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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 42260-2022

Electrochemical performance test of lithium iron phosphate - Test method for cycle life

磷酸铁锂电化学性能测试 循环寿命测试方法

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Standardization Administration of the PRC

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Foreword

This document was issued on 30 December 2022 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 April 2023.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 42260-2022 covers the cycle life test for lithium iron phosphate as a cathode material, not as a finished cell - the ambient conditions the work is carried out under, the reagents and materials, the instruments and equipment, the test steps, the data recording and the cycle life determination, the allowable difference between results, and the test report. The route is fixed from end to end: the powder, characterised by its particle size and specific surface area, is dried in a vacuum oven under nitrogen or argon, combined with a conductive carbon, made into an electrode, rolled in an atmosphere drier than the rest of the work demands, and built into a wound test battery that is then cycled while the discharge capacity of each cycle is recorded against that of the first. Fixing the build is the whole point of the document. Cycle life is the figure a cathode supplier quotes and a cell maker buys on, and it moves with moisture, electrode loading and press density at least as much as with the powder, so two suppliers' numbers mean nothing side by side unless the battery around the material was made the same way. Written for cathode material producers, cell manufacturers and qualification laboratories.

This document describes the test method for cycle life of lithium iron phosphate, i.e., the cathode material for lithium-ion batteries.

This document applies to the test using the winding method for cycle life of lithium iron phosphate, i.e., the cathode material for lithium-ion batteries.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 18287 General specification of lithium-ion cells and batteries for mobile phone

3 Terms and definitions

There are no terms or definitions to be defined in this document.

4 Test conditions

Unless otherwise specified, each test step should be carried out at a relative humidity not greater than 40.0 % and an ambient temperature of 20 degrees C ~ 30 degrees C. The rolling process should be carried out at a relative humidity not greater than 30.0 % and an ambient temperature not greater than 30 °C.

5 Reagents and materials

5.1 Lithium iron phosphate: the particle size characteristic value (D50) is 0.5 um ~ 8.0 um, and the specific surface area is 6 m2/g ~ 30 m2/g.

5.2 Conductive agent: conductive carbon material.

5.3 Polyvinylidene fluoride (PVDF): battery grade, the weight average molecular weight is not less than 5 x 105, and the moisture (mass fraction) is not greater than 0.10 %.

5.4 N-methylpyrrolidone (NMP): battery grade, the purity is not less than 99.9 %, and

the moisture (mass fraction) is not greater than 0.02 %.

5.5 Aluminum foil: the thickness is 8 um ~ 20 um.

5.6 Positive electrode tab (positive terminal): made of aluminum, with tab glue.

5.7 Lithium-ion battery separator: polyolefin porous membrane, the porosity is 35.0 % ~ 60.0 %, the air permeability is 100 s/100 mL ~ 500 s/100 mL, the average pore size is not greater than 1.0 um, and the thickness is 9.0 um ~ 25.0 um.

5.8 Graphite: D50 is 10.0 um ~ 22.0 um, the initial discharge specific capacity is not less than 340.0 mA - h/g, and the initial charge-discharge efficiency is not less than 90.0 %.

5.9 Sodium carboxymethyl cellulose (CMC): the main content (mass fraction) is not less than 99.5 %, and the relative molecular mass is 6.5 x 106.

5.10 Styrene-butadiene rubber emulsion (SBR): water-soluble binder, special for lithium batteries, the solid content is 35 % ~ 52 %, the viscosity is 80 mPa - s ~ 400 mPa - s, and the pH is 6.0 ~ 7.0.

5.11 Deionized water: GB/T 6682, not less than grade three.

5.12 Copper foil: the thickness is 5 um ~ 12 um.

5.13 Negative electrode tab (negative terminal): made of nickel, with tab glue.

5.14 Aluminum plastic film: special for lithium batteries, and the thickness is 110 um ~ 160 um.

5.15 Polyimide tape.

5.16 Lithium-ion battery electrolyte: lithium-ion battery electrolyte composed of lithium hexafluorophosphate (LiPF6) and mixed carbonate-based organic solvents [ethylene carbonate (EC), dimethyl carbonate (DMC), ethyl methyl carbonate (EMC), etc.], the moisture is not greater than 0.002 %, the free acid (HF) is not greater than 0.005 %, and the conductivity (25 degrees C) is not less than 7.0 mS/cm.

5.17 Nitrogen (or argon): the purity (volume fraction) is not less than 99.99 %.

6 Instruments and equipment

6.1 Balance: the accuracy is 0.01 g.

7 Test steps

7.1 Preprocessing

7.1.1 Place lithium iron phosphate (5.1) and conductive agent (5.2) into a vacuum oven (6.3); during drying, vacuum or circulate in a nitrogen (or argon) (5.17) atmosphere; bake at a temperature of 100 degrees C ~ 150 degrees C for 2 h ~ 20 h to dry; cool to room temperature and place in a desiccator (6.4).

7.1.2 Place PVDF (5.3) into a vacuum oven (6.3); during drying, vacuum or circulate in a nitrogen (or argon) (5.17) atmosphere; bake at a temperature of 70 degrees C ~ 90 degrees C for 4 h ~ 6 h to dry; cool to room temperature and place in a desiccator (6.4).

7.2 Preparation of positive electrode sheets

7.2.1 Weighing

Calculate the lithium iron phosphate, conductive agent, and PVDF pretreated in 7.1 according to the mass fractions of 90 % ~ 97 %, 1 % ~ 5 %, and 2 % ~ 5 % respectively, and weigh them with a balance (6.1). Calculate the amount of NMP (5.4) according to the design requirements of solid content (mass fraction) of 40 % ~ 65 %, and weigh with a balance (6.1).

7.2.2 Pulping

The cathode pulping process is as follows:

a) Add the weighed NMP into the mixing tank of a dispersing mixer (6.5); gradually add the weighed PVDF; disperse and stir until completely dissolved; to prepare a transparent glue;

b) Add the weighed conductive agent to the above transparent glue; vacuum, disperse and stir evenly;

c) Gradually add the weighed lithium iron phosphate in portions; vacuum, disperse and stir evenly;

d) Add another NMP according to the designed solid content (mass fraction), to control the slurry viscosity at 4000 mPa - s ~ 8000 mPa - s; vacuum, disperse and