IEC TS 62607-2-6

Edition 1.0 2024-08

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 2-6: Carbon nanotube-related products – Thermal diffusivity of vertically-aligned carbon nanotubes: flash method

IEC TS 62607-2-6:2024-08(en)

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

Draft	Report on voting
113/823/DTS	113/845/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

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INTRODUCTION

Vertically-aligned carbon nanotubes (VACNTs) possess array structures, in which nanotubes are oriented in the perpendicular direction to a substrate surface. 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. VACNTs are promising as thermal interface materials in electronics assembly owing to their high thermal conductivity, desirable mechanical properties, and good stability. Thermal transport properties in VACNT films really depend on their distribution and alignment behaviours of individual nanotubes, disorders such as defects and impurities.

Thermal diffusivity is one of the key parameters that govern thermal transport properties in solid materials. Flash method is a well-established, standard technique for measuring the thermal diffusivity. Originally, flash method was applicable to homogeneous monolithic (single layer) samples. In fact, some previous works reported thermal diffusivity measurements for self-standing VACNTs that were peeled off from the substrates after the CNT growth. However, VACNT films will be tightly connected to solid substrates in possible practical applications such as thermal interface materials. This means that flash method can not be simply applied to VACNT films grown on solid substrates. Hence, there is a need for new reliable protocols based on flash method for evaluating thermal diffusivity of VACNT films on solid substrates. This document specifies standardized protocols for measuring thermal diffusivity of VACNTs grown on solid substrates with flash method, where the specimen is a bilayer of the VACNT film and the substrate.

NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –

Part 2-6: Carbon nanotube-related products –
Thermal diffusivity of vertically-aligned carbon nanotubes: flash method

1 Scope

This part of IEC 62607 specifies a protocol for determining the key control characteristic

- thermal diffusivity

for vertically-aligned carbon nanotube (VACNT) films grown on solid substrates by

- flash method.

A light pulse from a flash lamp or a laser is irradiated onto the front surface (substrate side) of the VACNT film on solid substrates. Then, the temperature change of the other side of the specimen is monitored in real time after the pulse irradiation. The thermal diffusivity of the VACNT film can be analysed from the time variation of this temperature change.

– This method is applicable for evaluating the thermal transport properties of the VACNT films that can be used as thermal interface materials in electronics assembly.

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.

IEC TS 62607-2-5:2022, *Nanomanufacturing – Key control characteristics – Carbon nanotube materials – Mass density of vertically-aligned carbon nanotubes: X-ray absorption method*

ISO 18755:2022, *Fine ceramics (advanced ceramics, advanced technical ceramics) – Determination of thermal diffusivity of monolithic ceramics by flash method*

3 Terms, definitions and abbreviated terms

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 Terms and definitions

3.1.1
carbon nanotube
CNT
nanotube composed of carbon

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

3.1.2
vertically-aligned carbon nanotubes
VACNTs
carbon nanotube bundle grown in the perpendicular direction to a substrate surface

[SOURCE: IEC TS 62607-2-5:2022, 3.1.4]

3.1.3
thickness
h
dimension of the test specimen in the direction of heat transfer measurement

[SOURCE: ISO 19629:2018, 3.3]

3.1.4
mass density
ρ
at a given point within a three-dimensional domain of quasi-infinitesimal volume dV, scalar quantity equal to the mass dm within the domain divided by the volume dV

$\rho = \mathrm{d}m/\mathrm{d}V$

[SOURCE: IEC 60050-113:2011, 113-03-07, modified – The formula has been moved to a new line.]

3.1.5
specific heat capacity
C
heat capacity divided by mass

[SOURCE: IEC 60050-113:2011, 113-04-48, modified – The formula and Notes have been deleted.]

3.1.6
volumetric heat capacity
heat capacity divided by volume

3.1.7
thermal diffusivity
α
thermal conductivity divided by the volumetric heat capacity

3.1.8
thermal conductivity
k
density of heat flow rate divided by temperature gradient under steady state condition

Note 1 to entry: Thermal conductivity is calculated by using the equation $k = \alpha \rho C$.

[SOURCE: ISO 18755:2005, 3.2, modified – Note 1 to entry has been added.]

3.1.9
thermal effusivity
b
heat transport property given by the product of volumetric heat capacity and square root of thermal diffusivity

3.1.10
thermal diffusion time
τ
square of thickness divided by thermal diffusivity

3.1.11
maximum temperature rise
ΔT_{max}
difference between the steady temperature before the pulse heating and the maximum temperature of the rear face of the specimen after the pulse heating

[SOURCE: ISO 18755:2022, 3.10, modified – Note 1 to entry has been deleted.]

3.2 Abbreviated terms

CVD chemical vapour deposition

SEM scanning electron microscope

4 Measurement of thermal diffusivity of vertically-aligned carbon nanotubes on solid substrates with flash method

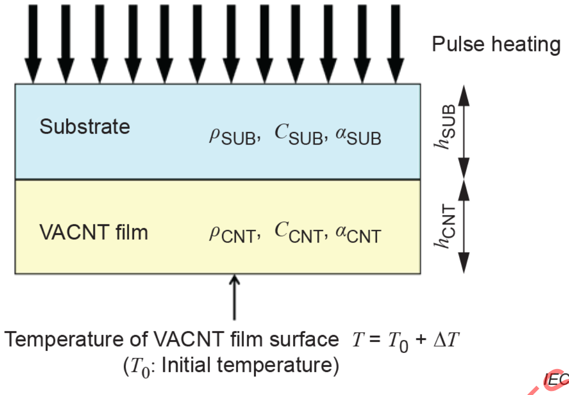
4.1 General

Flash method prevails as a well-established, standard technique for measuring the thermal diffusivity of solid materials [1][1]. A light pulse from a flash lamp or a laser is irradiated onto one side of a monolithic planar specimen under adiabatic conditions. Then, the temperature change of the other side of the specimen is monitored in real time after the pulse irradiation. The thermal diffusivity of the solid can be calculated from the time variation of this temperature change. In this document, flash method is applied to VACNT films grown on solid substrates. In this case, the specimen is a bilayer of the VACNT film and the substrate. Accordingly, it is desirable that the measurement protocols for the transient temperature curve are based on 4.2 to 4.7. Case studies of measuring thermal diffusivity of VACNT films grown on Si substrate are provided in Annex A. In addition, rough estimation of thermal conductivity of VACNT films from the measured thermal diffusivity values is described in Annex B.

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

4.2 Measurement principle

Figure 1 presents the schematic diagram of the flash measurement for the VACNT film grown on a solid substrate. A short light pulse is irradiated onto the front surface (substrate side) of the specimen and the energy of the light pulse is absorbed by the substrate. Successively, the heat conduction toward the VACNT film occurs and the resulting temperature rise in the rear surface (VACNT side), which is denoted as ΔT, is observed as shown in Figure 2.



Key

ρ mass density

C specific heat capacity

α thermal diffusivity

h thickness

Figure 1 – Schematic diagram of flash method for VACNT film on substrate

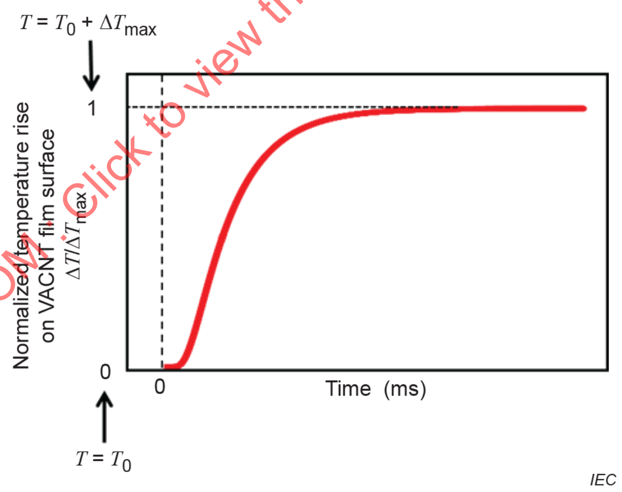


Figure 2 – Typical transient temperature curve for VACNT film on substrate

In Figure 2, an example of temporal variation in normalized temperature rise ($\Delta T/\Delta T_{max}$) is given. The origin of the horizontal axis is the time of light pulse irradiation onto the substrate. The baseline of the measured transient temperature curve corresponds to the initial temperature of the VACNT film surface, denoted as T_0. ΔT_{max} is the maximum temperature rise after the pulse heating. The thermal diffusivity of VACNT film can be calculated by utilizing an analytical model considered for the bilayer sample structure.

4.3 Thickness and mass density measurement for VACNT film

For measuring thickness and mass density of VACNT film grown on solid substrates, it is desirable to use non-destructive (non-contact) methods for each specimen in order to avoid any mechanical damage to the specimens. In this point, IEC TS 62607-2-5:2022 shall be applied.

4.4 Coating substrate surface

When a flash lamp or a laser is used as a light source in the flash measurement for VACNT films on substrate, a possible fraction of the incident light transmitted through the substrate can cause an undesirable heat transfer in the specimen system of measurement and result in measurement errors. Silicon wafers are widely used as substrate for VACNT film growth with CVD technique. Care should be taken for the possible transmission of incident light through the silicon substrate because silicon is considerably transparent for infrared light.

Therefore, when the substrate is transmissive for the incident light, coating the front surface (substrate side) with metal (for instance, gold and aluminium) is essential. The thickness in the range of several tens of nanometres is sufficient in the case of metal coating, which can cut off the transmission with a negligible influence on measurement results with flash method. Additional coating of highly absorptive material on the substrate such as black spray containing carbon particles is useful for enhancing incident light absorption of the substrate. The carbon layer shall be as thin as possible in a manner such that the effect of the carbon layer on the physical properties of the substrate can be ignored.

For obtaining an appropriately coated substrate surface, ISO 18755:2022, 5.3 shall be applied.

4.5 Incident light pulse

The duration of incident light pulse should be less than 1 % of the thermal diffusion time of the sample. Otherwise, the centre of light intensity distribution should be identified as the starting point of time.

The area covered by the incident light on the front surface of sample is sufficiently larger than the area at which the temperature measurement is made.

The distribution of incident light intensity over the front surface of sample should be as spatially as uniform as possible, so that one dimensional heat flow is ensured to take place in the sample.

It should be possible to change the incident light pulse energy to evaluate the dependency of measured thermal diffusivity on the incident light pulse energy.

To appropriately determine the conditions of the incident light pulse, ISO 18755:2022, 6.6 shall be applied.

4.6 Temperature measurement at the rear surface of sample

The response of the thermometer should be faster compared with the thermal diffusion time of the sample.

An infrared radiation thermometer should be used for the measurements of the rear surface of sample.

The bare surface of VACNT film usually looks black in the visible and the emissivity is close to unity in the infrared as well. Therefore, any coating on the rear surface of VACNT film is unnecessary for infrared radiation thermometers.

It is not recommended to use a thermocouple attached on the rear surface of sample because the response is usually too slow for typical VACNT films to be measured accurately.

4.7 Measurement environment

Measurements for VACNT films can be conducted in air, under vacuum, and in an inert gas atmosphere. The measurement atmosphere will not give a significant effect on the measurement results for VACNTs, because they comprise highly-oriented nanotubes and have high thermal diffusivities. However, it is recommended to give care to the measurement environment for reliable assessment of the thermal diffusivity.

5 Data analysis

5.1 Bilayer analytical model

Transient temperature curves measured for bilayer structures are analysed by employing a mathematical solution of conventional, one-dimensional heat diffusion equation (see Clause A.3). The example of the bilayer analytical model and its mathematical solution are provided in Clause A.4.

5.2 Evaluation of thermal diffusivity of VACNT film

As seen in Clause A.4, the analytical solution of temporal variation in $\Delta T/\Delta T_{\text{max}}$ includes various parameters such as mass density (ρ_{SUB} and ρ_{CNT}), specific heat capacity (C_{SUB} and C_{CNT}), thickness (h_{SUB} and h_{CNT}) and thermal diffusivity (α_{SUB} and α_{CNT}) for each layer. Hence, all physical parameters except for the thermal diffusivity of the VACNT film (α_{CNT}) should be obtained before analysing the transient temperature curves.

After the data other than α_{CNT} are substituted into the physical parameters in the analytical formula of the transient temperature curve, the α_{CNT} value shall be evaluated by least squares fitting method. In this calculation procedure, the reliability of the data analysis shall be checked by examining if the measured temperature change is well fitted to the theoretical curve. Attention should be paid if a large difference between the measured curve and the fitting result is found.

6 Reporting

All items listed in Table 1 shall be reported, where the table format is an example. Additionally, it is recommended to show transient temperature rise curves measured for VACNT films on substrates.

If there are deviations from this document, give detailed procedures used and their justification.

Table 1 – Example table format for reporting

Item	Data
Parameters used	Preparation method of the VACNT sample: [ ] Type of the substrate: [ ] Lateral size of the substrate [ ] mm × [ ] mm Thickness of the substrate: [ ] mm Type of metal coating and thickness: [ ], [ ] nm Mass density of the substrate [ ] kg m^{-3} Specific heat capacity of the substrate [ ] J kg^{-1} K^{-1} Thermal diffusivity of the substrate [ ] m^2s^{-1} Initial temperature [ ] K
Measurement results	Thickness of the VACNT film [ ] µm ± [ ] ∝m Mass density of the VACNT film: [ ] kg m^{-3} ± [ ] kg m^{-3} Thermal diffusivity of the VACNT film: [ ] m^2 s^{-1} ± [ ] m^2 s^{-1} Measurement atmosphere: [ ]

Annex A
(informative)

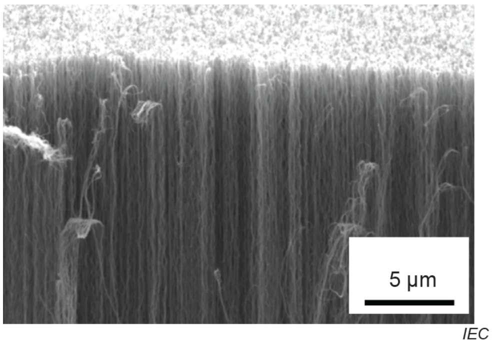
Case study of thermal diffusivity measurements for vertically-aligned carbon nanotubes grown on Si substrates

A.1 General

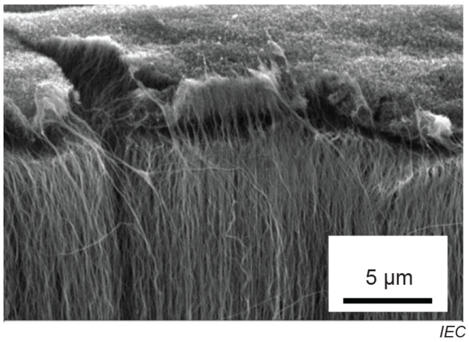
In this Annex A, case studies of measuring thermal diffusivity of VACNTs grown on Si substrate based on the flash method are described. The density and thickness measurement with X-ray absorption method were also performed as complementary assessments for VACNTs.

A.2 Sample preparation for VACNTs

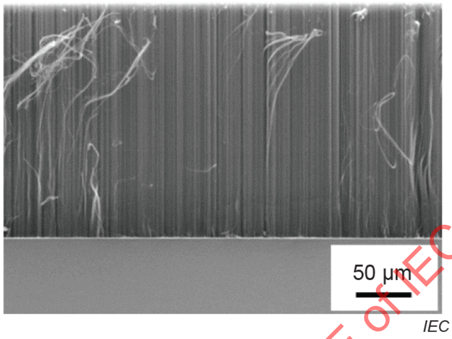
VACNT films were prepared by using chemical vapour deposition (CVD) method. VACNT films were grown from Fe nanoparticles with an ethanol precursor [2]. SiO_2 (300 nm) on Si wafers with a size of 10 mm × 10 mm were employed as substrates without any surface treatment. The thickness of the wafers was 0,5 mm. 20-nm-thick aluminium layers were deposited onto the substrates by radio frequency (rf) sputtering. After air exposure of the substrates, 2-nm-thick Fe films as metal catalysts were deposited by rf sputtering. Then, the Fe-loaded substrates were put into an encapsulated reaction chamber of a mini CVD system. The partial pressures of the nitrogen and ethanol vapours in the reaction chamber were controlled to be 10 kPa and 40 kPa, respectively. Next, the catalyst precursor heating unit using heated filament was switched on in order to generate activated radicals and accelerate CNT growth. Then, the substrate was heated up and maintained at the temperature of the VACNT growth for several minutes or several tens of minutes. The growth temperature was controlled in the range from 650 °C to 750 °C. Three VACNT samples with different thicknesses were prepared and named as Sample A, Sample B and Sample C. Cross-sectional scanning electron microscope images of these samples are presented in Figure A.1. These SEM images were obtained after conducting the measurements of both flash method and X-ray absorption method.



a) **Sample A**



b) **Sample B**



c) **Sample C**

Figure A.1 – Cross-sectional scanning electron microscope images of vertically-aligned carbon nanotube films on Si substrates

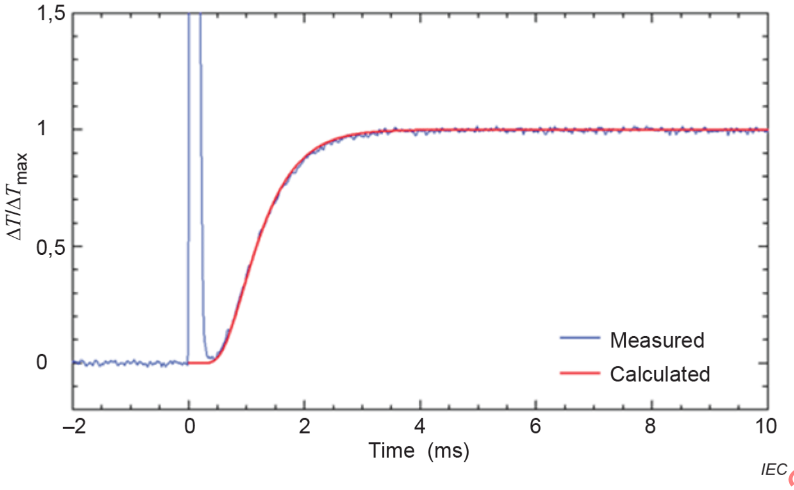
A.3 Result of flash measurements for VACNT samples

Clause A.3 reports flash measurements for VACNT films specimens (Samples A, B, and C). Before performing the flash measurements, a thin gold layer with a thickness of several tens of nanometres was sputter-deposited on the front surface (substrate side) of all the test specimens. After that, spray coating of carbon onto the front surface was performed. In fact, flash measurements without carbon spray coating were not successful because the resulting temperature rise in the VACNT side was too small to be detected.

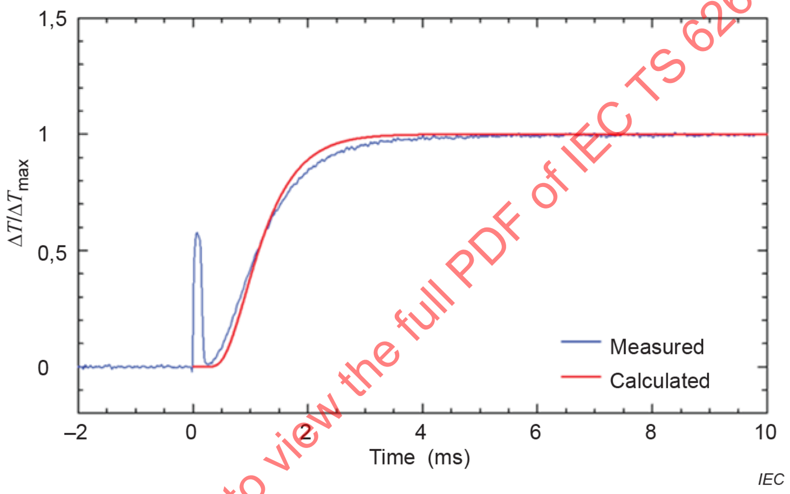
Figure A.2 shows normalized transient temperature curves for three different samples that were measured in an argon atmosphere. The initial temperature in the normalized transient curves was room temperature for all the measurements. In addition, the VACNT film thickness and film mass density were also evaluated by X-ray absorption method as specified in IEC TS 62607-2-5:2022. All experimentally-determined parameters are listed in Table A.1. Here, literature values of specific heat capacity for silicon (718 J kg^{-1} K^{-1}) and graphite (710 J kg^{-1} K^{-1}) were used for Si substrate and VACNT film, respectively [3].

The temperature change measured for Sample A is well fitted to the theoretical curve based on the bilayer of VACNTs and the substrate. Therefore, the reliability of the measurement can be confirmed. On the other hand, the temperature change measured for Sample B and Sample C can not be completely fitted by the theoretical curve based on the bilayer of VACNTs and the substrate, probably due to the low mass density of carbon nanotubes (13 kg m^{-3} for Sample B and 14 kg m^{-3} for Sample C).

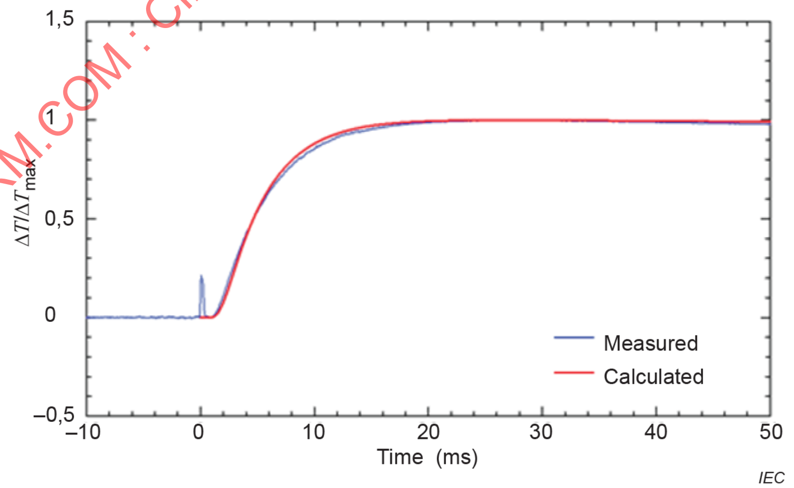
The values of thermal diffusivity of VACNT films in this case study are in the same order (10^{-5} m^2 s^{-1}) of magnitude as the values reported for supergrowth single wall carbon nanotube forests ($4,7 \times 10^{-5}$ m^2 s^{-1} to $7,7 \times 10^{-5}$ m^2 s^{-1} for approximately 1-mm-thick samples [4]). From these results, it was confirmed that the flash method can be used for VACNT film grown on Si substrates with a thickness from 100 µm to 500 µm.



a) Measurement result for Sample A



b) Measurement result for Sample B



c) Measurement result for Sample C

Figure A.2 – Normalized transient temperature curves measured for vertically-aligned carbon nanotube films on Si substrates

Table A.1 – Physical parameters obtained from VACNT samples

Sample	Thickness, h_{CNT} (µm)	Density, ρ_{CNT} (kg m^{-3})	Thermal diffusivity, α_{CNT} m^2 s^{-1}
Sample A	101	36	$1{,}1 \times 10^{-5}$
Sample B	150	13	$2{,}6 \times 10^{-5}$
Sample C	410	14	$1{,}6 \times 10^{-5}$

A.4 Analytical solution of transient temperature rise for bilayer sample measured with flash method

Heat conduction in a bilayer material (as shown in Figure 2) with light pulse heating, which is similar to the measurement system of flash method, is theoretically formulated and analysed by solving the heat diffusion equation [5], [6], [7], [8], [9].

Assuming some conditions; one-dimensional heat flow, no heat loss from the sample surface, no thermal contact resistance between layers, and uniform heat input over the front surface, the normalized transient temperature curve on the VACNT sample surface ($\Delta T/\Delta T_{max}$) is given as follows [5], [6], [7], [8]:

$$\frac{\Delta T}{\Delta T_{max}} = 1 + \sum_{n=1}^{\infty} \frac{4\left(b_{SUB}\sqrt{\tau_{SUB}} + b_{CNT}\sqrt{\tau_{CNT}}\right)\exp\left(-\frac{z_n^2}{\tau_{CNT}}t\right)}{\left(b_{SUB}+b_{CNT}\right)\left(\sqrt{\tau_{SUB}}+\sqrt{\tau_{CNT}}\right)\cos\left(\left(\sqrt{\frac{\tau_{SUB}}{\tau_{CNT}}}+1\right)z_n\right) + \left(b_{SUB}-b_{CNT}\right)\left(\sqrt{\tau_{SUB}}-\sqrt{\tau_{CNT}}\right)\cos\left(\left(\sqrt{\frac{\tau_{SUB}}{\tau_{CNT}}}-1\right)z_n\right)} \tag{A.1}$$

where ΔT_{max} is the maximum temperature rise after the pulse heating, and z_n is the n-th solution of the following characteristic equation:

$$\left(\frac{b_{SUB}}{b_{CNT}}+1\right)\sin\left(\left(\sqrt{\frac{\tau_{SUB}}{\tau_{CNT}}}+1\right)z\right) + \left(\frac{b_{SUB}}{b_{CNT}}-1\right)\sin\left(\left(\sqrt{\frac{\tau_{SUB}}{\tau_{CNT}}}-1\right)z\right) = 0 \tag{A.2}$$

In these equations, b_{SUB}, τ_{SUB} are thermal effusivity and thermal diffusion time for the substrate, respectively, and b_{CNT}, τ_{CNT} are thermal effusivity and thermal diffusion time for the VACNT film, respectively. The parameters of thermal effusivity and thermal diffusion time can be given as follows:

$$b_{SUB} = \rho_{SUB}C_{SUB}\sqrt{\alpha_{SUB}},\quad b_{CNT} = \rho_{CNT}C_{CNT}\sqrt{\alpha_{CNT}} \tag{A.3}$$

$$\tau_{SUB} = \frac{h_{SUB}^2}{\alpha_{SUB}},\quad \tau_{CNT} = \frac{h_{CNT}^2}{\alpha_{CNT}} \tag{A.4}$$