isopropanol as quickly as possible, until the thickness is 0.1 mm ~ 0.3 mm. BAKE in the oven (3.2) at 200 °C ~ 220 °C for 10 h ~ 15 h. After drying, USE a pole piece punching machine (3.7) to make a circular positive plate having a diameter of 10 mm ~ 13 mm and a thickness of 0.1 mm ~ 0.3 mm. After weighing, USE the roller machine (3.6) to roll onto a current collector aluminum mesh (2.9). The net weight (m) of lithium nickel cobalt manganese oxide in each positive plate prepared is calculated one by one, numbered and recorded.

(containing aluminum foil) having a thickness of 60 μm ~ 110 μm ; and USE a pole piece punching machine (3.7) to make a circular positive plate having a diameter of $\phi 10\text{ mm}$ ~ $\phi 13\text{ mm}$. The net weight (m) of lithium nickel cobalt manganese oxide in each positive plate prepared is weighed and calculated one by one, numbered and recorded.

4.3 Battery assembly

4.3.1 The battery assembly shall be carried out under the environmental conditions of relative humidity $\leq 40.0\%$ and temperature 20 $^{\circ}\text{C}$ ~ 30 $^{\circ}\text{C}$. The instruments and equipment used shall be clean.

4.3.2 In the argon glove box (3.9), USE the metal lithium sheet (2.10) as the negative electrode; USE the glass fiber diaphragm (2.11) or the lithium-ion battery diaphragm (2.12) as the battery diaphragm; USE the prepared positive plate as the positive electrode; USE the lithium-ion battery electrolyte (2.1) as the electrolyte; and they are assembled into a test battery. After the battery is sealed using button battery packaging machine (3.10), USE dust-free paper (2.16) to wipe; PLACE for 20 min ~ 30 min.

4.3.3 The assembled battery stacking order is from bottom to top: positive electrode case, positive plate, glass fiber diaphragm or lithium-ion battery diaphragm, lithium sheet, gasket, spring support piece, negative electrode case successively. Steps and requirements for assembling batteries:

- The positive electrode case is open upwards and placed flat on a horizontal table;
- The positive plate is placed into the positive electrode case using tweezers so that one side with the aluminum mesh (or aluminum foil) is downward; and is in contact with the plane of the positive electrode case and is flat in the center of the positive electrode case;
- USE tweezers to place two glass fiber diaphragms (2.11) or lithium-ion battery diaphragms (2.12), to completely cover the positive plate and center them;
- USE an injector (3.8) to inject the electrolyte into the positive electrode case of the battery;
- USE tweezers to place the lithium sheet in the middle of the diaphragm;
- USE tweezers to place the gasket and spring support piece; and align-center them with the lithium sheet;
- USE tweezers to lay the negative electrode case to cover the positive

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