CNTs as Ultra-Low-Powered Aqueous Flow Sensors in PDMS Microfluidic Systems

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***Abstract* — We have developed ultra-low-powered CNT aqueous flow sensors integrated in microfluidic channels using MEMS-compatible fabrication technology. The sensors are first built on a glass substrate with microelectrode array. Electronic-Grade CNTs (EG-CNTs) are then aligned between the microelectrodes using AC dielectrophoretic (DEP) technique, which are utilized as sensing elements. The EG-CNTs exhibit very stable adhesion to the electrodes, therefore greatly increase the repeatability of the sensors. The CNT sensors are enclosed in a microfluidic chamber by bonding a polydimethylsiloxane (PDMS) microchannel to the glass substrate with the CNT sensor array. We demonstrated the response of the CNT flow sensor to different aqueous flow rates at room temperature. The sensors only needed ultra-low-power (~1μW) to detect the DI-water flow rate ranging from 0.8ms^{-1} to 2.1ms^{-1}. Preliminary experiments show that there is a linear relation between the resistance changing ratio of the sensor and the flow rate. Our sensors show hysteresis of ~ 10% before temperature compensation. Based on our results, we proved the feasibility of utilizing CNTs as aqueous flow sensors with ultra-low-power consumption. Meanwhile, our results show great potentials of CNTs-based flow sensor for microfluidic, biomedical and bio-molecular applications.**

Keywords — ***Aqueous Flow Sensor, Carbon Nanotubes, CNT Sensor, Microfluidic System, Ultra-Low-Powered Sensor***

I. INTRODUCTION

Since the discovery of carbon nanotubes (CNTs), their distinct mechanical, electrical and physical properties have been intensively explored. The advantages of CNTs over conventional materials, such as small dimension, high mechanical strength, high electrical and thermal conductivities, and high surface-to-volume ratio, stimulate the utilization of CNTs as sensing materials in pressure, thermal, gas, and flow sensors [1]. Our current work aims to investigate the possibility of using CNTs as aqueous flow sensors. Kral and Shapiro [2] theoretically proposed phonon-induced electron drag effect and Columbic effect on the generation of electric voltage/current in a metallic CNT due to the flowing liquid. Ghosh [3] observed the voltage in the order of mV generated by the flow of a liquid over single-walled carbon nanotube (SWNT) bundles along the direction of flow. And the magnitude of the voltage depended sensitively on the ionic conductivity and the polar nature of the liquid. Liao [4] experimentally showed that the flow-induced current on the surface of multi-walled carbon nanotube (MWNT) thin films closely depended upon the flow velocity and temperature of the liquid. Ni [5] developed a fluid flow/shear sensor based on the vertically oriented MWNTs. By monitoring the polarization and intensity of the transmitted light through the MWNT mat, the shear force of fluid flow was determined. We have previously demonstrated simple CNT thermal and flow sensors [6] [7]. In this paper, we will discuss our latest study of utilizing CNTs as sensing elements to sense aqueous flow in a PDMS microfluidic system. We believe that, due to the extremely low activation energy required to use CNT flow sensors, they may offer advancements in flow sensing than conventional MEMS flow sensors, which suffer from high power consumption, induced hydrothermal motion and low selectivity.

II. SENSOR FABRICATION

The simplified representation of the fabrication process for the CNT flow sensor chip is outlined in Figure 1. Briefly, the microelectrode array was fabricated using a standard

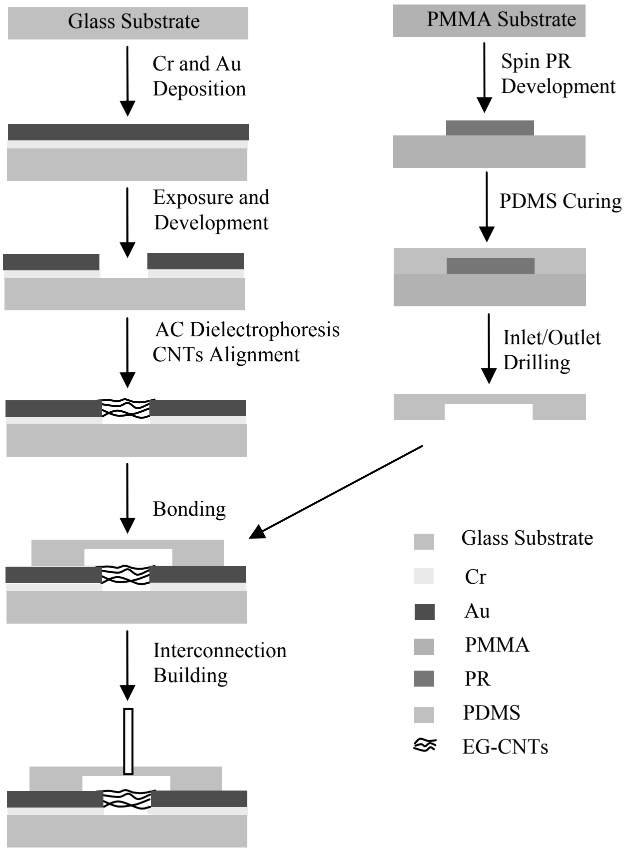

Fig. 1. Fabrication process for CNTs based aqueous flow sensor in PDMS microfluidic systems.

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lithography and wet chemical etching process. First, a layer of ~3000 Å Au was deposited onto the soda lime glass substrate after the deposition of an adhesion layer of ~1000 Å chromium (Cr) using a sputtering deposition process. Then EG-CNT (Brewer Science, Inc., Rolla, Missouri, USA) bundle was batch assembled between the microelectrodes to serve as sensing element by using the dielectrophoretic manipulation technique reported in [6]. Figure 2(d) shows the SEM image of DEP manipulated EG-CNTs. It can be seen that the EG-CNTs were well-aligned between the electrodes. The EG-CNTs showed very stable adhesion to the Au electrodes after DEP manipulation, therefore, a specific protection process to fix the CNTs onto the electrodes was not needed. Meanwhile, a PDMS (SYLGARD 184 Silicone Elastomer Kit, Dow Corning) microchannel was fabricated by using SU-8 molding method. The channel is 21mm long with a cross-section area of 500μm × 40μm. Even though a seal using thermal bonding has an advantage that the PDMS channel can be easily removed for cleaning, it may not withstand the high flow velocity and repeated use. In order to achieve a good seal to withstand the expected flow rate up to 5ms^{-1}, we produced a permanent seal between the PDMS and glass surfaces by exposing them to a plasma discharge in oxygen for 30 seconds at 0.5mBar, then immediately bringing them into contact. A prototype of CNTs-based aqueous flow sensor chip is shown in Fig. 2(a). Our flow tests presented in the later section of this paper proved that the plasma-treated bonding chip exhibited very good seal quality.

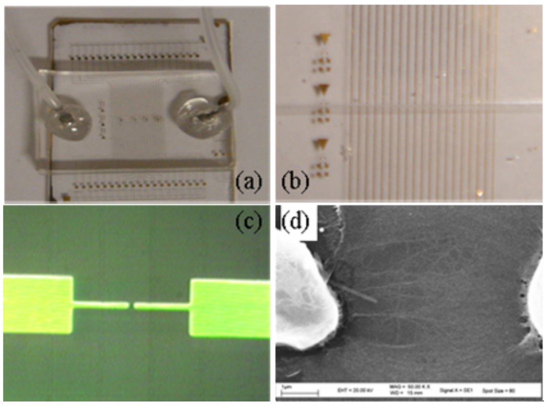

Fig. 2. (a) Photograph of an aqueous flow sensor with EG-CNTs as sensing elements; (b) Photograph of an array of EG-CNT sensors inside a PDMS channel (L × W × T=21mm × 500μm × 40μm); (c) Microscope image of a pair of gold microelectrodes (W × G = 5μm × 2μm); (d) SEM image of EG-CNT network between microelectrodes after DEP alignment.

III. EXPERIMENTAL SETUP

The experimental setup was integrated as shown in Fig. 3. A Versapump 6 (Kloehn Ltd., USA) syringe pump was used to generate the aqueous flow rate and inject the fluid into the CNT flow sensor chip. The fluid (DI-water) flowed in the direction perpendicular to the CNT bundle axis, while a SourceMeter (Keithley 2400, Keithley Inc., USA) was used to generate the operating current to activate the sensor and output the measured CNT resistance to the computer via a digital output port. A PCB board served as the interface for the sensor chip and the SourceMeter. We tested the response of the sensors in a constant current mode at room temperature.

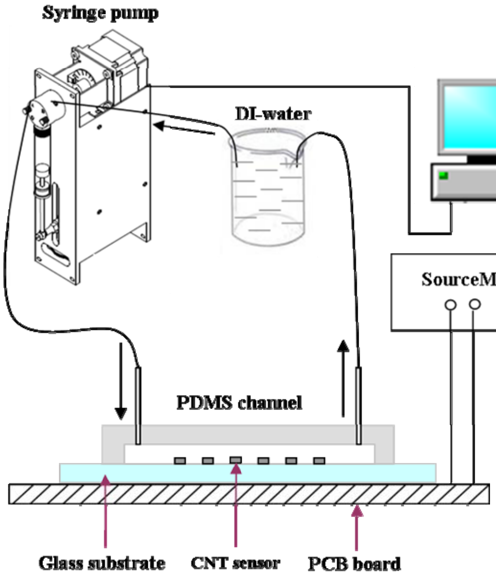

Fig. 3. Schematic diagram of the experimental setup for flow rate detection.

IV. EXPERIMENTAL RESULTS

A. Electrical Characterization of EG-CNTs

Our previous study showed that CNT bundle manipulated by DEP technique between electrodes was composed of complex network of individual CNT, and exhibited similar electrical properties as individual CNT. We have determined the I-V characteristics of bundled EG-CNTs at room temperature. The result of I-V measurement is shown in Fig. 4. As shown, the experimental measurements of voltage were quite close to those of Ohm's Law expectation for the current ranging from 0 to 50μA, which is called linear region. Outside the linear region, the higher the current, the bigger the deviation between the measured and expected voltages. That

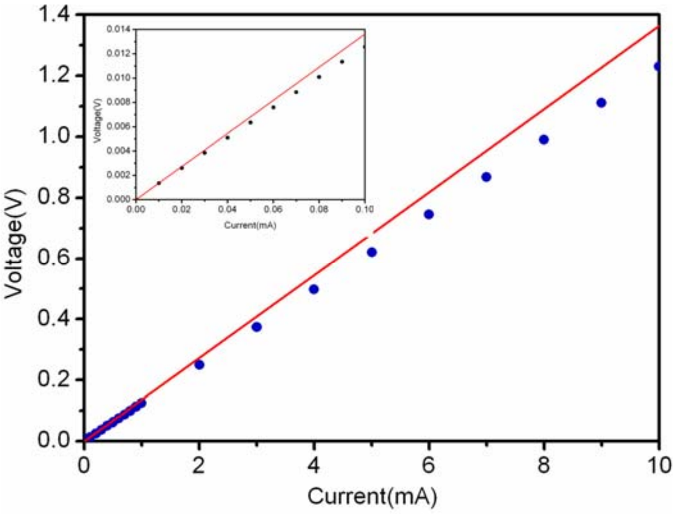

Fig. 4. I-V characteristics of EG-CNTs.

means CNTs experienced the pronounced temperature rise due to ohmic heating.

B. Typical Response

Aqueous flow sensing characterizations of our sensors were conducted in still air at room temperature. We observed in situ measurement of the electrical resistance of our sensor by cycling the chamber with DI-water from still to motive. After pumping the DI-Water to the microchannel, we waited ~10mins until no obvious resistance change was observed. The CNT resistance increased ~ 8.47% as shown in Fig. 5 due to flow running. Adequate time delay was allowed between tests to make the resistance recover to its original value. Output resistance responded linearly and stably for a total volume of 2.5ml of DI-water at a constant flow velocity of 1.6ms^{-1} inside the microchannel. After the cessation of fluid flow we observed a time-dependent recovery of the CNT resistance. We have not yet performed heating exposure test to optimize the recovery time.

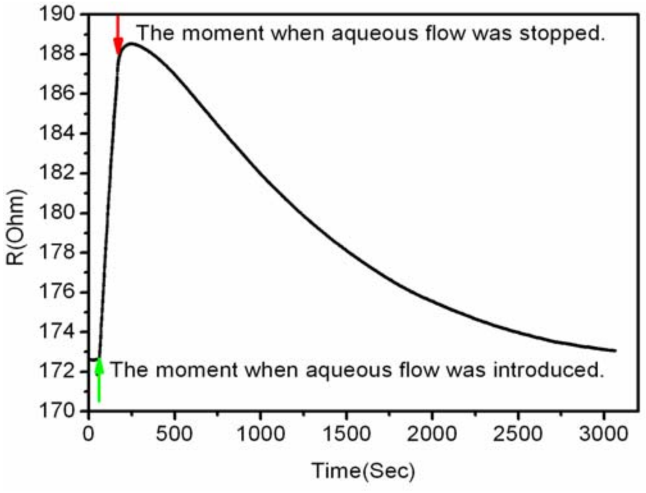

Fig. 5. Electrical resistance output vs. time.

C. Power Consumption

We have observed resistance change for different operating currents from 0.1mA to 1mA. Fig. 6 shows the sensing response of our sensor at room temperature upon exposure to DI-water flow. The resistance of the sensor was found to increase linearly for all these three currents. And higher resistance change was found under higher current. However, the higher current may produce higher operating temperature, which may burn the CNTs. In all the experiment reported in this paper, to balance between the working temperature and sensitivity, we activated the sensors at 0.1mA with a relatively accurate digital output of our SourceMeter, then the power consumption of our sensor was only ~1-2µW. That suggests we can activate our flow sensors with an ultra low power.

D. Repeatability

Fig. 7 shows the observed sensor resistance change during repeated DI-water flow running at 1.3ms^{-1} at room temperature. The resistance of our sensor increased each time the flow was pumped into the microchannel, and recovered after the flow stopped. The full reversal of these characteristics upon flow cessation may have important implication. This CNT behavior is quite similar to [8]. The reversibility in the electrical property may indicate that the Au electrode-nanotube contact is not affected when the flow passes over the CNTs. A possible mechanical effect of the fluid on the CNTs may account for the observed change in sensor resistance, that deserves further study on fundamentally understanding the sensing mechanism of CNT aqueous flow sensor.

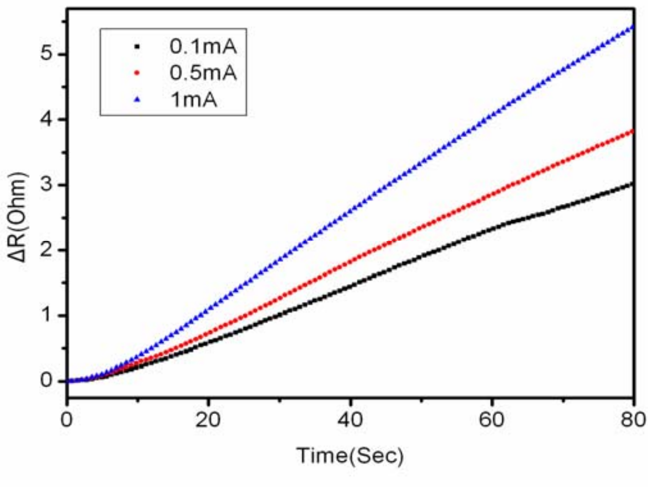

Fig. 6. Electrical resistance of sensor for different currents.

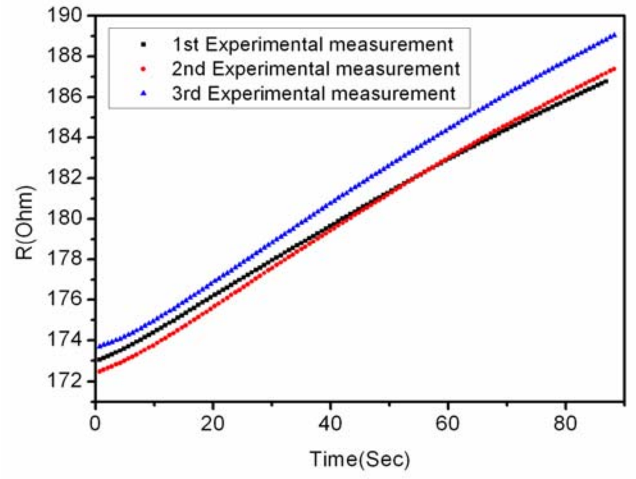

Fig. 7. Sensor resistance at flow rate of 1.3ms^{-1}.

E. Flow Sensitivity Test

The sensor's responses to different aqueous flow rates varying from 0.8 to 2.1ms^{-1} have been measured and the results are shown in Figure 8. During the tests, the flow rates were well-controlled by a syringe pump with 0.2% accuracy. The resistance ratio of 2.1ms^{-1} DI-water flow was 0.182Ωs^{-1}. For the same sample, lowering the flow to 0.8ms^{-1} led to response ratio of 0.0718Ωs^{-1}. It is evident that the sensitivity and resistance of the sensor increase almost linearly with the increase of DI-water flow from 0.8 to 2.1ms^{-1}. Fig. 9 shows the flow dependency on the sensitivity of CNT sensors before temperature compensation. As shown, when the flow rate changed from low to high, the sensor sensitivity increased linearly to the flow rate. And, without temperature compensation the hysteresis of our sensor was approximately 10 %.

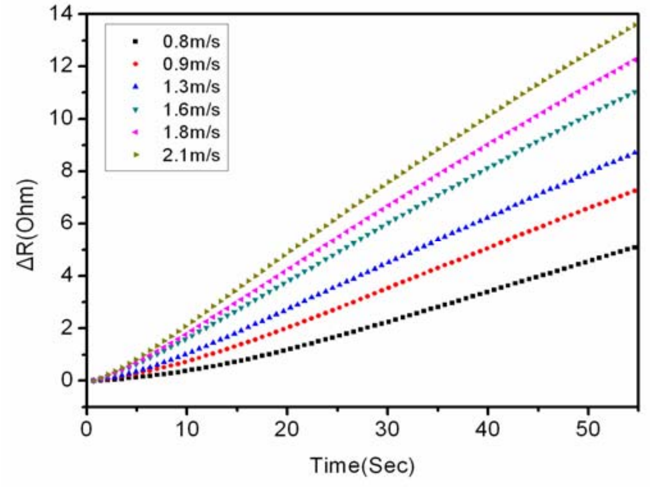

Fig. 8. Electrical resistance of sensor vs aqueous flow velocity.

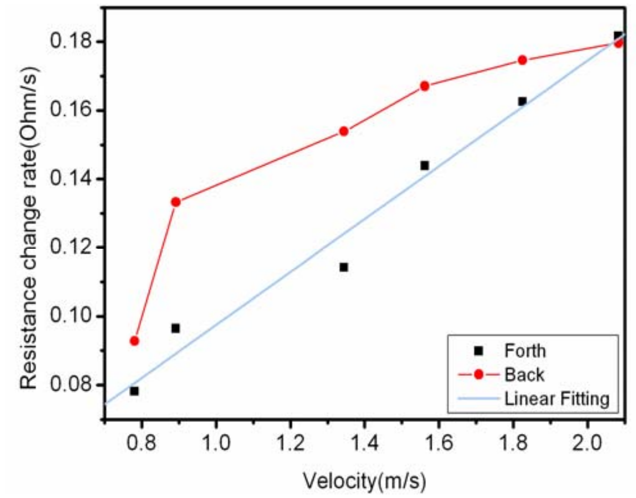

Fig. 9. Flow test without temperature compensation.

V. CONCLUSIONS

In conclusion, CNT aqueous flow sensors in PDMS microfluidic systems have been designed and fabricated by MEMS-compatible fabrication technology. We demonstrated the linear relation between aqueous flow rate of DI-Water and resistance changing ratio of CNT sensors. Moreover, the sensor can be operated at an ultra-low-power level (~1μW) with acceptable sensitivity. In addition, the reversibility of the electrical property of CNT sensors indicates the EG-CNTs show a very stable connection to the Au electrodes. In the future, we will focus on further understanding the sensing principle of our sensors. The sensitivity of our CNT sensors for different microfluidic flow media will also be investigated.

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