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GB/T 33818-2017

Carbon nanotube conductive paste

碳纳米管导电浆料

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Issued on: May 31, 2017

Implemented on: December 1, 2017

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration Committee.

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed by the Chinese Academy of Science.

This standard is under the jurisdiction of the Sub Technical Committee of Nanomaterial (TC279/SC1) of the National Technical Standardization Committee of Nanotechnology.

The drafting organizations of this standard: Beijing Cnano Technology Limited, Shenzhen BTR Nano Technology Co., Ltd., Hefei Guoxuan High-tech power energy Co. Ltd., Cnano (Zhenjiang) Material Technology Ltd, Shenzhen Jinzhaoshidai New Energy Technology Co. Ltd, Metallurgical Industry Information Standards Institute, etc.

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Carbon nanotube conductive paste

1 Scope

This standard specifies the terms, requirements and testing methods and rules, as well as marking, packaging, transportation, storage and order contents of carbon nanotube conductive paste.

This standard applies to quality inspection and acceptance of liquid phase system products using multi-walled carbon nanotubes as conductive medium in the fields of lithium ion batteries, conductive coatings and conductive adhesives, etc. Single wall carbon nanotubes paste products can be implemented with reference to this standard.

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 606 Chemical reagent - General method for the determination of water - Karl Fischer method

GB/T 1725 Paints, varnishes and plastics - Determination of non-volatile-matter content

GB/T 6753.1 Paints, varnishes and printing inks. Determination of fineness of grind

GB/T 22235-2008 Determination for viscosity of liquids

GB/T 24490 Test method for purity of multi-walled carbon nanotubes

GB/T 24491 Multi-walled carbon nanotubes

QB/T 2303.10 Paste coated paper separator for batteries. Part 10: Determination of pH

3 Terms and definitions

The following terms and definitions apply to this document.

calibration of the standard sample.

4.3.2 The total content of magnetic foreign bodies (Fe, Cr, Ni, Zn, Co) in the paste should not exceed 0.002% (mass fraction) of the carbon tube content.

4.3.3 The content of soluble metallic impurities (Fe, Cr, Ni, Cu, Zn, Co) in the paste should not exceed 0.2% (mass fraction) of the carbon tube content. If the user has special requirements, it may be determined by consultation of both supply-demand parties.

4.3.4 The water content in the anhydrous system should not be more than 2000×10^{-6} (mass fraction). If the user has special requirements, it may be determined by consultation of both supply-demand parties.

4.3.5 The pH value of water-based paste should generally be within the range of 5.0~9.0.

5 Test methods

5.1 Appearance

Visual inspection.

5.2 Solid content

Measure and calculate as per GB/T 1725.

5.3 Content of carbon nanotubes

5.3.1 Make a sample of dried paste according to the steps specified in 5.2.

5.3.2 Measure and calculate the content of carbon nanotubes in accordance with the steps specified in GB/T 24490.

5.4 Paste fineness

Measure as per GB/T 6753.1.

5.5 Transmittance

5.5.1 Instruments and materials

Ultrasonic cleaner, UV-VIS spectrophotometer, high speed mixer (maximum speed at least 3000rpm), cuvette and other auxiliary appliances.

Four-probe resistivity meter, air dry oven, press machine, glass plate, standard scraper coater, high temperature resistant polyester film (PET film) and other auxiliary tools.

5.6.2 Preparation of sample slice

5.6.2.1 Seal the sample in a self-sealing bag, then stir for 5min with a glass bar to make the paste in an even state.

5.6.2.2 Drop some ethanol on a clean glass plate, lay the PET film on the glass plate, and wipe the PET film with paper to make the film tightly attach to the glass plate. Put appropriate amount of paste sample on the PET film, use standard scraper coater (gap 200 μ m) to scrape the paste to be flat.

5.6.2.3 Place it in the air-dry oven at 80°C~120°C to dry for 1h~2h.

5.6.2.4 Cut the dried sample film into a thin wafer of 15mm diameter, make more than 2 pieces. Put it in the press machine, press 3min~5min under the sustained pressure of 10t.

5.6.2.5 Use a spiral micrometer to measure the thickness of the compacted paste on the sample film. Measure 3 different points and take the average value.

5.6.2.6 Use the four-probe resistivity meter to test the volume resistivity of the sample, select the appropriate range, input the thickness of the paste and the diameter of the wafer (15mm) to the parameter settings of the tester, other parameters are automatically selected by the tester.

5.6.3 Result calculation

5.6.3.1 Tests 2 slices for the same sample. Each slice should be tested in 3 different points for volume resistivity data. If the deviation between the two-test data is not more than 10%, then the test data is valid.

5.6.3.2 Use the four-probe resistivity meter's own software to process the data, get the result, or directly take the arithmetic average value.

5.6.3.3 If the deviation between the two-test data is more than 10%, the test data is invalid. Retest should be carried out; if necessary, the sample can be reproduced.

5.7 Viscosity

It is measured according to the requirements of rotary viscosity measurement method in GB/T 22235-2008.

6.4 Inspection and acceptance

6.4.1 Products shall be inspected by the supplier's quality supervision department to ensure that the quality of the product meets the requirements of this standard or order contract, and the quality certificate should be filled in.

6.4.2 When the paste product is not qualified in appearance inspection, the quality of the products is deemed not qualified.

6.4.3 If the paste product is not qualified in any test of the composition, fineness, relative absorbance and the conductivity etc., the product is not qualified.

6.4.4 The demand-party should inspect the received product according to this standard; if the test result does not comply with this standard or the order contract, it should raise the issue to the supplier within 15 days from the receipt date of the product, and seek settlement through negotiation of both parties. If arbitration is required, arbitration shall be conducted at the place of the demand-side with joint participation of both parties.

7 Marking, packaging, transportation, storage

7.1 Packing and marking

7.1.1 After inspection, the qualified products are packed generally 20L~50L as a package. The packing should be tight; the outer wall of the packing bucket should be labeled with exit-warehouse label with information as follows:

- a. Supply number (supply list number of the goods supplied);
- b. The brand and name of the company;
- c. Name and specification;
- d. Date of production;
- e. Contact information and address of the company.

7.1.2 If the customer has specific requirements, corresponding packaging and logo design can be carried out.

7.1.3 When supplied, every batch of products should be accompanied with the <carbon nanotubes conductive paste inspection report>, with content including the relevant technical parameters of the batch products.

7.1.4 Each batch of products is accompanied by <shipping list>, with content including

A.3.2.2 Take out the magnetic rod, put it into a 250mL beaker, and add deionized water to do ultrasonic cleaning for 30s. Wash 3 to 5 times until the cleaning liquid is clear (After cleaning, the directly visually inspect the magnetic rod, carefully observe if the magnetic rod surface has obvious metal particles; if obvious, this sample should be paid special attention when conducting test for data).

A.3.2.3 Take two 100mL beakers, one without any sample, as blank samples. Put the cleaned magnetic rod into the other beaker. Added 60mL of dilute aqua regia (1:1) (the rod is just immersed) in each of the beakers; boil until the liquid is nearly dry, add small amount of deionized water.

A.3.2.4 When the boiled liquid is cooled to room temperature, filter it into a 100mL volumetric flask with a funnel. Wash the beaker and the magnetic rod with deionized water for 2 to 3 times. The cleaning liquid is also transferred to the volumetric flask, meter the volume, shake it up and wait for test.

A.3.3 Test

Use ICP-OES to test the content of Fe, Zn, Ni, Cr and Co.

A.4 Result representation method

A.4.1 Magnetic foreign-matter content is the sum of the 5-metals content of Fe, Zn, Ni, Cr and Co.

A.4.2 Parallel test for 3 times. If the deviation of each set of test data is no more than 10%, the test data is valid, the result is the arithmetic average.

A.4.3 If the deviation of each set of test data is more than 10%, the test data is invalid. It should be retested; the sample can be reproduced when necessary.