

Heat Capacity, Thermal Conductivity, and Interface Resistance Extraction for Single-Walled Carbon Nanotube Films Using Frequency-Domain Thermoreflectance

Yuan Gao, Amy M. Marconnet, Rong Xiang, Shigeo Maruyama, and Kenneth E. Goodson

Abstract—Thermal interface materials (TIMs) based on vertically aligned carbon nanotube (VACNT) films promise the unusual combination of high thermal conductance and low elastic modulus, properties that are critical for TIM performance and reliability. This paper reports through-plane thermal conductivity, heat capacity, and boundary resistances measured using frequency domain thermoreflectance for seven VACNT films. The films are composed of single-walled carbon nanotubes grown using chemical vapor deposition on Si substrates and have thicknesses from 5.3 to 81 μm . The top surfaces of the films are coated with 100-nm Ti and 100-nm Ag as a transducer layer for the thermoreflectance measurement. The roughness of the top surface of each sample is measured using atomic force microscopy. The average thermal conductivity of the films ranges from 2.1 to 3.5 $\text{W m}^{-1} \text{K}^{-1}$ and the average heat capacity ranges from 5.5 to 9.3 $\text{J cm}^{-3} \text{K}^{-1}$. The heat capacity of the films is used to calculate the volume fraction of tubes that contributes to thermal conduction. The CNT-metal boundary resistances are below 1 $\text{m}^2 \text{K MW}^{-1}$ and the CNT-substrate resistance ranges from 3 to 80 $\text{m}^2 \text{K MW}^{-1}$. Variations of the nanotube alignment and morphology throughout the film are also examined using scanning electron micrographs to provide a comprehensive analysis of these films.

Index Terms—Carbon nanotubes (CNTs), nanotechnology, thermal management, thermoreflectance, thin films.

I. INTRODUCTION

THERMAL interface materials (TIMs) are critical for a variety of applications including microprocessors and

thermoelectric power generators [1]–[3]. The temperature drop across these interface materials is a key factor influencing the reliability of computers and the efficiency of thermoelectric generators. Though compliant materials such as thermal greases mitigate reliability concerns resulting from mismatches in the coefficient of thermal expansion (CTE) at interfaces, they do not conduct heat as well as a solder joint. Recently, there has been interest in TIMs based on vertically aligned carbon nanotube (VACNT) films. A VACNT film makes use of the high axial thermal conductivity and flexibility of individual carbon nanotubes (CNTs) and avoids drawbacks of polymer-based greases such as pump-out, drying, and degradation at high temperatures [1], [2]. VACNT films can currently be grown to millimeters in height and on a variety of substrate materials, including metals [4], [5]. They are promising for applications with large CTE mismatch or substantial gaps between the contacting surfaces.

Given the wide range of past data on VACNT thermal properties, there is a need to better understand and improve the performance of these films. Past experiments have only determined one or two properties at a time, including thermal conductivity, heat capacity, and boundary resistance, in part due to limitations in measurement techniques. VACNT films have a complex morphology that can affect the thermal and mechanical behaviors of the film. The thermal properties are rarely linked to analysis of the film morphology.

Past measurement and simulation of the thermal conductivity of an individual nanotube, k_{ind} , range from around 34 to 10000 $\text{W m}^{-1} \text{K}^{-1}$ [6]–[8], with most values around 3000 $\text{W m}^{-1} \text{K}^{-1}$ [6], [9]–[11]. Most modeling of nanotubes is performed on single-wall CNTs (SWCNTs) due to the complexity of multiwalled CNTs (MWCNTs). Though individual MWCNT have been measured to have k_{ind} as high as 3000 $\text{W m}^{-1} \text{K}^{-1}$ [10], [11], Pettes *et al.* [12] measured a significantly lower k_{ind} in their MWCNT compared to their SWCNT, possibly due to increased defects in the MWCNT.

Previous measurements of the effective thermal conductivity of VACNT films, k_{film} , have nearly all been performed on MWCNT films. This is in part due to the difficulty of growing vertically aligned SWCNT films, though SWCNT films generally have higher CNT quality [13], [14]. Past work measuring MWCNT films report k_{film} ranging from 0.5 to 267 $\text{W m}^{-1} \text{K}^{-1}$ [15]–[24]. Most of these values of k_{film} are much lower than predictions based on scaling the ideal

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$k_{\text{ind}} = 3000 \text{ W m}^{-1} \text{ K}^{-1}$ by f , the nominal volume fraction of CNT's in the film. A review of past measurements of k_{film} scaled by f is provided in Gao *et al.* [3]. The specific reasons for the relatively low and scattered values of k_{film} remain poorly understood—the mechanisms may be due to the lower quality of CNTs in a film compared to the high-quality tubes used for individual tube measurements, effects from tube–tube interaction, or poor engagement between the film and surrounding materials. The quality of the contact between the film and surrounding surfaces also affects boundary resistances, which can be a significant contribution to the total thermal resistance of the VACNT layer [18], [24], [25].

There have been few measurements of both k_{film} and film heat capacity for a given film. Past measurement techniques include laser flash [17], [23], which gives film diffusivity (α), nanosecond thermoreflectance (NSTR) [25], which gives film effusivity (e), and steady-state methods [16], [19], which give film thermal conductivity. The volumetric heat capacity of the film, $C_{v,\text{film}}$, is sometimes estimated using f and then used to extract k_{film} from thermal diffusivity or effusivity measurements [17], [22], [23]. $C_{v,\text{film}}$ is related to f through the density and heat capacity of an individual nanotube. The value of f is typically estimated from an image analysis or mass measurements. Previous observations of the nanotube structure show that the CNT density and alignment in a VACNT film can vary throughout the film thickness [26]–[28], which may result in errors in density calculated from image estimates. Furthermore, if there are nanotubes present that do not participate in thermal conduction, the heat capacity will be overestimated [29]. Thus k_{film} and $C_{v,\text{film}}$ should be measured separately for each film.

This paper represents the first study on the thermal properties of a set of seven high-quality single-wall VACNT film samples grown under identical conditions over a range of thicknesses. A frequency domain thermoreflectance (FDTR) technique provides individual sensitivity to k_{film} , $C_{v,\text{film}}$, and boundary resistances. The surface roughness of the metalized films is characterized using an atomic force microscope (AFM). The film morphology is quantified using image analysis and provides insight into its thermal response. This also serves as a method to quantify differences between samples, which may help explain the large variation in the past measured thermal properties.

II. SAMPLE PREPARATION

Vertically aligned SWCNT films are grown by alcohol catalytic chemical vapor deposition at 800 °C with ethanol as the carbon source. Details of the synthesis and growth procedure have been previously reported in [14], [28], [30], [31]. The Si substrates are fabricated by means of growth of a 50-nm SiO_2 layer, followed by evaporation of catalyst films of 10-nm Al and 0.2-nm Co. This growth procedure produces SWCNT with an average diameter of 2 nm. The number density of nanotubes is around 10^{15} m^{-2} [32]. The nanotubes often form bundles, typically composed of 5–6 tubes [32].

Growth times are varied to yield seven SWCNT film thicknesses of 5.3, 6.3, 16, 40, 42, 45, and 81 μm . The mass

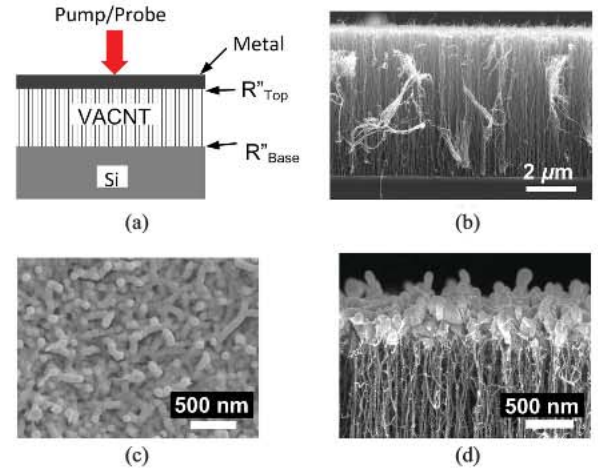


Fig. 1. (a) Schematic of the sample structure for thermoreflectance measurements. The top and bottom boundary resistances are indicated by R''_{top} and R''_{base} respectively. (b) Cross section of a VACNT film on Si. (c) Top-down SEM image of the metalized surface of a SWCNT film. (d) SEM image of a cross section of a SWCNT sample, showing the metal layer on top of the vertically aligned nanotubes.

densities of the SWCNT films are determined using their measured mass, thicknesses, and areas. The masses of the sample before and after the SWCNT growth are measured using a microbalance (SE2 Ultra Micro Balance, Sartorius). This value is confirmed by performing a second measurement that involves removing the SWCNT film and reweighing the substrate. The measured mass density of 94–102 kg m^{-3} translates to $\sim 7\%$ volume density, assuming an individual SWCNT density of 1.5 g cm^{-3} based on the density of graphite (2.23 g cm^{-3}), graphite interlayer spacing (0.34 nm), and radius of the SWCNT (1 nm). This value is not used in any calculations of thermal properties since the effective heat capacity of the film is measured in this paper. It should be noted that this value is not necessarily constant between samples or between regions in a sample, but nominally describes the overall density of these films.

For the thermal measurements, a 100-nm-thick Ti layer is evaporated on top of the SWCNT array as an adhesion layer, followed by a 100-nm-thick Ag film for high optical reflectance. The sample structure is shown in Fig. 1(a), with a cross sectional scanning electron microscope (SEM) view of the film shown in Fig. 1(b). The SEM images of the sample in Fig. 1(c) and (d) shows that the metal forms a discrete layer over the nanotubes, with pore diameters $< 100 \text{ nm}$. The rough surface of the metal-coated VACNT film often poses a challenge for thermoreflectance measurements. Past techniques for overcoming this include bonding or pressing other substrates onto VACNT films, but these methods require more specialized sample preparation and can affect the measurement [18], [23], [24]. Because of the rough nature of the film, there is some uncertainty regarding the heat capacity and thickness of the film—these are discussed later.

For image analysis, the VACNT samples are first split by cleaving the Si substrate to gain visual access to the CNT's away from the sample edges. The cleaved edge is sputter coated with $\sim 4\text{-nm}$ Au to enhance the SEM image contrast.

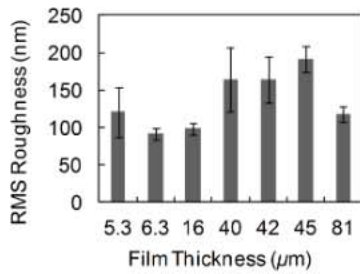


Fig. 2. RMS surface roughness of metalized VACNT films. The error bars are based on the standard deviation between images.

Images of the film are taken using a FEI XL30 Sirion SEM from the top to the base of the film. The image analysis and procedure to extract alignment and volume fraction are described in Gao *et al.* [27].

The surface roughness of the metalized VACNT films is examined using a Topometrix Explorer scanning probe microscope in contact mode. The VACNT films without a metal layer did not provide clear AFM images due to the soft and porous film surface. The AFM cantilevers used have silicon nitride tips with 20-nm radius. At least three images with scan size 10-μm square or 20-μm square are taken of each sample. Images are leveled and horizontal strokes are corrected using data analysis software (Gwyddion 2.22) before the root mean square (RMS) roughness is calculated. Fig. 2 shows the results of AFM surface topography measurements. There does not appear to be a strong trend with film thickness.

III. THERMAL MEASUREMENTS

A. Experimental Apparatus

The films are measured using two optical noncontact methods: an NSTR technique and an FDTR technique. The NSTR experimental setup and theory has been described in detail in the previous work [25], [33]. The FDTR method has also been well documented in the past literature [34]–[36]. This paper describes this particular setup for the first time. This FDTR setup is integrated with the NSTR rig, where the sample is located at the same position and the probe laser and photodetector are shared. The FDTR setup provides sensitivity to individual material properties, as explained in the following section, and the NSTR technique is only used to confirm the FDTR results for these measurements.

Fig. 3 shows the FDTR setup, in which the intensity of a heating laser, or “pump” (Nd:YAG, 1064 nm), is modulated by an electro-optic modulator (EOM) (Conoptics, Inc. Model 360) that is controlled by a high-voltage analog driver (Conoptics, Inc. Model 25A, dc to 30 MHz). A function generator controls the modulation frequency of the EOM. An optical isolator is placed before the EOM to prevent back reflections into the laser cavity that cause instabilities in the heating beam.

The pump heats the metalized surface of the VACNT sample with a sinusoidal heat flux. A probe beam (658-nm wavelength) is reflected off the surface and the amplitude of the reflected beam is detected by an 80-MHz balanced photodiode (New Focus, Model 1807). The reflectivity of the

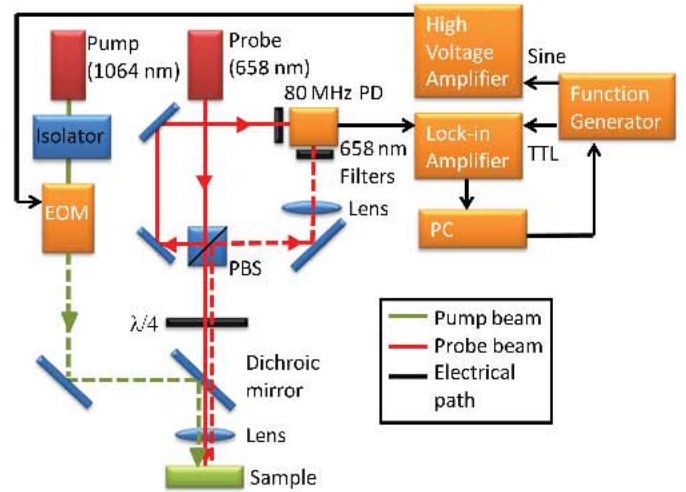


Fig. 3. Schematic of the FDTR setup. A PC controls the function generator and captures data from the lock-in amplifier, allowing sweeps over various frequency ranges at customizable data rates and sensitivities. The photodetector is a balanced detector; the photodiode depicted on the lower edge detects the modulated probe beam and the detector on the left edge captures the unmodulated probe beam. The intensity of the two beams is adjusted using filters and wave plates.

metal film is proportional to its temperature, so the reflected intensity of the probe provides the temporal dependence of the sample’s surface temperature. The photodiode is operated in balanced mode to increase the signal-to-noise ratio and includes 658-nm band-pass filters on both the detectors. The photodiode voltage is sent to a lock-in amplifier that can operate from dc to 100 kHz (Stanford Research Systems, Inc. Model SR830). Lock-in amplifiers can detect the amplitude and phase shift of signals with poor signal-to-noise ratios by locking in to the fundamental frequency of the signal and rejecting all other frequencies. The function generator used to control the EOM also sets the reference frequency and phase of the lock-in amplifier.

The amplitude and phase shift of the pump beam is determined in the same manner by detecting the pump beam reflected off the sample using the same optical path and components (e.g., photodetector, cables). This is used to find the amplitude and phase shift due to the sample of interest by accounting for phase and amplitude shift of the setup itself. To do this, the probe amplitude is divided by the pump amplitude, after both are normalized, and the pump phase shift is subtracted from the measured probe phase shift.

The amplitude shift and phase delay of the reflected probe beam relative to the heating beam depends on the thermal properties of the sample and the frequency of heating. The solution to the 3-D, radially symmetric heat diffusion equation for a multilayer stack [34] is fit to the phase shift and normalized magnitude data using a multiparameter, least-squares-fitting algorithm. The sample structure consists of a semi-infinite air layer, metal layers, VACNT film layer, and semi-infinite Si substrate layer with boundary resistances between each layer. This allows extraction of the effective thermal properties of the VACNT film and its interfaces.

Both the pump and probe beams are focused onto the sample using a 10X objective lens. The radii of the heating

and probe spot affect the thermal response and can be tuned by adjusting the divergence of the beams. For these measurements, a $1/e^2$ heating spot radius (r_{pump}) of $183 \mu\text{m}$ is chosen. The large heating diameter allows the spot size to be directly measured using a charge-coupled device beam profiler (Thorlabs, Inc. BC106-VIS) and reduces the temperature rise of the region. It also reduces sensitivity to the film anisotropy, which is assigned a value based on past literature. The probe radius (r_{probe}) is calculated to be $8 \mu\text{m}$ at the focus by fitting a set of beam widths, measured by the beam profiler, to a Gaussian beam profile. The thermal response has low sensitivity to r_{probe} . The beam profiler is also used to ensure that the beams are coaxial. The maximum temperature rise of the sample is given by $\Delta T_{\text{avg}} = Q_{\text{avg}} / (2\sqrt{\pi} r_{\text{pump}} k)$, where Q_{avg} is the average laser power and k is the thermal conductivity of the substrate [34], [36]. The average absorbed heating power is estimated to be 20 mW using a power meter and the reflectivity of Ag. For the worst case scenario of a semi-infinite, low conductivity CNT film with $k = 2 \text{ W m}^{-1} \text{ K}^{-1}$, the temperature rise is around 15 K.

B. Data Analysis and Uncertainty

Thermoreflectance measurements are particularly challenging for CNT films because the thermal response of these samples is independently sensitive to as many as four thermal properties, depending on their relative magnitudes and the thermal diffusion depth. These include $C_{v,\text{film}}$, k_{film} , the top-side boundary resistance R''_{top} , and the base-side boundary resistance R''_{base} . The thickness of the CNT film and the properties of the metal layer and substrate are set as known values. Fig. 4 shows examples of imperfections in a VACNT film that affect the unknown parameters. Fig. 4(a) shows how incomplete CNT-metal contact can create thermally isolated CNTs due to high CNT-CNT thermal resistance. These isolated nanotubes (in blue) would not contribute to thermal conduction and heat capacity, but they would be included in mass measurements and image estimates of f . Fig. 4(b) indicates that CNT-CNT contact may reduce k_{ind} by affecting phonon modes in a CNT. Fig. 4(c) shows how defects may also reduce k_{ind} by acting as scattering sites for phonons and can create kinks in the nanotube structure. Fig. 4(d) shows that reduced density at the base of the film may affect the calculated f of the film, although if the nanotubes contact the top metal layer they will still contribute to heat capacity in a thermoreflectance measurement. This figure also illustrates decreasing nanotube alignment toward the base, which may indicate defects and increased tube-tube contact. The detachment of tubes may also manifest as a high boundary resistance.

The values of f and k_{ind} for CNT films are unknown but can be related to the previous four unknown parameters as follows. For the analysis, f is defined as the volume of tubes that contribute to thermal conduction within the film, $k_{\text{film}} = f \times k_{\text{ind}}$. An equal number of nanotubes should contribute to heat capacity and thermal conductivity, giving the additional condition $C_{v,\text{film}} = f \times C_{v,\text{ind}}$, where $C_{v,\text{ind}}$ is the volumetric heat capacity of an individual SWCNT. $C_{v,\text{ind}}$ is assumed to be $1 \text{ MJ m}^{-3} \text{ K}^{-1}$, assuming the specific heat of a SWCNT is

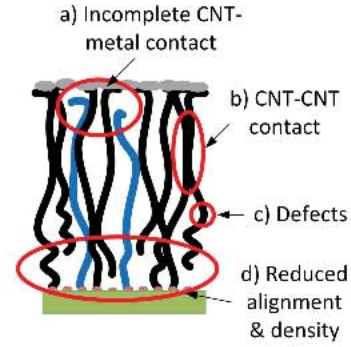


Fig. 4. Diagram of a VACNT film coated with a metal layer (gray) grown from catalysts (orange) on a substrate (green). Various imperfections in a VACNT film, which govern the properties extracted in this paper, are illustrated.

that of graphite, $710 \text{ J kg}^{-1} \text{ K}^{-1}$, and the density of a SWCNT is 1.5 g cm^{-3} .

Though it is possible to simultaneously fit four unknown parameters, during data fitting there are often multiple solutions that fit a single thermal trace. In many cases, a thermoreflectance measurement may only be sensitive to α or e . This is the case for the NSTR technique, which gives sensitivity to e . The advantage of the FDTR technique is that it provides sensitivity to k and C_v separately due to radial heat spreading [39], [40]. Sensitivity is defined as

$$S = \frac{\% \text{ change in } Y}{\% \text{ change in } X} = \lim_{\Delta Y, \Delta X \rightarrow 0} \frac{\Delta Y / Y_0}{\Delta X / X_0} \quad (1)$$

Sensitivity is generally dictated by the thermal diffusion depth (L_p) into the sample, the degree of radial heat spreading, and the relative contribution of a parameter to the overall thermal response. Thermal diffusion depth is given by $L_p = \sqrt{2\alpha/\omega}$ where ω is the angular frequency of the heating. These effects are illustrated by the following examples. At a frequency of 1 kHz and α of $55 \text{ mm}^2 \text{ s}^{-1}$, $L_p = 132 \mu\text{m}$, whereas at a frequency of 100 kHz, $L_p = 13 \mu\text{m}$. If L_p is much lower than the thickness of the film, the results will not be sensitive to R''_{base} . If the ratio of r_{pump} to L_p is high, the thermal diffusion through the sample will become 1-D and the thermal response will only give sensitivity to e . For the VACNT films less than $40\text{-}\mu\text{m}$ thick, the thermal response is not sensitive to k_{film} because the contribution of the film resistance to the overall thermal response is small. Similarly, the sensitivity to R''_{top} decreases when R''_{top} is small.

Fig. 5 shows the sensitivity of amplitude and phase to various parameters, illustrating how the sensitivity to each parameter is unique over the frequency range. The response exhibits unique sensitivity to k_{film} and $C_{v,\text{film}}$ and low sensitivity to R''_{top} . For example, at around 16 kHz, the phase response has zero sensitivity to k_{film} but has some sensitivity to $C_{v,\text{film}}$. The magnitude generally exhibits low sensitivity to k_{film} but not $C_{v,\text{film}}$. Near 100 kHz, the phase response exhibits sensitivity to e with no distinction between k_{film} and $C_{v,\text{film}}$. This is because as frequency increases, the thermal diffusion depth decreases, resulting in 1-D heat flow. The sensitivity to R''_{base} is also unique but is not shown for clarity.

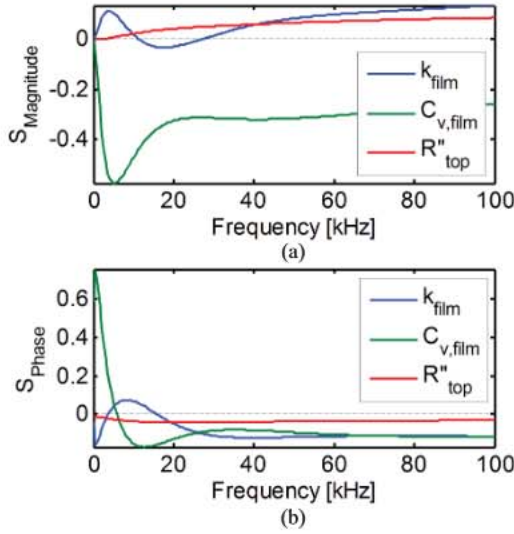


Fig. 5. Theoretical sensitivity to changes in k_{film} , $C_{v,\text{film}}$, and R''_{top} for (a) magnitude response and (b) phase response based on measured values for the 45- μm -thick sample. The dashed line indicates zero sensitivity.

Uncertainties due to local variations in the metal thickness, CNT thickness, and signal noise contribute to the scatter in the data. These errors are captured by taking multiple measurements per sample; each sample is measured at three different locations on the sample with at least two data sets per spot. This gives the standard deviation of the data at each frequency.

There is a range of possible best-fit solutions for each data set that fall within the scatter of the data. A single four-parameter fit would only represent one possible solution in the solution space. To interrogate the full range of values that fit the data, the following procedure is done for each sample over each of the four variables. The value of one variable is fixed (e.g., k_{film}) while data fitting is performed on the other three variables (e.g., R''_{top} , $C_{v,\text{film}}$, R''_{base}). This is repeated to find the minimum and maximum values of the fixed variable that give good fits to data (e.g., $k_{\text{film}} = 2\text{--}4 \text{ W m}^{-1} \text{ K}^{-1}$). For example, if k_{film} for the thickest sample is assumed to be $50 \text{ W m}^{-1} \text{ K}^{-1}$, there is no combination of variables that give a good fit to data, thus k_{film} cannot be that value.

Bounds on each parameter, shown in Table I, are used during data fitting to limit the solution space. The lower bound of the boundary resistances represents the typical contact resistance between two smooth well-contacted surfaces and is based on values from past modeling of CNT-substrate and graphene-metal contact resistance [29]. The upper bound is an order of magnitude higher than the highest measured boundary resistance for VACNT films. The range of k_{film} is based on the range of past measured data on VACNT films. The upper limit of f is based on the highest expected density of these VACNT films and is higher than the measured mass density of 7%. Anisotropy ranges between perfectly anisotropic (zero) to perfectly isotropic (one). The expected value of 0.01 is based on previous measurements of anisotropy that range from 0.014 to 0.042 [17], [21], [37] and the observation that these nanotubes are well aligned. The other expected values

TABLE I
BOUNDS FOR EACH UNKNOWN PARAMETER AND EXPECTED VALUES

Parameter	Lower Bound	Upper Bound	Expected Value
R''_{top} ($\text{m}^2 \text{ K MW}^{-1}$)	0.001	1000	1
k_{film} ($\text{W m}^{-1} \text{ K}^{-1}$)	0.5	300	5
f (%)	1	10	7
R''_{Base} ($\text{m}^2 \text{ K MW}^{-1}$)	0.001	1000	1
Anisotropy	0	1	0.01

in Table I represent the initial guesses used during data fitting. The results are independent of these values.

There are also errors due to the uncertainty in known parameters such as the thicknesses of each layer, known thermal properties, and spot sizes. This is calculated using standard error propagation techniques given by

$$\Delta Z_i = \sqrt{\sum_j \left(\frac{\partial Z_i}{\partial j} \Delta j \right)^2} \quad (2)$$

Here Z_i are the extracted parameters k_{film} , $C_{v,\text{film}}$, R''_{top} , and R''_{base} and j are the modeling parameters. ΔZ_i represents the error of each parameter. The thermal conductivity of the metal layers and the properties of the Si substrate are assumed to have low uncertainty and contribute negligibly to the total error. Table II shows that the errors due to these parameters are much lower than the error due to the scatter in the data, except for the error in k_{film} due to uncertainty in the thickness and heat capacity of the metal. L_{metal} represents the thickness of the metal layers and L_{CNT} represents the thickness of the SWCNT film. The uncertainties in the metal layer dominate the total uncertainty for each variable.

IV. RESULTS AND DISCUSSION

Fig. 6 shows an example of the best fit magnitude and phase response to data for the 45- μm sample. The best fit values that fall within the error range are reported in the results.

The results from the thermoreflectance measurements of the SWCNT films are shown in Fig. 7. The error bars encompass the full range of possible solutions. The extracted film properties all fall within those reported in past literature. As mentioned earlier, there is low sensitivity to the properties of the VACNT film itself as the film thickness decreases below 40 μm . k_{film} is not reported for those films and the error bars for f are larger.

Fig. 7(a) shows that the average values of k_{film} range between 2.1 and 3.5 $\text{W m}^{-1} \text{ K}^{-1}$. In Fig. 7(a), the results from the FDTR measurement are compared against NSTR measurements to check for consistency. The NSTR data are fit using a set value of f from the FDTR results and match well with the FDTR results. Fig. 7(b) shows the best fit values for f . $C_{v,\text{film}}$ ranges from 5.5 to 9.3 $\text{J cm}^{-3} \text{ K}^{-1}$ and the values of α range from 37 to 56 $\text{mm}^2 \text{ s}^{-1}$. This is comparable to past data of α that range from 42 to 90 $\text{mm}^2 \text{ s}^{-1}$ [17], [21], [37]. The large uncertainty in f for some samples makes it difficult to discern any trends with thickness.

TABLE II
EXAMPLE UNCERTAINTY ANALYSIS FOR THE 45- μm -THICK SWCNT FILM. ΔZ_i ARE GIVEN IN THE LAST FOUR COLUMNS

Parameter	Nominal Value	Uncertainty	Δk_{film} ($\text{W m}^{-1} \text{K}^{-1}$)	Δf	$\Delta R''_{\text{top}}$ ($\text{m}^2 \text{K GW}^{-1}$)	$\Delta R''_{\text{base}}$ ($\text{m}^2 \text{K GW}^{-1}$)
L_{metal}	200 nm	10 nm	0.12	0.01	11	338
$C_{v,\text{metal}}$	$2.47 \text{ MJ m}^{-3} \text{K}^{-1}$	10%	0.12	0.01	13	346
Anisotropy	0.01	0.01	0.02	0.00	6	47
L_{CNT}	45 μm	1 μm	0.08	0.00	0	2
r_{pump}	183 μm	5 μm	0.07	0.00	34	31
r_{probe}	8.5 μm	2 μm	0.00	0.00	1	13
Total Uncertainty	—	—	0.20	0.01	38	488

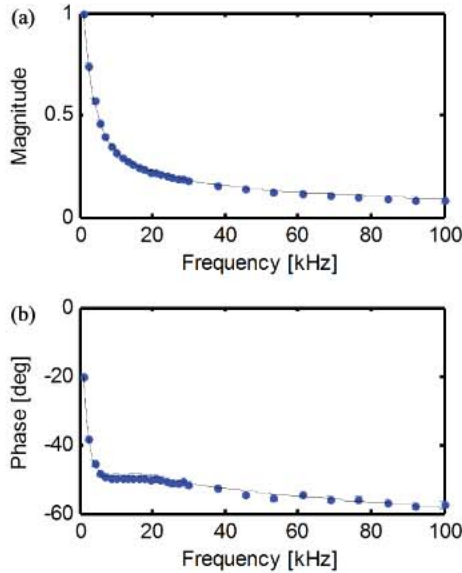


Fig. 6. FDTR data and best fit solution for the (a) magnitude response and (b) phase response for the 45- μm sample. The data points include error bars representing the standard deviation of the data. A higher number of data points are taken at frequencies below 30 kHz because the sensitivities are stronger and there is a higher rate of change in that region.

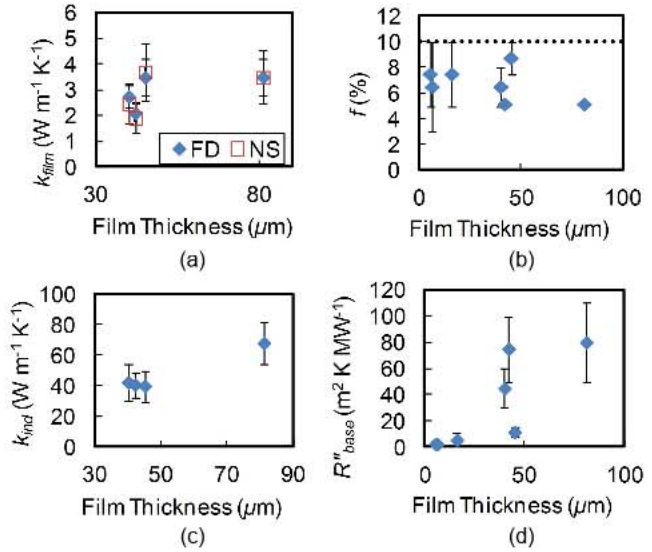


Fig. 7. (a) k_{film} of each sample represented by the film thickness. “FD” represents data taken using FDTR whereas “NS” represents data taken using NSTR. (b) f of each sample with $f = 10\%$ as the upper bound (dashed line). (c) k_{ind} calculated from data in (a) and (b). (d) R''_{base} of the SWCNT films.

Fig. 7(c) shows k_{ind} calculated from the data in Fig. 7(a) and (b) using the relationship $k_{\text{film}} = f \times k_{\text{ind}}$ given earlier. The results are much lower than the ideal values of $k_{\text{ind}} = 3000 \text{ W m}^{-1} \text{K}^{-1}$ though they match well with past values of k_{ind} extracted from measurements of VACNT films [16], [17], [19], [21], [24], [37]. There may be low k_{ind} from an increased probability of defects, vacancies, and impurities with tube length [17], [38]–[40]. Defects can develop during CNT growth from a variety of factors, including gas pressure and growth time [41]. Sidewall defects can cause kinking of the nanotubes, which may contribute to the tortuous morphology of the tubes [38], [42]. The CVD-film growth process typically produces CNTs with more defects and impurities than the pristine, short CNTs that give high k_{ind} values in individual CNT measurements [43]. Based on SEM and TEM analyses of these films, a majority of the tubes in the film are bundled [32]. Tube–tube coupling in the form of tube crossing or bundling may increase the number of phonon scattering sites along the CNT, causing lower k_{ind} [44]. Simulations by Berber *et al.* [45] have shown that the interlayer interactions of stacked graphene sheets (graphite) lowered the thermal conductivity of a single sheet by nearly an order of magnitude

and that the same should be expected of CNT bundles. Measurements on bundled tubes have shown lower k than single tubes, from ~ 2 to $683 \text{ W m}^{-1} \text{K}^{-1}$ [46]–[48].

Fig. 7(d) shows that R''_{base} ranges from 3 to $80 \text{ m}^2 \text{K}^{-1} \text{MW}^{-1}$ for the thick samples. Only an upper bound could be determined for R''_{top} because the measurement loses sensitivity to R''_{top} as its value becomes small. R''_{top} is estimated to be $0.5 \text{ m}^2 \text{K}^{-1} \text{MW}^{-1}$ or below for all samples because there is no expected thickness dependence of R''_{top} due to the base-side growth and the constant surface roughness with thickness. There is also no lower bound for R''_{base} for the thinnest three samples. High R''_{base} is possibly due to the poor thermal engagement between the CNTs and catalyst particles at the substrate, which may be related to the growth technique [29]. The ease in which CNT films delaminate from the substrate reflects this weak adhesion. There appears to be a trend of increasing R''_{base} with increasing thickness. This may be due to detachment of nanotubes from the base or increasing defect concentration with growth time, as mentioned earlier [26]. Thicker films seem to delaminate more easily during handling of the sample. Also, sample damage may cause variations between samples.

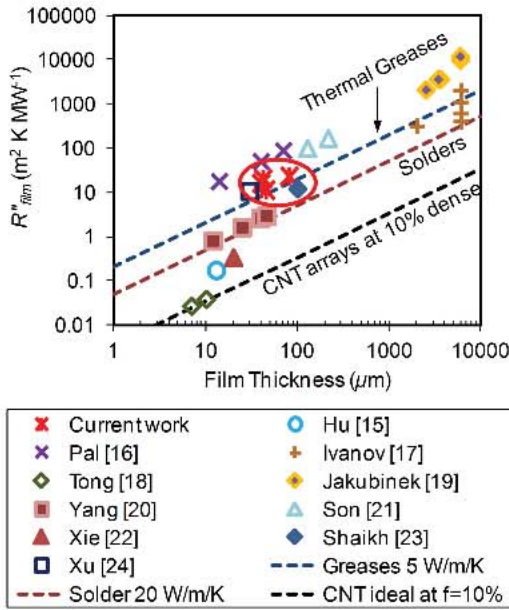


Fig. 8. Thermal resistance of VACNT films, without boundary resistances. Because k_{film} is unknown for the films $< 40 \mu\text{m}$, they are not plotted. The data are compared to grease at $5 \text{ W m}^{-1} \text{ K}^{-1}$, solder at $20 \text{ W m}^{-1} \text{ K}^{-1}$, and an “ideal” VACNT film with $k_{\text{ind}} = 3000 \text{ W m}^{-1} \text{ K}^{-1}$ and $f = 10\%$ giving $k_{\text{film}} = 300 \text{ W m}^{-1} \text{ K}^{-1}$.

Though R''_{base} is large in some samples, in practical applications, VACNT films would be solder bonded to the surrounding substrates. This has been shown to reduce a growth or un-bonded interface resistance by an order of magnitude or more [18], [49]. R''_{top} represents the CNT film bonded to metal, regardless of film thickness, so solder bonding the base side can potentially lower R''_{base} by an order of magnitude. VACNT films that are solder bonded would then have a total resistance close to the thermal resistance of the bulk film, defined as $R''_{\text{film}} = L_{\text{film}}/k_{\text{film}}$. In this case, the total resistance of the film would be comparable to thermal greases, which have thermal resistances on the order of $10 \text{ m}^2 \text{ K MW}^{-1}$ [1]. R''_{film} is plotted against film thickness in Fig. 8. R''_{film} is compared to values from past literature and to the thermal resistance of a typical layer of thermal grease, solder, and an “ideal” VACNT film. The data in this paper (circled) are within the past range of data for multiwalled VACNT films. From observation of the past data, k_{film} tends to decrease with film thickness, with films $< 50 \mu\text{m}$ having nearly ideal performance and films $> 100 \mu\text{m}$ never performing better than solder. This may be indication that thicker films have lower engagement and higher defect density with growth time. It could also be an experimental artifact, as it is difficult to measure thin films accurately and some of the high k_{film} values were calculated using assumptions about $C_{v,\text{film}}$ or k_{ind} .

Fig. 9 shows several representative image analysis results that may help elucidate trends in the thermal data. Fig. 9(a) shows the CNT alignment in the film, represented by an aspect ratio that is the ratio of the average vertical tube–tube spacing to the average horizontal tube–tube spacing. The alignment peaks around $15 \mu\text{m}$ from the top and decreases as growth time increases, similar to films observed in past work [26], [27]. Fig. 9(b) shows f quantified by estimating the volume

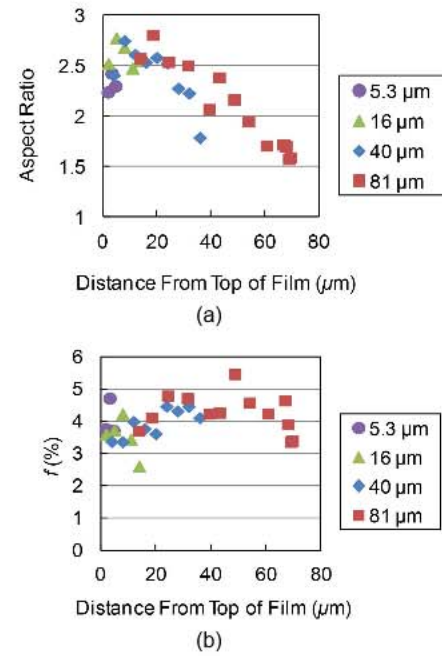


Fig. 9. (a) Aspect ratio calculated from SEM images taken from the top to bottom of SWCNT films. The nanotubes grow from the base side, so the top of the film corresponds to initial growth. (b) Calculated f based on the same images as in (a). The calculation assumes a bundle diameter of 5 nm [32].

of tubes in a given volume of the film. f is calculated using an assumed bundle diameter of 5 nm ; it does not represent an accurate comparison to the measured value of f . Using this assumption, the density appears to be fairly constant through the film thickness and between films and does not exhibit previously observed density decay [26], [27]. However, obtaining density information from SEM images is less accurate than the alignment due to the impact of the depth of field of the SEM and bundling, resulting in more scatter in f . In summary, the alignment of these SWCNT films shows that there are thickness-dependent effects that stem from the base side.

V. CONCLUSION

This paper presents the complete thermal properties of a set of vertically aligned SWCNT films ranging from 5.3- to $81\text{-}\mu\text{m}$ thick. The properties k_{film} , $C_{v,\text{film}}$, R''_{top} , and R''_{base} are measured independently, and the morphology of the films is studied using SEM and AFM. It is clear from the results that SWCNT films do not perform better than MWCNT films even though these samples had relatively higher quality and alignment. Thermal measurements and image analysis show that the thermal properties may be related to the thickness of the film, because the film seems to be increasingly disordered at the base as growth continues and R''_{base} appears to increase with film thickness. For many samples, R''_{base} is an order of magnitude higher than R''_{top} .

Quantifying the density and tortuosity of CNTs in a film can serve several purposes. Knowing the film structure can explain differences in measured properties between samples. This can also guide the growth process, if a certain structure and thermal property is desired. A model of the film's thermal properties based on structural information would enable prediction of the thermal performance of a CNT film.

Future improvements to the thermal performance of VACNT films include soldering bonding both surfaces of the film, increasing film density, and annealing of defects, which are all active areas of study in other work. At $f \sim 7\%$, there is potential to greatly increase the density of these films, either by modifying growth methods or mechanical densification. With these improvements VACNT films have the potential to consistently surpass the performance of solder TIMs.

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