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TECHNICAL SPECIFICATION

colour inside

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

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

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ELECTROTECHNICAL
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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 2-1: Carbon nanotube materials – Film resistance

FOREWORD

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- the required support cannot be obtained for the publication of an International Standard, despite repeated efforts, or;
- 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- withdrawn,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

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

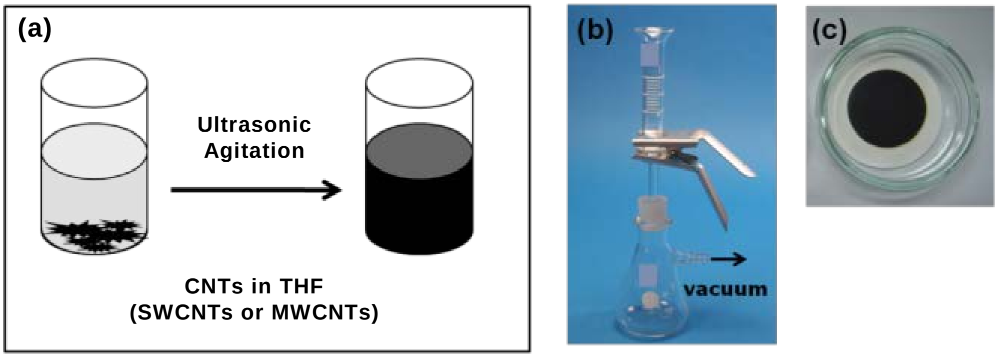
SWCNTs or MWCNTs in the as-received condition shall be used for this test, and with no additional conditioning performed.

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 $\pm$ 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

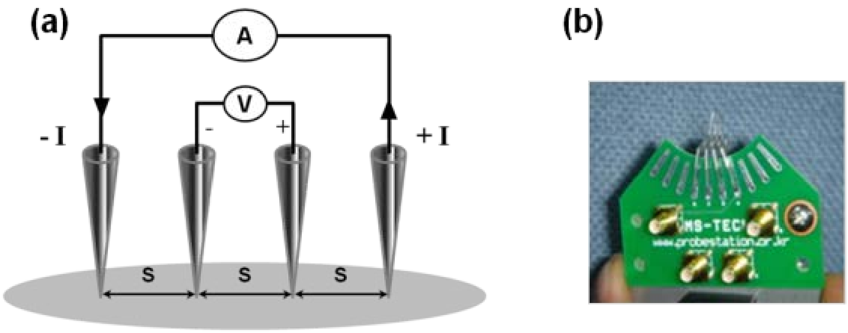
4.1 4-point measurement

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.



(a) Schematic diagram of four-point probe configuration, where ‘s’ is the probe spacing

(b) Photo of a typical four-point probe card.

Figure 2 – 4-point probe

A commercially available probe station may be used for the measurement (see Figure 3). Place the SWCNT or MWCNT film onto a height-controllable sample stage. Make contact the probe tips with the SWCNT or MWCNT films through adjustment of the height of the stage. Confirm physical contact between the point probe and sample surface with an optical microscope. The spacing ‘s’ between adjacent probes is uniformly 1 mm. Apply a low current (maximum 1 μA) during the measurement to avoid damage to the sample.

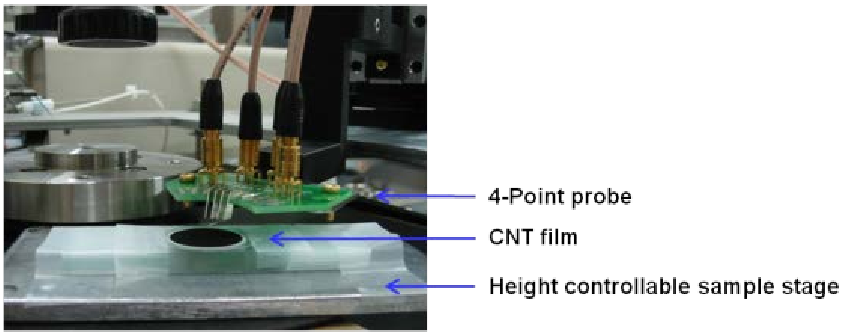


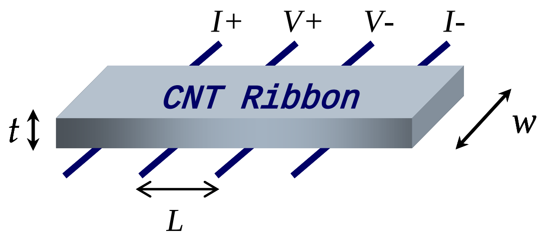
Figure 3 – Photo of typical 4-point measurement apparatus

4.2 4-wire measurement

4.2.1 Demarcation of method

This method is applicable to a SWCNT or MWCNT ribbon.

4.2.2 Experimental procedures and measurement conditions



Key

L = Spacing between adjacent probes

t = thickness of CNT ribbon

w = width of CNT ribbon.

Figure 4 – Diagram for 4-wire measurement apparatus

A convenient fixture for resistance measurements is shown in Figure 4. Perpendicularly fix the 4 Pt (platinum) wires with 0,1 mm diameter onto an insulating substrate with $L = 3$ mm spacing between each wire. Place the specimen onto the electrodes to make electrical contact without damaging the specimen. Apply a low current (maximum 1 μA) for the measurement in order to avoid damage to the sample.

5 Data analysis / Interpretation of results

5.1 Sheet resistance of SWCNTs or MWCNTs using 4-point measurements

Calculate sheet resistance using 4-point measurements as follows:

$$R_s = F \times \frac{V}{I} \tag{1}$$

Where:

R_s = sheet resistance,

V = measured voltage,

I = applied current,

V/I = the gradient of a plot of V versus I, and

F = geometrical correction factor [9,10].

In the case when the sample size is much larger than the spacing between the electrodes 's' (see Figure 2), $F = (\pi/\ln 2) = 4{,}53236$. For other specific cases see Figure 1 and Table 1 in [9]. For example, results with better than 99 % accuracy can be obtained by measuring in the centre of a circle with a diameter greater than 40 s; and results with better than 1 % error can be obtained by measuring in the centre of a circle with a diameter greater than 100 s.

5.2 Sheet resistance of SWCNTs or MWCNTs using 4-wire measurements

Calculate sheet resistance in 4-wire measurements as follows:

$$R_s = \left(\frac{w}{L}\right)\left(\frac{V}{I}\right) \tag{2}$$

Where:

R_s = sheet resistance,

V = measured voltage,

I = applied current,

V/I = the gradient of a plot of V versus I,

w = specimen width measured using a calibrated optical microscope, and

L = distance between wires.

Annex A
(informative)

Case study

A.1 Sample preparation

A.1.1 Single-wall and multiwall carbon nanotubes (SWCNTs and MWCNTs)

SWCNTs were obtained from 2 different manufacturers. MWCNTs were obtained from 3 different manufacturers. The CNTs in the as-received condition were used for this entire test.

A.1.2 Choice of dispersant

The organic dispersants used to disperse the CNTs were DMF, THF, and 1,2-DCE. Among these dispersants, THF was chosen as the best dispersant for dispersion after consideration of proper handling and dispersability. THF provides a well-dispersed CNT suspension, minimizes CNT surface damage during the sonication step, and dries rapidly to make a thin film.

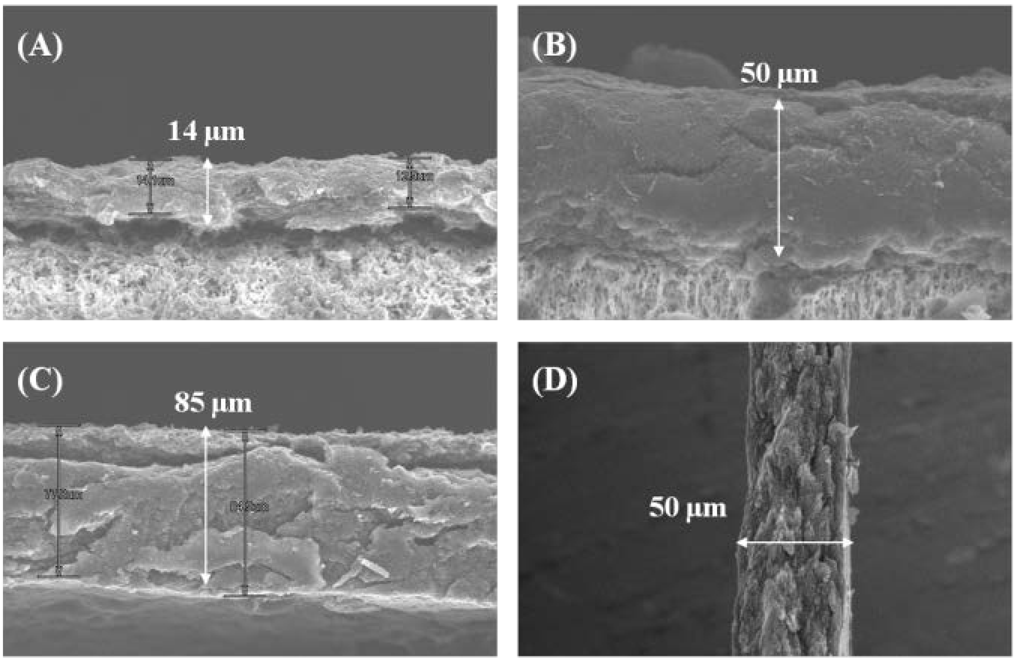
The properties considered in the selection of the best dispersant used to disperse CNTs and prepare thin-film samples are summarized in Table A.1.

Table A.1 – Properties of dispersants used to prepare thin-film samples.

	THF	DMF	1,2-DCE
Dispersion of CNTs	Good	Varied by types of CNTs	Good
Effect on electronic structure of CNTs during ultrasonic treatment in dispersant	No effect [7]	Effect (strong chemical disruption of the π bonding of nanotubes) [11]	Effect (Cl_2 or HCl doping) [12]
Speed of evaporation	Fast	Very slow	Fast

A.2 Determination of quantity of SWCNTs and/or MWCNTs

To determine a quantity of CNT to produce a CNT film of uniform thickness, it was tested that a specified quantity of CNTs dispersed in a predetermined volume of dispersant. When 1 mg of SWCNTs or MWCNTs was used, film thicknesses varied widely between 10 μm and 50 μm. The film thickness was relatively well-controlled to (90 ± 5) μm with a quantity (mass) of 5 mg, but the film became fragile, especially when mechanical force was applied to make the ribbon forms. 2 mg of SWCNTs or MWCNTs produced the most uniform film ((50 ± 1) μm) with homogeneously-bundled SWCNTs or MWCNTs, and were robust in the ribbon shape for the purpose of 4-probe measurements. From the results, it proposed that 2 mg of CNTs is a suitable quantity to produce CNT films with uniform thickness. Thicknesses of SWCNT or MWCNT ribbons were characterized by FE-SEM (field emission-scanning electron microscopy) as shown in Figure A.1.



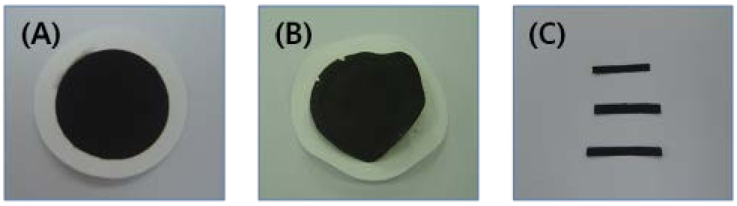
CNT ribbons on membranes were prepared using (A) 1 mg, (B) and (D) 2 mg, and (C) 5 mg of CNTs in 20 ml THF. (D) is a side view of a free-standing CNT ribbon which has uniform thickness.

Figure A.1 – FE-SEM images of CNT ribbons

A.3 Preparation of CNT films and ribbons

2 mg of SWCNTs or MWCNTs were dispersed in 20 ml THF with ultrasonic treatment (bath type, 40 kHz) for 30 min at 25 ºC. The SWCNT or MWCNT suspension was filtrated under vacuum using 220 nm pore-sized PVDF membranes (disc diameter: 25 mm) to form a thin film, then dried at 80 ºC for 12 h. The film diameter of the resulting CNT films was 18 mm.

The dispersion and filtration method was used to prepare CNT films with uniform film thickness and large area (see (A) of figure A.2) suitable for 4-point measurements. In some instances, uniform CNT films with suitable geometric factors for 4-point measurements were difficult to produce. A sample with curled edges is shown in (B) of Figure A.2. In these circumstances, the films were formed into ribbons (See (C) of Figure A.2). Then a 4-wire measurement was used to extract the sheet resistance.



(A) CNT film having uniform film thickness suitable for 4-point measurement, (B) a curled-edge CNT film not suitable for 4-point measurement, and (C) CNT ribbons produced from the sample in (B) for 4-wire measurement

Figure A.2 – Photos of fabricated CNT specimens

A.4 Results of sheet resistance (*Rs*) of carbon nanotubes

Table A.2 shows the measured *Rs* of SWCNT and MWCNT ribbons from 5 different vendors using 4-wire measurements. Each CNT material was measured with five ribbon samples prepared from five different films. Sheet resistance for each CNT sample is relatively similar.

The results show that homogeneous CNT ribbons can be fabricated through the test method described in this standard.

Table A.2 – Resistance and sheet resistance of MWCNTs and SWCNTs ribbons

CNT	Units	1	2	3	4	5	Combined relative uncertainty No. ± n(%)[a]
MWCNT (A)	R (Ω)	19,03	27,27	27,04	20,83	20,38	
	R_s (Ω/sq.)	5,45	5,45	5,41	5,42	5,43	5,43 ± 0,37 %
MWCNT (B)	R (Ω)	2 080	1 920	1 860	1 680	1 310	
	R_s (Ω/sq.)	693,3	672,0	620,0	616,0	679,5	656,17 ± 5,44 %
MWCNT (C)	R (Ω)	226,8	185,6	210,3	225,4	202,6	
	R_s (Ω/sq.)	83,92	89,09	92,53	78,89	83,07	85,50 ± 6,26 %
SWCNT (D)	R (Ω)	9,55	7,0	7,4	7,6	6,4	
	R_s (Ω/sq.)	1,43	1,40	1,53	1,52	1,79	1,53 ± 9,80 %
SWCNT (E)	R (Ω)	38,9	36,0	52,1	38,2	36,1	
	R_s (Ω/sq.)	14,00	12,60	18,24	16,43	14,44	15,10 ± 14,64 %

[a] n(%)covers all relative uncertainties, where n is the standard deviation/average × 100.

Table A.3 shows a comparison of the results between 4-point measurements of CNT films and 4-wire measurements of CNT ribbons prepared from the same vendor material. For 4-point measurements, both the centre and edge of the film were measured. The results from the 4-point and 4-wire measurements were nearly identical, indicating that the measured sheet resistance is not affected by the measurement method when specimen preparation conditions are the same.

Table A.3 – Results of 4-point measurements of CNT films and 4-wire measurements of CNT ribbons using the same sample preparation

Methods	*Sheet resistance, R_S*
4-point	5,45 (centre) and 5,45 (edge)
4-wire	5,43 ± 0,02 (Average)

- Specimen parameter conditions

For 4-point measurement: sample diameter was 18 mm and probe space (s) was 1,0 mm.

For 4-wire measurement: spacing between adjacent probes (L) was 3 mm and widths of CNT samples (w) were in a range between 0,6 mm and 0,8 mm.

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