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TECHNICAL SPECIFICATION

Nanomanufacturing – Key control characteristics – Part 2-1: Carbon nanotube materials – Film resistance

colour inside

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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FOREWORD

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- the subject is still under technical development or where, for any other reason, there is the future but no immediate possibility of an agreement on an International Standard.

Technical specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC 62607-2-1, which is a technical specification, has been prepared by IEC technical committee 113: Nanotechnology standardization for electrical and electronic products and systems:

The text of this technical specification is based on the following documents:

Enquiry draft	Report on voting
113/118/DTS	113/131/RVC

Full information on the voting for the approval of this technical specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts of the IEC 62607 series, published under the general title *Nanomanufacturing – Key control characteristics,* can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- transformed into an International standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

There are two major trends in the fabrication of new materials incorporating carbon nanotubes (CNTs) for next generation of industrial applications:

a) conducting composites in field-emission displays (FEDs), flexible displays, or printed electronics; and
b) nano-composites for mechanical applications, by taking advantage of their attractive mechanical properties such as high Young's modulus, elastic behaviour and high tensile strength.

This IEC technical specification is related to a), the conducting composites application. As conducting composites using CNTs are increasingly being used in the electronics industry, it is essential to establish a standard method to evaluate their electrical properties.

Characterization of the electrical properties of CNTs used in conducting composites is important to both manufacturers and users. This IEC technical specification describes simple methods to characterize the electrical properties of CNT materials that are to be used in conducting composites.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 2-1: Carbon nanotube materials – Film resistance

1 Scope

This part of IEC 62607 provides a standardized method for categorizing a grade of commercial CNTs in terms of their electrical properties to enable a user to select a CNT material suitable for their application. The method is intended to assess whether the delivered materials from different production batches of the same production process are comparable regarding electrical properties of the final product which are related to electrical conductivity. The correlation between the measured parameters by the proposed method and a relevant product performance parameter has to be established for every application. This specification includes

a) definitions of terminology used in this document,

b) recommendations for sample preparation,

c) outlines of the experimental procedures to measure sheet resistance of CNTs in thin films,

d) methods of interpretation of results and discussion of data analysis,

e) case studies and,

f) references.

2 Terms, definitions, acronyms and abbreviations

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

NOTE A comprehensive nanotechnology vocabulary is under ongoing development in IEC TC113/ISO TC229 Joint Working Group 1 in cooperation with ISO/TC 229. The vocabulary is/will be published as different parts of IEC/ISO/TS 80004. This document will be harmonized with the terms and definitions of TS 80004 prior to publication and later on during the maintenance of the document. Definitions not yet specified are taken from scientific literature.

2.1 Terms and definitions

2.1.1
single-wall carbon nanotube
SWCNT
carbon nanotube consisting of a single cylindrical graphene layer

Note 1 to entry: Its structure corresponds to a graphene sheet rolled up into a seamless honeycomb structure around a cylinder.

[SOURCE: ISO/TS 80004-3:2010, definition 4.4]

2.1.2
multiwall carbon nanotube
MWCNT
carbon nanotube composed of nested, concentric or near-concentric graphene sheets with interlayer distances similar to those of graphite

Note 1 to entry: Its structure is considered to be many single-wall carbon nanotubes nesting each other, and would be cylindrical for small diameters but tends to have a polygonal cross section as the diameter increases.

[SOURCE: ISO/TS 80004-3:2010, definition 4.6]

2.1.3
CNT film
film of SWCNT and/or MWCNT formed by non-destructive methods such as filtration on a substrate, etc.

SEE: Figure 1(c).

2.1.4
sheet resistance
Rs
measure of resistance of thin films that are nominally uniform in thickness

Note 1 to entry: Two-dimensional (x-y) sheet resistance (R_s) can be determined for electrically uniform thin films. In rectangular geometry $R_s = R/(L/w)$, where R is the measured resistance, $R = V/I$, L is the distance between parallel electrodes, between which the voltage drop (V) is measured, and w is the length of these electrodes. The electrical current (I) must flow along the plane of the sheet, not perpendicular to it (see Figure 4). The ratio L/w represents the number of squares of the film specimen. The unit of sheet resistance is expressed in ohms (Ω). However, for the purpose of this procedure, Ω shall represent the unit ohm/square (Ω/sq).

Note 2 to entry: See [1-4 [1]]

2.1.5
I-V characteristic
relationship between an electric current and a corresponding voltage, or potential difference typically represented as a chart or graph

2.1.6
4-probe measurement
method to measure the resistance of a material whose measured value is independent on the probe resistance

Note 1 to entry: In this method, 4 probes contact the test sample in a linear arrangement. A voltage drop is measured between the two inner probes while a current source supplies current through the outer probes. The resistance of the sample can be calculated by Ohm's law. Furthermore, the resistivity of the sample can be obtained by the consideration of the geometric factors of the sample. See references [3,4].

2.1.7
4-wire measurement
type of 4-probe measurement defined in 3.1.6 in which a wire is used as a probe

2.1.8
4-point measurement
type of 4-probe measurement defined in 3.1.6 in which a pointed electric tip is used as a probe

Note 1 to entry: A 4-point measurement is generally used to measure sheet resistance of a thin-film sample with relatively large width compared to the spacing between the probes.

2.2 Acronyms and abbreviations

DMF: N,N-dimethylformamide

THF: Tetrahydrofuran

DCE: Dichloroethane

PVDF: Polyvinylidene fluoride

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1 Numbers in square brackets refer to the Bibliography.

3 Sample preparation methods

3.1 General

For 4-probe measurements, a powder-like CNT product should be manipulated into a pellet or film sample [5-6]. A film sample is preferred because with a pellet sample, high pressure may induce deformation and change the intrinsic properties of the CNTs. For the purpose of this standard, it is critical to fabricate a uniform film over a large area, avoiding any external forces that might alter the measurements significantly. Two aspects are important in preparation of uniform CNT films for 4-probe measurements: (i) selecting a proper dispersant; and (ii) determining the amount of CNTs to use for thin-film formation. If it is difficult to prepare uniform CNT films with suitable geometric factors for electrical measurements, the film may be tailored into ribbon form.

3.2 Reagents

3.2.1 Carbon nanotubes

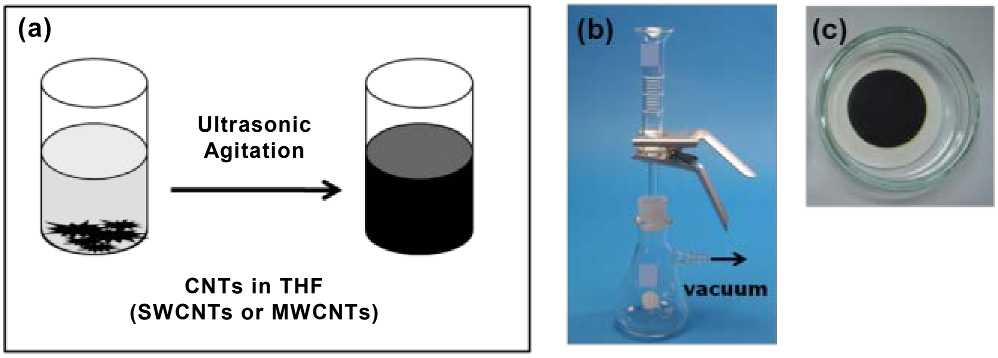
SWCNTs or MWCNTs in the as-received condition shall be used for this test, and with no additional conditioning performed.

3.2.2 Dispersants

THF is recommended as the standard dispersant by comparing its function with that of other organic dispersants such as DMF, ethanol or 1,2-dichloroethane, which are commonly used for CNT dispersion [7-8]. Among these dispersants, THF makes homogeneously dispersed CNT suspensions, helps to minimize CNT surface damage during the sonication step, and can be removed effectively after film formation. Spectrophotometric grade (> 99,8 %) is recommended to minimize contamination of the CNT. The test results obtained from each dispersant are compared and summarized in Annex A, Table A.1.

3.3 Preparation of SWCNT or MWCNT films

Disperse 2 mg of SWCNTs or MWCNTs in 20 ml of THF by ultrasonic treatment (bath type, 40 kHz) for 30 min at 25 °C. Filter the resultant suspension under vacuum using a 220 nm pore-PVDF membrane (disc diameter: 25 mm) to form a thin film, and then dry it for 12 h at 80 °C. See Figure 1. The film thickness of the resulting CNT films was 50 ± 1 μm and the film diameter was 18 mm. See Clauses A.2 and A.3.



(a) Procedure for dispersing CNTs in THF

(b) Filtration apparatus

(c) Resultant CNT film after filtration through PVDF membrane with 25 mm diameter and 220-nm pore size

Figure 1 – Preparation of SWCNT and MWCNT films

3.4 Preparation of SWCNT or MWCNT ribbons

Ribbon-type samples are prepared by tailoring the SWCNT or MWCNT film using an antistatic cutter to a size suitable for 4-wire measurements. The recommended size is 1~2 mm wide × ~10 mm long.

4 Measurement of sheet resistance of SWCNT or MWCNT films

4.1 4-point measurement

4.1.1 Demarcation of method

This method is applicable for measuring sheet resistance of SWCNT or MWCNT films that maintain their uniformity in shape and flatness during sample preparation and measurement.

4.1.2 Experimental procedures and measurement conditions

A schematic of a 4-point probe configuration and a picture of a probe card are shown in Figure 2. The 4-point setup consists of four equally spaced platinum metal tips with uniform tip radius. Typical probe spacing is 1 mm. The current source (*A*) supplies current through the outer two probes, and a voltmeter (*V*) measures the voltage across the inner two probes (Figure 2(a)) to determine the sample resistance. The voltmeter must be of high input impedance. Otherwise, equations (1) and (2) shown in Clause 6 cannot be used.