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Multi-walled carbon nanotubes

多壁碳纳米管

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Table of Contents

Foreword 3
1 Scope 4
2 Normative references 4
3 Terms and definitions 4
4 Classification 5
5 Technical requirements 5
6 Test methods 6
7 Inspection rules 7
8 Packaging, marks and quality certificates 8
9 Storage and transportation 9
10 Safety precautions 10
Annex A (normative) Determination of average outer diameter and length of multi-walled carbon nanotubes 11

Multi-walled carbon nanotubes

1 Scope

This Standard specifies the terms and definitions, classification, technical requirements, test methods, inspection rules, packaging, marks as well as quality certificates, storage and transportation, safety precautions for multi-walled carbon nanotubes.

This Standard applies to multi-walled carbon nanotube powder products.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 3780.8, *Carbon black - Part 8: Determination of heating loss* (GB/T 3780.8-2008, ISO 1126:2006, MOD)

GB/T 10722, *Carbon black - Determination of total and external surface area by nitrogen adsorption*

GB/T 19619, *Terminology for nanomaterials*

GB/T 24490-2009, *Test method for purity of multi-walled carbon nanotubes*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 19619 as well as the followings apply.

3.1 multi-walled carbon nanotubes; MWCNTs

A hollow quasi-one-dimensional tubular nanocarbon material that is coaxially nested by curling three or more layers of graphite sheets.

4 Classification

Multi-walled carbon nanotubes are divided into three categories according to their average outer diameter:

- Category 1: the average outer diameter ≤ 20 nm.

- Category 2: the average outer diameter > 20 nm ~ 50 nm.

- Category 3: the average outer diameter > 50 nm ~ 150 nm.

Each category is divided into four grades according to the content (mass fraction) of multi-walled carbon nanotubes:

- Grade I: the content is greater than 98%.

- Grade II: the content is greater than 95%.

- Grade III: the content is greater than 85%.

- Grade IV: the content is greater than 70%.

5 Technical requirements

5.1 Basic features

a) Appearance: Black powder or granule, without obvious agglomeration.

b) Morphology: Observed with transmission electron microscope magnification over 100,000 times, multi-walled carbon nanotubes are fibrous. The ratio of length to diameter is greater than 20. Along the axis of the fiber, it is a hollow tube. There can be a bamboo-like partition inside the hollow tube. The graphite sheets on the tube wall can have a certain inclination relative to the central axis of the tube.

5.2 Requirements

The technical requirements of multi-walled carbon nanotubes shall meet the requirements in Table 1.

7 Inspection rules

7.1 Inspection classification

The inspection specified in this Standard is divided into exit-factory inspection and type inspection.

7.1.1 Exit-factory inspection

Each batch of products shall be inspected for appearance and morphology, average outer diameter, multi-walled carbon nanotube content, and ash content.

7.1.2 Type inspection

Type inspection shall be carried out randomly when one of the following situations occurs:

a) When there is a change in the batch number, model, supplier, etc. of raw materials;

b) When there is a change in the production process;

c) When the normal production lasts for 3 months;

d) When the production equipment stops production for more than 3 months and starts production for the first time;

e) When customers make special requirements.

All technical requirements specified in this Standard shall be inspected.

7.2 Inspection and acceptance rules

7.2.1 Inspection and acceptance

7.2.1.1 The exit-factory inspection and acceptance of multi-walled carbon nanotubes shall be carried out by the quality and technical supervision department of the supplier. The supplier shall ensure that the delivered multi-walled carbon nanotubes comply with the provisions of this Standard or the contract.

7.2.1.2 The buyer has the right to inspect and accept any inspection item specified in this Standard or the contract. The acceptance inspection shall be carried out within 15 days from the date of delivery of the goods.

7.2.2 Batching rules

Multi-walled carbon nanotubes shall be submitted in batches for acceptance. Each batch consists of multi-walled carbon nanotubes with the same production process, the same outer diameter, and the same quality grade. The net weight of each batch of products

bag and seal it. Then put a layer of polyethylene sealing bag outside the sealing bag. Seal it. The net weight of each package is 1.0 kg or 5.0 kg. If there is any special packaging requirement, it can be negotiated by both parties.

8.2 Each bag shall have a firm mark on the front. The mark contains the following information:

a) Product name;

b) Trademark;

c) Reference to this Standard, product quality grade;

d) Net weight, batch number;

e) Name and address of the manufacturer;

f) Words or signs such as "moisture-proof" and "fire-proof";

g) Production date, production batch number or exit-factory date and serial number.

8.3 Each batch of multi-walled carbon nanotube products shall be accompanied by a quality certificate in compliance with the order contract and product standards. The quality certificate shall have the following information:

a) Name, address and contact information of the manufacturer;

b) Product name;

c) Product batch number and batch weight;

d) Various test results;

e) Production date and inspection date;

f) Reference to this Standard.

9 Storage and transportation

9.1 Multi-walled carbon nanotube products shall be stored in a ventilated, cool and dry warehouse. The stacking shall be neat. Heavy pressure is strictly prohibited.

9.2 There shall be coverings during the transportation of multi-walled carbon nanotube products. Load and unload the products lightly. Prevent package damage. Prevent rain and moisture. Do not mix with strong oxidants.

Annex A

(normative)

Determination of average outer diameter and length of multi-walled carbon nanotubes

A.1 Principle

Transmission electron microscopy can magnify the sample image hundreds of thousands or even millions of times. Point resolution can reach 0.1 nm. During the test, the electron beam passes through the sample through the lens system, which can reflect the shape, microstructure and impurity content of the sample. This method can qualitatively analyze the samples as multi-walled carbon nanotubes. It can quantitatively determine the inner and outer diameters and lengths of multi-walled carbon nanotubes. At the same time, it can quantitatively test the number of particulate matter in the multi-walled carbon nanotube sample.

A.2 Reagents

Ethanol: analytically pure.

A.3 Devices

a) Transmission electron microscope: the point resolution is less than 0.3 nm.

b) Ultrasonic cleaner: 100 W.

c) Desiccator: Φ300 glass desiccator.

A.4 Specimen preparation and storage

a) Put about 0.1 mg of multi-walled carbon nanotubes into an agate mortar. After grinding it, put it in a specimen bottle.

b) Add 10 mL of ethanol to the specimen bottle. After capping the sample bottle, put the sample bottle into the ultrasonic cleaner and sonicate for 20 min.

c) Use a pipette with a diameter of less than 3 mm to pipette a little suspension. Drop it on the surface of the previously prepared microgrid for transmission electron microscopy. Dry in air.

d) Put the microgrid that carries the sample into a sample box. Then transfer the sample box to a desiccator for storage.

A.5 Test procedures

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