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Lithium iron phosphate-carbon composite cathode materials for lithium ion battery

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Lithium iron phosphate-carbon composite cathode materials for lithium ion battery

1 Scope

This Standard specifies the terms and definitions, classification and codes, technical requirements, test methods, inspection rules and packaging, marking, storage and transportation for lithium iron phosphate-carbon composite cathode materials for lithium ion battery.

This Standard applies to lithium iron phosphate-carbon composite cathode materials for lithium ion battery.

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 191, *Packaging and storage marks*

GB/T 5162, *Metallic powders -- Determination of tap density*

GB/T 6283, *Chemical products -- Determination of water Karl-Fischer method (general method)*

GB/T 6388, *Transport package shipping mark*

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

GB/T 9724, *Chemical reagent -- General rule for the determination of pH*

GB/T 12805, *Laboratory glassware -- Burettes*

GB/T 13732, *General rules for sampling inspection of bulk materials with uniform size*

GB/T 19077.1, *Particle size analysis -- Laser diffraction methods -- Part 1: General principles*

GB/T 19587, *Determination of the specific surface area of solids by gas adsorption using the BET method*

GB/T 20123, *Steel and iron -- Determination of total carbon and sulfur content Infrared absorption method after combustion in an induction furnace (routine method)*

GB/T 24533-2009, *Graphite negative electrode materials for lithium ion battery*

JCPDS (01-077-0179), *Lithium iron phosphate X-ray powder diffraction standard pattern*

IEC 62321, *Electrotechnical products - Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 lithium iron phosphate-carbon composite cathode materials

Lithium-ion battery cathode material composed of lithium iron phosphate and carbon with an olivine structure. Lithium iron phosphate belongs to the orthorhombic crystal system. It has a one-dimensional lithium embedded channel. Lithium ions can be reversibly deintercalated within the crystal lattice. The theoretical specific capacity of the material is 170 mA·h/g.

NOTE: LFP@C mentioned in this Standard all represents lithium iron phosphate-carbon composite cathode materials.

4 Classification and codes

4.1 Product classification

Lithium iron phosphate-carbon composite cathode materials are divided into energy type and power type according to charge and discharge characteristics and usage requirements, represented by LFP@C-E and LFP@C-P respectively. LFP represents lithium iron phosphate, @ represents the composite of two materials, C represents carbon, E represents energy type, and P represents power type:

- Energy type lithium iron phosphate-carbon composite cathode materials (LFP@C-E) are divided into three categories, represented by LFP@C-E-I, LFP@CE-II, and LFP@C-E-III. The specific performance requirements are detailed in Table 2;

- Power type lithium iron phosphate-carbon composite cathode materials (LFP@C-P) are divided into three categories, represented by LFP@C-P-I, LFP@CP-II, and LFP@C-P-III. The specific performance requirements are detailed in Table 2.

6.17 Conductivity

Measure according to the measurement method specified in Annex G.

6.18 Restricted substance content

The measurement is carried out according to the measurement method specified in IEC 62321.

7 Inspection rules

7.1 Sampling method

7.1.1 Sampling

7.1.1.1 Lithium iron phosphate-carbon composite cathode materials for bagged lithium-ion batteries shall be sampled in accordance with GB/T 13732. Open the bag of lithium iron phosphate-carbon composite cathode material to be collected in a dry environment. Use a clean stainless steel sampling drill to insert into the bag along the axis. The insertion depth shall not be less than 4/5 of the depth of the sampling bag. Samples are taken within 20 mm around the central axis of the material in the bag.

7.1.1.2 In order to make the collected samples represent the quality of this batch of products, all collected samples are combined. Place on a square film of sufficient strength and appropriate size or firm, soft drying paper. Mix evenly by rolling repeatedly (turn more than 15 times). The mixed sample shall not be less than 500 g. Cut off two 250 g specimens, one for testing and one for backup.

7.1.2 Sample label

After the sample is placed in the PVC sample jar, a label is immediately attached to the outer wall. The label includes the following:

a) Sample type and number;

b) Overall material batch number and quantity;

c) Sample size;

d) Sampling date;

e) Name of sampler.

7.1.3 Storage of samples

7.1.3.1 Samples shall be sealed and stored in anti-breakage bags, rain-proof, moisture-proof and other environments.

7.1.3.2 The effective storage period of spare samples is 12 months.

7.2 Inspection

7.2.1 Exit-factory inspection

Inspect the particle size, carbon content, moisture content, BET specific surface area, tap density, electrochemical performance, iron ion dissolution amount, conductivity and other indicators of each batch of samples. They can be shipped only after passing the inspection.

7.2.2 Type inspection

All technical requirements specified in this Standard shall be inspected. Type inspection is carried out when one of the following situations occurs:

a) When there are changes in raw material models, supplier manufacturers, etc.;

b) When there are changes in the production process;

c) When the production equipment has been shut down for more than half a year and then starts production for the first time;

d) When customers have special requirements.

7.3 Acceptance rules

7.3.1 Products that meet all the technical indicators required by a certain category in Table 2 are qualified products. If an indicator fails to meet the requirements of this category, double samples shall be taken from the sampling bags of the same batch of products to re-inspect the unqualified items. The re-inspection results shall be taken as the final result. Products that do not fall into the product categories in Table 2 or have special requirements must be determined through negotiation between the supplier and the purchaser to determine whether they are qualified.

7.3.2 The inspection department of the manufacturer shall ensure that the products shipped meet the requirements of this Standard. When each batch of products leaves the factory, a product quality inspection report is sent to the recipient.

7.3.3 The consignee has the right to accept products in accordance with this Standard and the right to reject products that do not meet the requirements of this Standard.

7.3.4 The consignee shall conduct acceptance inspection of the product within one month after receiving the product. If there is any objection, a spare sample shall be used for re-inspection. If there is still a dispute, it will be arbitrated by the superior quality supervision department.

Annex A

(normative)

Method for determination of lithium content (excluding carbon content)

A.1 Scope of application

This appendix is applicable to the determination of lithium content (except carbon content) in specimens by inductively coupled plasma optical emission spectrometry.

A.2 Method summary

The specimen is dissolved in hydrochloric acid. In hydrochloric acid medium, according to the optimized working conditions of the instrument, use the working curve method and the inductively coupled plasma emission spectrometer to measure the lithium ion content.

A.3 Reagents and materials

Without special instructions, the water used in the tests in this Standard refers to water that meets the requirements in GB/T 6682 for the grade three water. All reagents used in the test are analytically pure.

A.3.1 Concentrated hydrochloric acid

ρ 1.19 g/mL.

A.3.2 Argon gas

The volume fraction is not less than 99.999%.

A.3.3 Lithium standard solution

The concentration is 1000 μg/mL. It is a national certified standard sample and can be stored for 1 year.

A.3.4 Preparation of reserve standard solutions

Take 5.00 mL of lithium standard solution (see A.3.3) in a 100 mL volumetric flask. Add 2.00 mL of concentrated hydrochloric acid (see A.3.1). Add water and set the volume constant to the scale. Shake well. Prepare a stock standard solution with a lithium ion concentration of 50.00 μg/mL.

A.3.5 Preparation of series of standard solutions

Accurately measure 0 mL, 1.00 mL, 2.00 mL, 5.00 mL, and 10.00 mL of lithium ion

reserve standard solutions (see A.3.4) and place them into five 100 mL volumetric flasks. Add 2.00 mL of concentrated hydrochloric acid to each (see A.3.1). Add water and set the volume constant to the scale. Shake well. Prepare standard sample blanks and a series of standard solutions with lithium ion concentrations of 0 μg/mL, 0.50 μg/mL, 1.00 μg/mL, 2.50 μg/mL, and 5.00 μg/mL. The ion content in the solution to be tested shall be within the range of the standard curve.

A.4 Instruments and equipment

A.4.1 Inductively coupled plasma emission spectrometer.

A.4.2 Microwave digestion apparatus (including sample tank) or digestion device with equivalent performance.

A.4.3 Electronic balance (resolution: 0.0001 g).

A.5 Analysis steps

A.5.1 Number of measurements

Take two samples for parallel sample testing. Take the arithmetic mean.

A.5.2 Blank test

Do a blank test along with the specimen.

A.5.3 Standard curve

Linear correlation coefficient ≥0.9995.

A.5.4 Preparation of sample solution to be tested

Weigh 0.10 g ± 0.02 g (accurate to 0.0001 g) of specimen into a clean sample jar. Add 9.00 mL of concentrated hydrochloric acid (see A.3.1). Place in microwave digestion apparatus (see A.4.2). After digestion is complete, cool to room temperature. Filter and set the volume constant. Dilute the sample solution to a constant volume appropriately (refer to the preparation method of the reserve standard solution for dilution). Ensure that the lithium ion concentration in the sample solution to be tested is within the standard curve.

A.5.5 Determination

After the instrument is running stably, in accordance with the conditions in Table A.1, inject the standard sample blank and the series of standard solutions (see A.3.6) in sequence for measurement. Draw a curve. Then measure the specimen blank and the sample solution to be tested (see A.5.4) in the same way. Use blank subtraction to correct the results.

Annex B

(normative)

Method for determination of iron content (excluding carbon content)

B.1 Scope of application

This appendix is applicable to the potassium dichromate standard solution titration method for determining the total iron content (except carbon content) in the specimen.

B.2 Method summary

After the specimen is dissolved with perchloric acid, $CuSO_4$-isatin is used as the indicator, and tin dichloride and titanium trichloride are used to reduce the ferric iron to divalent iron. Use sodium diphenylamine sulfonate as indicator and potassium dichromate standard solution for titration test.

B.3 Reagents and materials

Without special instructions, the water used in this Standard test refers to grade three water specified in GB/T 6682, and the reagents used in the test are all analytically pure.

B.3.1 Perchloric acid, ρ 1.76 g/mL

Guaranteed reagent.

B.3.2 Hydrochloric acid, 1:1

Dilute with hydrochloric acid (ρ 1.19 g/mL) and water at a volume ratio of 1:1.

B.3.3 15% sulfuric acid/phosphoric acid mixed acid

Add 150 mL of sulfuric acid (ρ 1.84 g/mL) to 500 mL of water. After cooling, slowly add 150 mL of phosphoric acid (ρ 1.69 g/mL). After cooling, dilute with water to 1 L. Mix well.

B.3.4 Tin dichloride solution, 50 g/L

Weigh 5 g of tin dichloride and dissolve it in 10 mL of hydrochloric acid (see B.3.2). Dilute to 100 mL with water (if the solution is turbid, filter it) and set aside.

B.3.5 Sodium diphenylamine sulfonate indicator

Weigh 0.5 g of sodium diphenylamine sulfonate and dissolve it in water. Dilute with water to 100 mL of water. Mix well and set aside.

B.3.6 Titanium trichloride solution, 20 g/L

Measure 10 mL of 20% $TiCl_3$ and add 20 mL of hydrochloric acid (see B.3.2). Dilute to 100 mL with water. Add a small amount of zinc granules.

B.3.7 $CuSO_4$-isatin indicator

Weigh 0.5 g of indigo. Add 0.5 mL of sulfuric acid (1+4) dropwise. Add water and dissolve in 100 mL of 0.1% copper sulfate solution.

B.3.8 Ferrous ammonium sulfate solution, 45 g/L

Weigh 6.2 g of ferrous ammonium sulfate hexahydrate [$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$]. Dissolve it in sulfuric acid (5+95) solution and dilute to 100 mL. Mix well.

B.3.9 Potassium dichromate standard solution [c (1/6 $K_2Cr_2O_7$) =0.0500 mol/L]

Weigh 2.4518 g of potassium dichromate (standard reagent) that has been dried at 150°C for 1 h. Dissolve in a small amount of water and transfer to a 1 L volumetric flask. Dilute to volume with water. Mix well.

B.4 Instruments and equipment

B.4.1 Electric heater

Working range: 10℃~300℃.

B.4.2 Electronic balance

Resolution: 0.0001 g.

B.4.3 Burette

Capacity is 50 mL; conform to Class A standard in GB/T 12805.

B.5 Analysis steps

B.5.1 Weigh 1.000 g±0.005 g (accurate to 0.0001 g) of specimen into a 100 mL beaker. Add perchloric acid (see B.3.1). Place on an electric hot plate and heat until completely dissolved. Remove and cool to room temperature.

B.5.2 Add water to the cooled solution to make it up to volume in a 100 mL volumetric flask for later use.

B.5.3 Use a pipette to transfer 20 mL of the above solution. Add 30 mL of water and 5 mL of hydrochloric acid (see B.3.2) and place on the electric heating plate to heat until slightly boiling. Add tin dichloride solution (see B.3.4) dropwise while it is hot until it turns light yellow. Add 2 drops of $CuSO_4$-isatin indicator (see B.3.7) to turn green. Add $TiCl_3$ solution (see B.3.6) dropwise until the green color disappears. Overdosage by half

Annex C

(normative)

Method for determination of phosphorus content (excluding carbon content)

C.1 Scope of application

This appendix is applicable to the ammonium phosphomolybdate volumetric method for determining the phosphorus content (except carbon content) in the specimen.

C.2 Method summary

After the specimen is dissolved by perchloric acid, in the nitric acid medium, phosphate radicals and ammonium molybdate form ammonium phosphomolybdate precipitation. After filtration, dissolve the ammonium phosphomolybdate precipitate with sodium hydroxide standard solution. Excess sodium hydroxide standard solution with phenolphthalein as indicator. Back titrate the excess sodium hydroxide standard solution with nitric acid standard solution until the pink color just disappears as the end point (pH≈8).

C.3 Reagents and materials

Without special instructions, the water used in the tests in this Standard refers to meeting the requirements for GB/T 6682 for grade three water, and the reagents used in the tests are all of analytically pure.

C.3.1 Perchloric acid, ρ 1.76 g/mL

Guaranteed reagent.

C.3.2 Potassium nitrate solution, 20 g/L

Dissolve 20 g of analytically pure potassium nitrate in water. Add water to make the volume up to 1 L and shake well.

C.3.3 Ammonium molybdate solution, 70 g/L

Slowly pour Solution A (made by dissolving 70 g of ammonium molybdate in 53 mL of ammonia and 267 mL of water) into Solution B (made by mixing 267 mL of nitric acid and 400 mL of water). Shake well and cool. Add water to make the volume up to 1 L. Let stand for 12 h.

C.3.4 Nitric acid standard solution

Concentration: 0.1 mol/L.

Annex F

(normative)

Method for determination of first coulombic efficiency, first reversible specific capacity, rate performance

F.1 Scope of application

This appendix is applicable to the first coulombic efficiency, first reversible specific capacity, and rate performance of the test specimen of the battery test system.

F.2 Method summary

Use metallic lithium as the negative electrode and the specimen electrode piece as the positive electrode to assemble a simulated battery or button battery. Use a battery test system at 23°C ±2°C to test. Test the first reversible specific capacity (lithium insertion) and first coulombic efficiency of the simulated battery or button battery. The rate performance is calculated based on the discharge specific capacity (lithium insertion) at different rate discharge currents.

F.3 Reagents and materials

F.3.1 Conductive agent

Acetylene black or conductive carbon black with equivalent performance.

F.3.2 Solvent

N-methylpyrrolidone (NMP), electronic grade.

F.3.3 Binder solution, 5%

Dissolve 5.000 g ± 0.001 g (accurate to 0.0001 g) of polyvinylidene fluoride (PVDF) powder in 95.000 g ± 0.005 g (accurate to 0.0001 g) of N-methylpyrrolidone (see F.3.2) to obtain 5% viscosity binder solution. It is recommended that the molecular weight of polyvinylidene fluoride is about 1.1 million.

F.3.4 Metal lithium sheet or lithium strip

They shall meet battery grade lithium requirements.

F.3.5 Aluminum foil

The aluminum foil thickness is 20 μm.

F.3.6 Current collector

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