CNT BUNDLES GROWTH ON MICROHOTPLATES FOR DIRECT MEASUREMENT OF THEIR THERMAL PROPERTIES

Cinzia Silvestri[*]*, Bruno Morana, Giuseppe Fiorentino, Sten Vollebregt, Gregory Pandraud, Fabio Santagata, Guo Qi Zhang and Pasqualina M. Sarro*
[*]Delft University of Technology, ECTM-DIMES, Delft, The Netherlands

ABSTRACT

Vertically aligned Carbon Nanotubes (CNT) arrays were successfully grown on top of a freestanding microhotplate, to investigate the thermal dissipation properties of CNT bundles and their applicability as heat exchanger. Two CNT configurations are employed: a group of six bundles, each with a diameter of 20 µm, and a single CNT bundle with a diameter of 200 µm. In both configurations the bundles are 70 µm high. The microhotplate consists of a platinum thin film microheater integrated on a freestanding silicon nitride membrane. The microhotplate is used as heat source and as temperature sensor. Results show that at 300 °C, 20% and 31% of power can be saved with the circular six and single bundle configurations, respectively

INTRODUCTION

Due to the increasing performance requirements, system downscaling, and features integration, thermal management has become a critical issue in electronic design. It is therefore crucial to efficiently drain off the heat generated within the integrated electronics, in order to avoid damage or malfunctioning of the components.

To increase the effectiveness of thermal management components, new nanomaterials and advanced micro-scale features are explored [1]. Due to their prominent thermal properties, vertical aligned CNTs have been suggested as promising material for thermal management [2]. Numerous studies, mostly theoretical, have been published to evaluate the thermal performance of CNTs and their applicability for heat removal in integrated system devices. However, to estimate the thermal performance of nanotubes is still a big challenge, because conventional thermal measurement techniques do not have enough spatial resolution to accurately detect temperature drop across micrometer length scales [1]. Most data published on CNTs thermal performance, are extracted employing complex techniques, like photothermal metrology [3], time-resolved infrared pyrometry tests [4] and the three-ω method [5]. However, none of these techniques is adopted for CNT structures with high aspect ratio. Moreover, they require a top metallization after the growth process, which can affect CNT quality.

Here we propose a novel approach to evaluate the thermal properties of CNT bundles based on a direct measurement technique to assess their suitability for thermal management applications such as thermal interface material (TIM) [6] or as external heat-sink [7]. This technique has been successfully employed for the thermal characterization of thin films [8]and single silicon nanowire [9].

The test structure used in this study consists of an array of vertical CNT bundles, directly synthetized on top of a freestanding microheater (Fig 1). In this way the thermal isolation necessary to detect the cooling effect of the CNT bundles is achieved. No further process steps are required after the CNTs growth, thus keeping intact their intrinsic qualities. By means of this microfabricated device, we want to understand the average CNT bundle performance instead of the single nanotube, in order to demonstrate the feasible application as heat exchanger material and, more generally, as sensing element. Two different CNT configurations are studied: a group of six symmetrically distributed bundles each with a diameter of 20 µm, and a single bundle with a diameter of 200 µm. The power dissipated through the air by CNTs is then measured to extract their thermal properties.

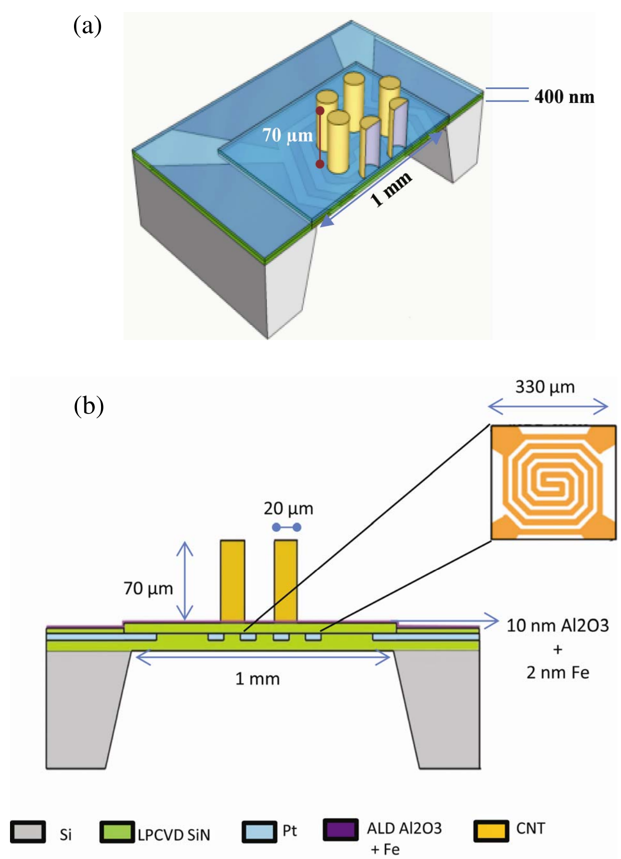

Figure 1: The proposed test structure and the main geometrical parameters: (a) 3D sketch, and (b) detailed schematic cross-section (not to scale). The heater side is 1/3 of the membrane width. The spiral layout is designed to guarantee a uniform temperature distribution.

DEVICE DESIGN

The microhotplate consists of a squared-shaped low-stress SiN_x membrane, with a side length of 1 mm and a thickness of 400 nm, and a spiral Pt microheater. The microheater is a 3.3 mm long and 18 µm wide spiral, set in the middle of the membrane, covering an area of 330 µm x 330 µm. It is equipped with four contact pads in order to measure the average resistance of the heated part with the four-point method. The spiral layout is designed to guarantee uniform temperature distribution. The CNT bundles are directly grown in the center of the suspended microhotplate. A 3D sketch and a detailed schematic cross-section of the proposed test structure are shown in Fig 1. In order to perform reliable measurements, three different configurations are processed on the same wafer: a bare sample without CNT bundles to be used as reference device; a group of six bundles, having a diameter of 20 µm each, uniformly distributed in a circular pattern (Fig 2(a)); a single bundle with a diameter of 200 µm (Fig 2(b)).

DEVICE FABRICATION

The starting material is a single side polished silicon (Si) wafer, with a thickness of 525 µm and 100 mm diameter. First, a 200 nm thick low stress LPCVD SiN_x layer is deposited to provide electrical insulation. This step is followed by the evaporation of 160 nm of Pt on an adhesion layer of 20 nm of Tantalum (Ta). A lift-off process is used to pattern the Ta/Pt layer into an 18 µm wide resistive spiral. A second layer of 200 nm of low-stress LPCVD SiN_x is deposited to seal the heater and to create a symmetrical thermal configuration. The final value of the spiral sheet resistance is equal to 1.76 Ω/□. Contact openings to the Pt contact pads are realized by dry etching of the SiN_x to allow electrical characterization of the microheaters. In order to release the 1 x 1 mm^2 membrane, the backside is patterned and the Si substrate is removed in a 33wt% KOH solution at 85°C, using SiN_x as hard mask.

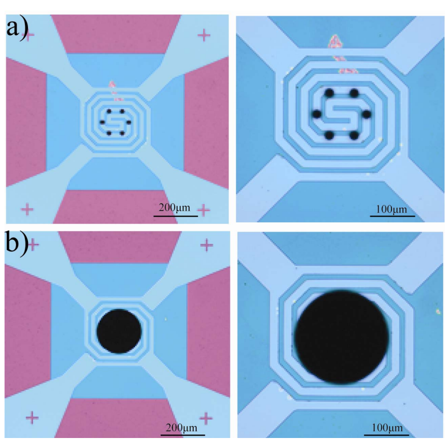

Figure 2: Optical microscope images of the fabricated devices. The CNT bundles directly grown on a suspended microheater are visible as black spots. The geometries fabricated are: a) six circular CNT bundles (diameter 20 µm each); b) a single large CNT bundle (diameter 200 µm).

The microhotplates are now ready for the CNTs growth. First, a 10 nm layer of Aluminum Oxide (Al_2O_3) is deposited by Atomic Layer Deposition (ALD). The Al_2O_3 layer is employed not only as buffer layer, but also to promote the formation of nanosized Iron (Fe) particles suitable for CNT growth. A 1.5 nm of Fe, catalyst layer for CNTs growth, is evaporated and patterned by lift-off, to define circular features of 20 µm and 200 µm. The microheater samples with the Al_2O_3/Fe catalyst are then loaded into a commercial system (Black Magic II, Aixtron) for CNT synthesis. The CNTs are grown using low-pressure CVD at 600°C. The CNTs height can be tuned by varying the process time. In our case, after five minutes, instead of the expected 150 µm high CNTs, a height of about 70 µm is achieved (Fig 3). The growth rate on a suspended membrane, although successful, is almost half of what is achieved on the bulk silicon substrate. This can be caused by a different thermal transfer in the released structure. Further, investigation is needed to identify all the possible causes and to optimize the growth process.

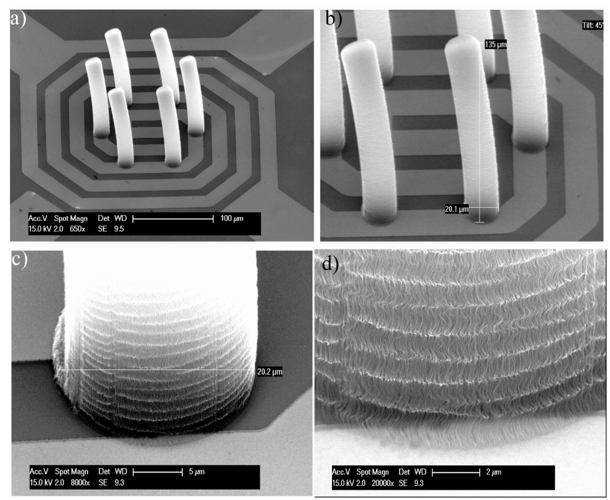

Figure 3: SEM images of the active area of the device: (a) Global view of the six CNT bundles on the microheater; (b) close-up of the CNT bundles. Each feature is 20 µm large and ~70 µm long; (c-d) close ups of the CNT base showing the good adhesion to the substrate;

MEASUREMENT PRINCIPLE

The purpose of this work is to measure the thermal properties of vertically aligned CNT bundles, using a microfabricated device. In our microhotplate without CNTs, the total heating power is composed by the effective power, defined as the amount of power necessary to raise the temperature of the test device, and the power loss. Under steady-state operating conditions, the electrical power applied to the microhotplate, P_{heat}, is dissipated into the surrounding area through natural convection, thermal radiation and conduction within the membrane material. The described thermal loss paths are defined in terms of power losses, P_{loss}. In first approximation, the heating power of our heater fully embedded in a membrane, can be expressed as:

$$P_{heat} = R_{heat} * I^2{}_{heat} = R_0(1 + \alpha\, \Delta T) * I^2{}_{heat} \quad (1)$$

were I_{heat} is the current applied through the terminals and R_{heat} is the Pt resistance in function of the heater temperature. The resistance of the platinum spiral changes linearly with the temperature, where, α corresponds to the temperature coefficient of resistance (TCR) and R_0 is the resistance value at ambient temperature.

To estimate the temperature of the heater, the Pt layer of a bare microhotplate is characterized to determine resistance and temperature coefficient of resistance (TCR). The reference sample experienced the same processing steps of the sample equipped with CNTs bundles, as they are processed on the same wafer. The extracted values are obtained performing a four-probe measurement.

The TCR, extracted from the relative change of the heater resistance with the temperature, corresponds to 2300 ppm K^{-1} with a standard deviation of 40 ppm K^{-1}. The TCR value is based on previously calibrated hotplates with the same Ta/Pt layer. Once the heater calibration curve is obtained, the heater can also be used as sensing element.

In the case of the microhotplate equipped with CNT bundles, additional thermal loss paths are introduced. In fact, the CNT bundles extend the surface area in contact with the environment, increasing the radiative and convective contributions (Fig 4). As the power losses increase, we need to supply more power to the entire system to reach the same temperature value of the reference microhotplate.

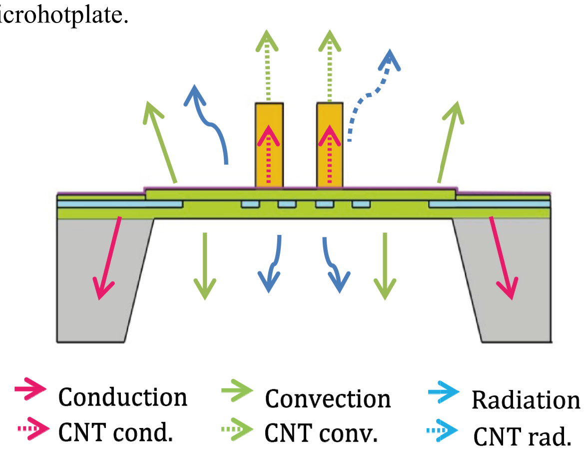

Figure 4: Schematic cross-section with possible thermal paths after the surface extension due to the CNTs growth. The additional contribution appears in terms of convective and radiative losses (dashed arrows).

DEVICE CHARACTERIZATION

Device characterization was performed using an Agilent 4156C Semiconductor Parameter Analyzer and a Cascade probe station with a temperature-controlled chuck.

As mentioned before, the microhotplate is connected to the contact pads by four Pt tracks, allowing four-probe resistance measurements. The measurements are performed in air. The recorded value of the resistance is representative of the average temperature of the microhotplate. The microhotplate was electrically heated up from room temperature to 400°C by supplying a stepwise current with steps of 2 s. In order to preserve the structural integrity of the device under test, the maximum input current value used is 10 mA.

RESULTS AND DISCUSSION

Our preliminary study is carried out on two types of CNT patterns: six circular CNT bundles, with a diameter of 20 µm each; a single big bundle, 200 µm in diameter. The six bundles occupy about 2% of the total microheater area, and the single bundle covers around 29% of the total area. The thermal performances of CNT geometries are extracted by comparison with a reference microhotplate without CNTs. Applying current between contact pads, Joule heating will heat the testing structure. The applied current and the corresponding voltage across the heating spiral are recorded.

In Figure 5, the normalized resistance (R/R_0) as function of the applied current for a six bundle and a big single bundle samples are shown. The normalized resistance of the microheater with CNT bundles is lower compared to the reference one. This is due to the additional CNT thermal contribution and related surface extension. In particular, at 10 mA up to 9% of resistance decrease between the reference and the single bundle, is recorded.

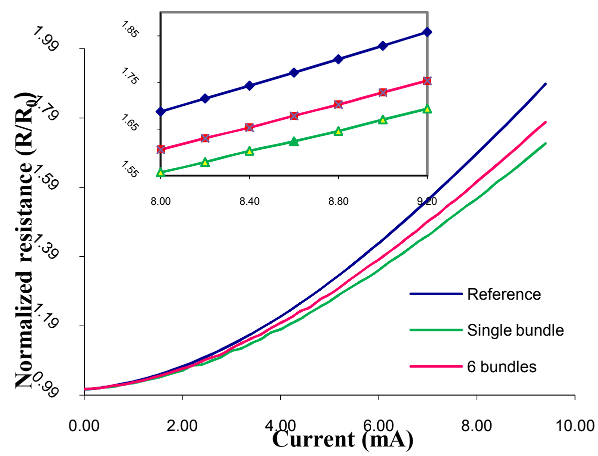

Figure 5: Normalized resistance (R/R_0) versus applied current. For a given current the resistance of the microheaters equipped with the CNTs is lower compared to the reference one (up to 9% at 10mA). The recorded data between 8mA and 9.2mA are shown in the inset.

Figure 6 shows the heating power of both reference and test structures versus temperature. As expected the power required to achieve a certain temperature value is higher for the microheater equipped with CNTs. For example, setting the temperature at 300°C the power surplus is 20% for the microhotplate equipped with 6 bundles and 31% for the single bundle. Results are summarized in Table 1.

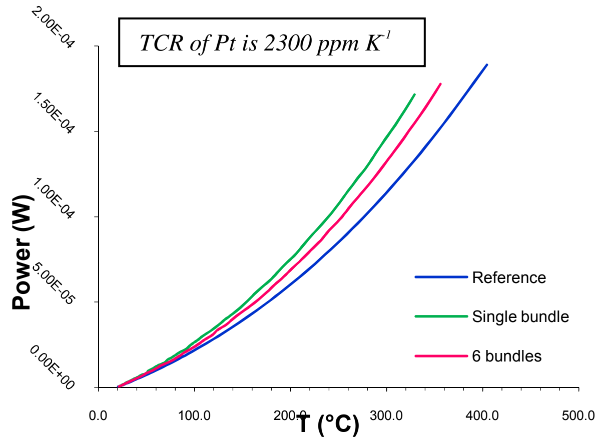

Figure 6: Steady-state power consumption versus temperature measured for a microhotplate with and without CNT bundles. The power required to achieve a certain temperature is higher for the microheaters equipped with the CNTs. At 300°C the power increase is 20% and 31% for the six bundles and the single bundle respectively.

Table 1: Summary of the preliminary results.

Sample	Resistance at T= 300°C	Power diss. at T= 300°C	Dissipation percentage
Reference	324 Ω	1,17 E-4	/
6 bundles	588Ω	1,34 E-4	~ 20%
Single bundle	570 Ω	1,54 E-4	~ 31%

As the measurement results show, the single bundle configuration is more effective then the six bundles one. Indeed, the total surface are of the single bundle is exactly 1.6 times larger than the total surface area of the six bundles added up together. This proportion is reflected also on the power percentage reported.

The recorded values confirm the effectiveness of the proposed measurement technique and the possibility of using the CNTs bundles as heat exchangers.

CONCLUSIONS

A simple method of measuring the dissipative power of vertically aligned CNTs has been proposed. A Pt/SiN_x microhoplate device has been used as test structure. CNT bundles as high as 70 µm were grown directly on top of the suspended microhotplate. No further processing steps are required after CNTs growth, leaving intact their properties. Using a differential measurement method, it is possible to emphasize only the CNTs contribution, shadowing the basic thermal effects of the bare membrane.

Heat characterization was performed measuring the dissipative power of two types of CNT patterns. When fixing the temperature at 300°C, 20% and 31% of power can be saved with the six bundles and single bundle geometries, respectively.

The described results demonstrate the dissipative power of the investigated CNT bundle geometries and the effectiveness of the proposed test microstructure for thermal performance measurement.

In order to quantify the magnitude of the thermal loss contributions, further investigation is required. In particular, a comparison between measurements recorded both in air and vacuum environment can help to extract relevant data such as the average thermal conductivity of the CNT bundle.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the technical support and advice of the staff at the Dimes Technology Centre. This work was performed in the framework of NanoNextNL Project.

REFERENCES

[1] X. Tong, *Advanced Materials for Thermal Management of Electronic Packaging*, Springer S. 2011.

[2] J. Che, T. Cagin, and W. G. III, "Thermal conductivity of carbon nanotubes," *Nanotechnology*, vol. 65, 2000.

[3] Y. Xu, Y. Zhang, E. Suhir, and X. Wang, "Thermal properties of carbon nanotube array used for integrated circuit cooling," *J. Appl. Phys.*, vol. 100, no. 7, p. 074302, 2006.

[4] M. Gaillard, H. Mbitsi, A. Petit, E. Amin-Chalhoub, C. Boulmer-Leborgne, N. Semmar, E. Millon, J. Mathias, and S. Kouassi, "Electrical and thermal characterization of carbon nanotube films," *J. Vac. Sci. Technol. B Microelectron. Nanom. Struct.*, vol. 29, no. 4, p. 041805, 2011.

[5] S. Vollebregt, S. Banerjee, K. Beenakker, and R. Ishihara, "Thermal conductivity of low temperature grown vertical carbon nanotube bundles measured using the three-ω method," *Appl. Phys. Lett.*, vol. 102, no. 19, p. 191909, 2013.

[6] A. J. McNamara, Y. Joshi, and Z. M. Zhang, "Characterization of nanostructured thermal interface materials – A review," *Int. J. Therm. Sci.*, vol. 62, pp. 2–11, Dec. 2012.

[7] F. Santagata, G. Almanno, S. Vollebregt, C. Silvestri, G. Q. Zhang, and P. M. Sarro, "Carbon Nanotube based heat-sink for solid state lighting," *8th Annu. IEEE Int. Conf. Nano/Micro Eng. Mol. Syst.*, vol. 1, pp. 1214–1217, Apr. 2013.

[8] A. Roncaglia, F. Mancarella, M. Sanmartin, I. Elmi, G. C. Cardinali, and M. Severi, "Measurement of Thermal Conductivity on Thin Films," pp. 20–23, 2006.

[9] D. Li, Y. Wu, P. Kim, L. Shi, P. Yang, and A. Majumdar, "Thermal conductivity of individual silicon nanowires," *Appl. Phys. Lett.*, vol. 83, no. 14, p. 2934, 2003.

CONTACT

*C. Silvestri, tel: +31630187549; C.Silvestri@tudelft.nl