IEC TS 62607-2-5

Edition 1.0 2022-11

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 2-5: Carbon nanotube materials – Mass density of vertically-aligned carbon nanotubes: X-ray absorption method

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The text of this Technical Specification is based on the following documents:

Draft	Report on voting
113/674/DTS	113/696/RVDTS

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts in 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 document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Vertically-aligned carbon nanotubes (VACNTs) are array structures, in which nanotubes are oriented in the perpendicular direction to a substrate surface. VACNTs are useful in many electronic device applications such as field-emission devices, gas and biological sensors, thermal interface materials, supercapacitors, and so on. Chemical vapour deposition (CVD) is one of the common methods for the synthesis of VACNTs, where CNTs can be grown in the presence of metal catalysts, via thermal decomposition of hydrocarbon sources such as methane, ethylene, acetylene, ethanol, and so on.

Physical (electrical, thermal, etc.) properties of VACNT films really depend on their density, which is reflected by distribution and alignment behaviours of individual CNTs. The mass density of nanotubes in VACNT samples was evaluated in various ways. The first choice is measuring the sample mass gain, which is successively divided by the height and the area of the VACNT samples for obtaining density values. However, this mass gain method is a destructive method, and is effective only if the mass of CNTs can be measured with a microbalance, so that the mass density can be estimated from the mass gain during the CVD growth. The second method is counting the number of CNTs in scanning electron microscope (SEM) or transmission electron microscope (TEM) images. However, this counting method is less reliable when the nanotubes are not grown straight on the substrate and the density is low. Liquid-induced compaction can compact the VACNT samples to a maximum density with wetting or drying process of alcohols. However, these methods are destructive analyses (except for SEM) and are not designed for incorporating the wide distribution in size and alignment of nanotubes observed in realistic VACNT samples. Hence, there is strong demand for the development of new reliable methods for evaluating density in VACNTs.

In this context, an X-ray absorption method is proposed as a standard protocol for evaluating density of VACNTs. X-rays can transmit through the film parallel to the substrate surface, and the transmitted X-rays are detected by a high-resolution X-ray imaging apparatus. The observed X-ray projection images can enable the substrate, VACNT film, and air regions to be identified easily. The film density can be calculated from the measured X-ray transmittance of the film. This method is an effective and versatile technique of nondestructive analysis for VACNT film density.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 2-5: Carbon nanotube materials – Mass density of vertically-aligned carbon nanotubes: X-ray absorption method

1 Scope

This part of IEC 62607 specifies the protocols for determining the mass density of vertically-aligned carbon nanotubes (VACNTs) by X-ray absorption method. This document outlines experimental procedures, data formats, and some case studies. These protocols are applicable to VACNT films with thickness larger than several tens of micrometres. There are no limitations in materials for substrate.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

There are no normative references in this document.

3 Terms, definitions, and abbreviated terms

3.1 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1.1
carbon nanotube
CNT
nanotube composed of carbon

[SOURCE: ISO/TS 80004-3:2020 [1], 3.3.3, modified – Note 1 to entry has been deleted.]

3.1.2
single-walled carbon nanotube
SWCNT
carbon nanotube consisting of a single cylindrical graphene layer

[SOURCE: ISO/TS 80004-3:2020, 3.3.4, modified – The term "single-wall carbon nanotube" and Note 1 to entry have been deleted.]

3.1.3
multi-walled carbon nanotube
MWCNT
carbon nanotube composed of nested, concentric or near-concentric graphene layers with interlayer distances similar to those of graphite

[SOURCE: ISO/TS 80004-3:2020, 3.3.6, modified – The term "multiwall carbon nanotube" and Note 1 to entry have been deleted.]

3.1.4
vertically-aligned carbon nanotubes
VACNTs
carbon nanotube bundle grown in the perpendicular direction to a substrate surface

3.2 Abbreviated terms

CVD chemical vapour deposition

SEM scanning electron microscope

4 Measurement of mass density of vertically-aligned carbon nanotubes with X-ray absorption method

4.1 General

X-ray is well known for its high penetrating power and has been widely used for nondestructive inspection of various materials. In addition, the absorption (transmission) rate of every element has been extensively studied for a long time and tabulated with very good accuracy. Hence, when the elemental composition of the material is known, the density of the thin film material can be calculated from the measured transmittance of X-rays in a nondestructive way.

4.2 Measurement principle

Figure 1 presents the principle for measuring density of VACNT film by X-ray absorption method. The absorption of X-rays by the VACNT film is expressed as follows: [2][1]

$$I = I_0 \exp\left[-\mu_\rho (E,Z)\rho l\right] \tag{1}$$

where I_0 and I are incident and transmitted X-ray intensity, respectively. μ_ρ is the mass attenuation coefficient of the material , ρ is the mass density of the sample, and l is the path length of X-ray. μ is a physical parameter as a function of the X-ray energy (E) and the elemental composition (Z).

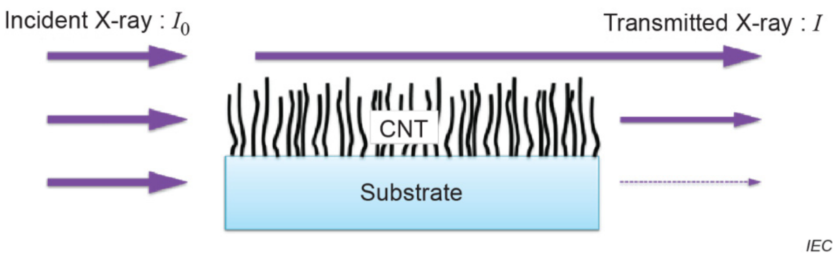

Figure 1 – Measurement principle of X-ray absorption method

[1] Numbers in square brackets refer to the Bibliography.

4.3 Description of measurement equipment and apparatus

In order to measure absorption of the VACNTs, the incident X-ray should be completely parallel to the substrate and the spatial resolution of the measurement should be high enough to detect transmitted X-ray signals separately from substrate, VACNT, and outer surface areas [3]. The spatial resolution of the present method Δ_R is determined by the convolution of beam divergence factor and spatial resolution of the X-ray detector as follows:

$$\Delta_R = \sqrt{\left[\delta_B \left(l + d_{SD}\right)\right]^2 + R^2} , \qquad (2)$$

where δ_B is the X-ray beam divergence, l is the path length of the sample, d_{SD} the distance from the substrate to the X-ray detector, and R is the spatial resolution of the X-ray detector, as depicted in Figure 2.

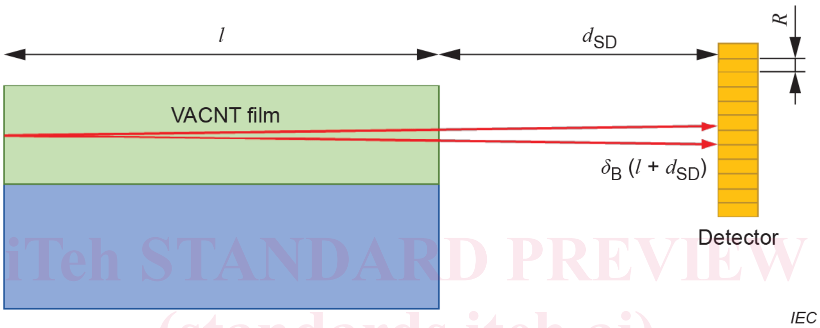

Figure 2 – Parameters determining the spatial resolution of X-ray absorption method

4.4 Sample preparation

VACNT films are usually prepared by using a variety of chemical vapour deposition (CVD) methods such as plasma-enhanced CVD [4], thermal CVD [5], water-assisted CVD [6], and so on. At first, metal catalyst nanoparticles are deposited onto a substrate by physical vapour deposition methods (e.g. sputtering, thermal deposition, and electron-beam deposition) or chemical processes including reduction of metal oxides or oxides solid solutions. After that, the substrate is put into a CVD chamber and heated to a certain CNT growth temperature of several hundred degrees Celsius, followed by the introduction of hydrocarbon sources (e.g. methane, ethylene, acetylene, or alcohols) and process gases (e.g. nitrogen, hydrogen, or ammonia) into the reaction chamber. Then, VACNT films are grown in the presence of metal catalysts via thermal decomposition of hydrocarbon sources. An actual example of VACNT film preparation using CVD is given in Annex A.

4.5 Thickness measurement with X-ray absorption method

Figure 3 shows an X-ray projection image of VACNTs grown on Si substrate, which is a representative result obtained with X-ray absorption method. Three regions can be clearly recognized: substrate, VACNT film, and air. For example, the thickness of the VACNT film presented in Figure 3 can be measured to be approximately 120 µm from the image. Specific stripe patterns can be seen along the air–film interface in Figure 3, which are derived from refraction of X-ray photons near the edge of the film surface due to the difference in X-ray refractive indices between VACNT and air.