Proceedings of the 3rd IEEE Int. Conf. on
Nano/Micro Engineered and Molecular Systems
January 6-9, 2008, Sanya, China

Ultra-Low-Powered CNTs-Based Aqueous Shear Stress Sensors Integrated in Microfluidic Channels

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***Abstract* —We have developed carbon nanotubes (CNTs) based aqueous shear stress sensors integrated in microfluidic channels. The sensors utilized electronics-grade carbon nanotubes (EG-CNTs) as sensing elements, and were built by combining MEMS-compatible fabrication technology with AC dielectrophoretic (DEP) technique. The assembled sensing element has a room-temperature resistance of ~100 to 200Ω by using the original concentration of 1:1 EG-CNTs in DI-water. The I-V measurements of EG-CNTs show the heating effects of the sensors, and the current required to induce the nonlinearity of EG-CNTs is in the order of 100μA, which implies the operation power of the sensor is in the range of μW. Upon exposure to DI-water flow, the characteristics of the sensor have been investigated at room temperature under constant current (CC) activation mode. It was found that the electrical resistance of the CNT sensors increased linearly with the introduction of constant fluidic shear stress. We have tested the response of the sensors with flow velocity from 0.3 to 3.4m/s. The experimental results show that there is a linear relation between the output resistance change and the flow velocity to the one-third power. This result proved that the CNT sensors work with the same principle as conventional MEMS thermal shear stress sensors but only require ultra-low activation power (~1μW), which is ~1000 times lower than that of conventional MEMS thermal shear stress sensor.**

Keywords — ***Aqueous Shear Stress Sensors, Carbon Nanotubes, CNT Sensors, Microfluidic Channels, Ultra-Low-Power Sensors***

I. INTRODUCTION

A fluid flowing past a surface boundary exerts normal and tangential stresses on the surface. The tangential stress is called the surface or wall shear stress [1]. The ability to accurately measure time-resolved wall shear stress in flows impacts a broad application spectrum that ranges from fundamental scientific research to industrial process control and biomedical applications. In particular, the measurement of flowrate, pressure, and shear stress based on MEMS technology is of great importance for many fluid-related studies and applications, such as micro/nano fluidic, biomedical, and bio-molecular systems [2]. With the use of MEMS technology, the size of the conventional shear stress sensors (e. g., hot-wire sensors) has been greatly reduced, while uniform geometry and consistent performance have been improved in the past decade, and miniature polysilicon shear stress sensors are now available [3]. Typical micromachined thermal shear stress sensors have been successfully developed for aerial and underwater applications, which use polysilicon resistors as sensing elements and consist of suspended vacuum cavity underneath the sensing elements to reduce conductive heat loss to the substrate [4, 5]. However, the size of existing polysilicon sensors is still in hundred-micron range, which may not be suitable for some scientific applications that require smaller size sensors. In addition, their power dissipation (in the range of mW) is still relatively high, i. e., the heat generation from the sensors may affect the minute fluidic motion through thermal convection, crippling their abilities to sense the true fluidic flow parameters. Hence, our current work aims to develop extremely small and low-power-dissipation aqueous shear stress sensors that will minimize disturbance to the flow-fields.

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, have already stimulate the utilization of CNTs as novel sensing materials for pressure, thermal, gas, and flow sensors [6]. Therefore, in the past few years, CNTs have drawn attention from worldwide researchers to investigate the possibility of using them as micro shear force/flowrate sensors. In 2003, Ghosh et al., [7] observed the voltage in the order of mV generated by an aqueous flow 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 aqueous medium. Similarly, Liao et al., [8] observed the flow-induced current on the surface of multi-walled carbon nanotube (MWNT) thin films, which was experimentally found to closely depend upon the flow velocity and temperature of the aqueous medium. In 2007, a fluid flow/shear shear sensor was developed based on the vertically oriented MWNTs by Ni et al, [9]. By monitoring the polarization and intensity of the transmitted light through the MWNT mat, the shear force of fluid flow was determined. In earlier publications we have demonstrated the utilization of CNTs for fluidic/thermal measurements, and later as a novel material for pressure sensing elements [10-13]. In this paper, we will demonstrate our latest results of using aligned EG-CNTs to detect aqueous shear stress in microfluidic channels.

II. EXPERIMENTAL DETAILS

The CNT aqueous shear stress sensor chip was built by combining MEMS-compatible fabrication technology with AC dielectrophoretic (DEP) technique. The Au microelectrode array was first fabricated on the soda lime glass substrate by using the conventional lithography and wet chemical etching process (Fig. 1). Then, EG-CNTs (BSI-CNT-16, Brewer

This project is funded by Hong Kong Research Grants Council (Project code: CUHK/413906), Hong Kong Innovation and Technology Fund (Project code: ITS/027/06), and National Natural Science Foundation of China (Project code: 60675060).

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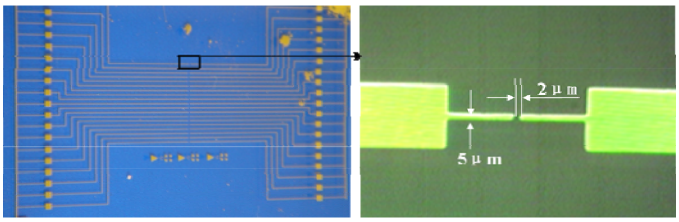
978-1-4244-1908-1/08/$25.00 ©2008 IEEE.

Science Inc., Missouri) were batch aligned between the microelectrode pair to serve as the sensing element by AC DEP with a sine wave voltage of 16V peak-to-peak and 1MHz. Fig. 2 shows a typical SEM image of DEP manipulated CNTs between a microelectrode pair. The details of the DEP fabrication process can be found in our previous paper [11]. Our experimental results proved that the EG-CNTs exhibited very stable adhesion to the Au electrodes after DEP assembly, therefore, a specific protection process to fix the CNTs onto the electrodes was not needed. Finally, the CNT sensors were permanently integrated in glass-polydimethylsiloxane (PDMS) microfluidic channels (21mm long, 500µm wide, and 40µm tall) by standard glass-PDMS bonding process [13].

The experimental setup was integrated as shown in Fig. 3. A Versapump 6 (Kloehn Ltd., USA) syringe pump was used to control the 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 also to provide the CNT resistance values to the computer via a digital output port. A PCB board served as the interface for the sensor chip and the Sourcemeter. The Reynolds number based on the channel dimension and average velocity was about 326, meaning a laminar flow. The distance between a typical CNT sensor in the channel and the inlet was ~8mm, which ensured that the



(a) (b)

Figure 1. (a) Photograph of the fabricated Au microelectrode array on a glass substrate. (b) Optical image of an Au microelectrode pair before CNTs assembly.

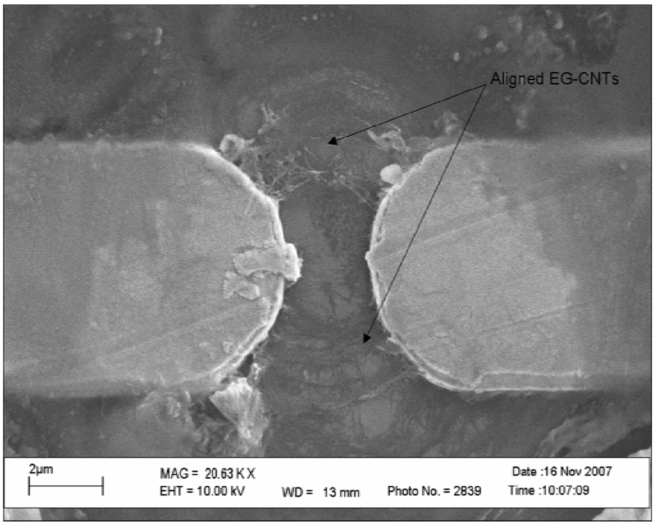


Figure 2. SEM image showing the formation of EG-CNTs between an Au microelectrode pair after DEP force is applied.

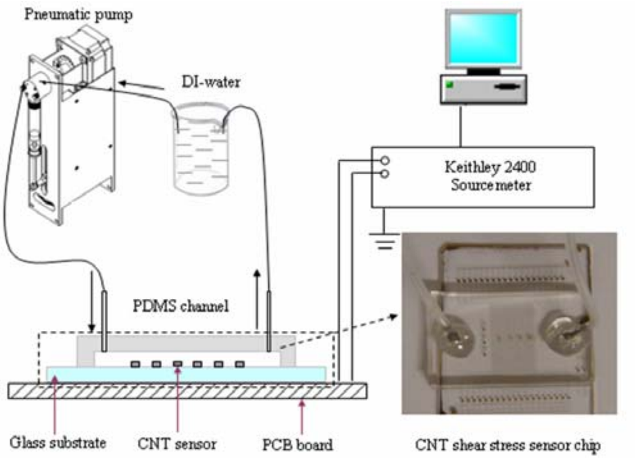


Figure 3. Schematic diagram of the experimental setup for flow rate detection.

sensor was in the fully developed laminar flow region. Resistance measurements were conducted under CC activation mode. Furthermore, prior to each measurement, the sensor was activated and saturated with DI-water for ~20mins to eliminate the influence of sudden change of humidity, and flow impact. After each measurement, adequate time delay (~30mins) was allowed for the resistance to recover to its original value.

III. Experimental Results and Discussion

A. *Characteristics of EG-CNTs*

1) *I-V Curve*

The assembled sensing element has a room-temperature resistance of ~100 to 200Ω by using the original concentration of 1:1 EG-CNTs in DI-water. The I-V characteristics of the CNT sensors was obtained inside a programmable oven (KBF-115, Binder Co., German), which had a well-controlled chamber temperature and humidity. For the I-V curve tests, the temperature and humidity have been set as 24°C and 50%, respectively. A typical I-V curve of the CNT sensors is shown in Fig. 4. The inset shows that EG-CNTs began to exhibit an obvious I-V nonlinearity at ~100µA, with an overheat ratio of ~8.03%. This implies that the CNT sensors could be used as thermal flow sensors with as little as a few µW of input power. The nonlinear relationship proved that the EG-CNTs experienced a pronounced temperature rise due to Joule heating.

2) *Temperature Coefficient of Resistance*

The temperature-resistance relationship of the CNT sensors was measured and determined by putting the CNT sensors inside the Binder oven, whose temperature was controlled from 20°C to 60°C with 5°C increment with a constant humidity of 50%. Each incremental temperature was kept for 20mins in order to reach thermal equilibrium. A typical measured data of a CNT sensor is plotted in Fig. 5, which has a negative TCR of ~0.127%°C^{-1}. In general, the absolute TCR value of the EG-CNTs based on around twenty sensors ranged from 0.1 to 0.4%°C^{-1}.

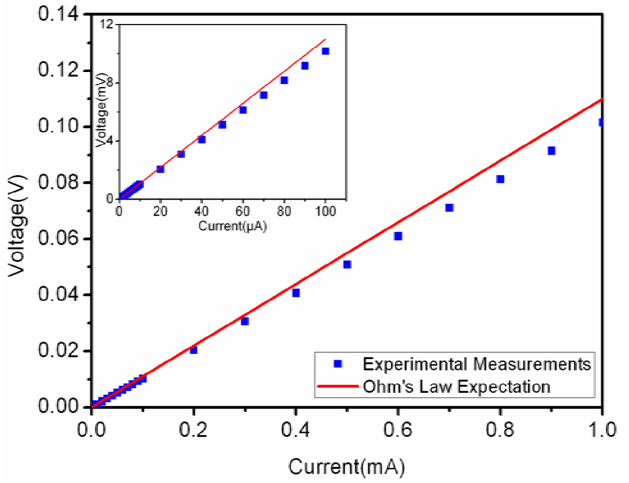


Figure 4. Typical I-V characteristics of EG-CNTs.

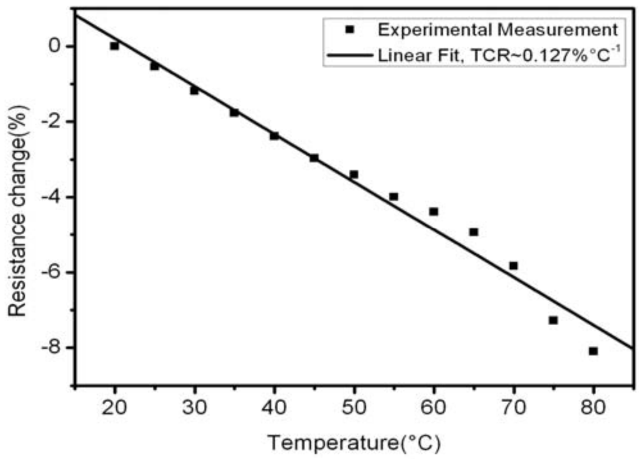


Figure 5. The temperature coefficient of resistance (TCR) of EG-CNTs under 50% humidity.

Then the CNT surface temperature change was determined by using the data from Fig. 4 and the measured TCR of EG-CNTs. The estimated temperature change of CNTs is ~63.2°C at 100μA. Hence, it is clear that the thermal coupling between the EG-CNTs and glass substrate is rather weak compared with its counterpart, polysilicon, so that the heat loss to the substrate has been minimized. At relative low current input, the EG-CNTs had a high enough temperature to allow convective heat transfer for flow sensing. This is very important for the implementation of a successful shear stress sensor, and also a big disadvantage for traditional MEMS thermal shear tress sensors. Because metal or polysilicon are routinely used as the heating and sensing elements, the resistance of sensing elements is relatively low and a large biasing current is typically required to produce adequate surface-heating effects.

B. *Micro flow rate measurements*

The response of the CNT sensors was measured at room temperature under constant current (CC) activation mode by cycling the chamber with DI-water from still to dynamic flow. As shown in the inset of Fig. 6, with the activation current of

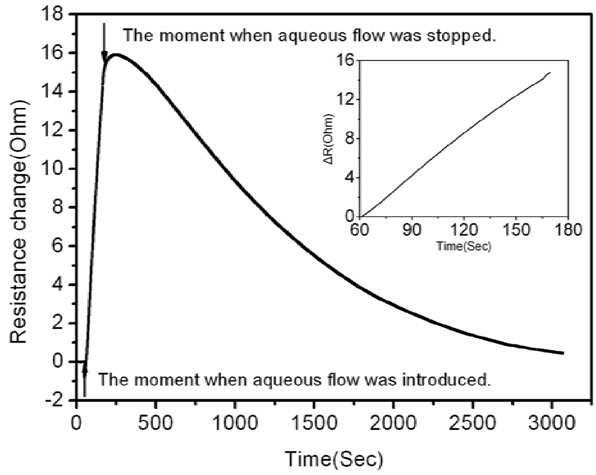


Figure 6. Typical resistance change of EG-CNT sensor with the introduction of DI-water flow of 1.8ms^{-1}.

100μA, the resistance was observed to increase linearly and stably upon exposure to a total volume of 2.5ml of DI-water at a constant flow velocity of 1.8ms^{-1} inside the microchannel. The CNT resistance increased ~8.5% over 110 seconds due to flow introduction. After the flow was stopped, we observed a time-dependent recovery of the CNT resistance.

We observed further that different activation currents elicited different resistance responses at the same flow rate. As shown in Fig. 7, with activation current of 1mA, the CNT resistance also showed linear response to the flow introduction. However, when the activation current is ranged from 5 to 17mA, the sensor seemed to show a compounded response. The resistance first increased linearly after the flow introduction, and then a significant linear decrease dominated the remaining process. The higher the current, the bigger the resistance decreased. After 20mA, the resistance response differed in both profile and magnitude from those two previous cases. It reached to its maximum in 1.4sec, and then dropped. Once the flow stopped, resistances in all those three cases can recover to their original values. We conducted these tests on several sensors, and similar behaviors were found. Therefore, we deduced that there obviously existed a current-related resistance decrease for higher activation currents. For the time being, the reason for such a change of resistance is unclear. As a result, we presently activate our sensor at a current smaller than 1mA to avoid long term damage to sensor due to potential high current density, to minimize the current-induced resistance change and natural convection caused by the heating of the resistive element, we choose 100μA as the activation current, thus the power input to the sensor is only ~1 to 2 μW, i. e., the voltage across the sensors are ~ 10 to 20mV.

C. *Sensitivity experiments*

The dynamic sensing response of EG-CNT sensors were measured at room temperature upon exposure to DI-water

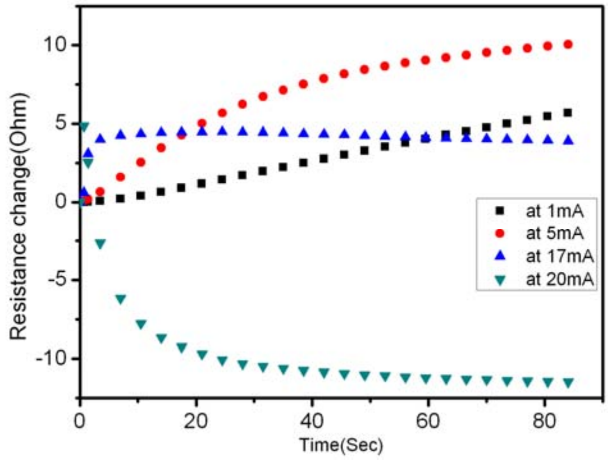


Figure 7. Sensor response at different activation currents with DI-water flow of $1.3ms^{-1}$.

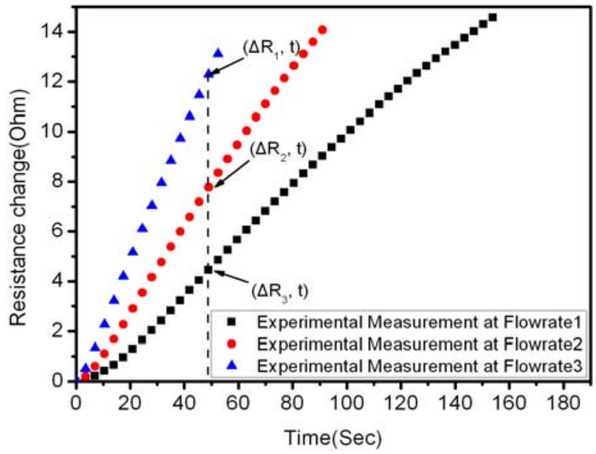


Figure 8 The change of resistance of EG-CNT sensors with DI-water flow of different velocity.

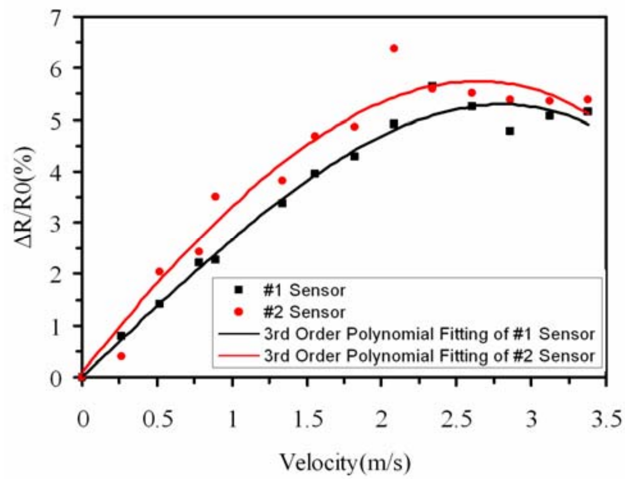
flow with different flow rate ranging from 0.3 to $3.4ms^{-1}$. Fig. 8 shows the change of resistance of EG-CNT sensors under different flow rates. We calculated the change of sensor resistance to different flow velocities within the same time range of t, which is determined to be smaller than the time of flow introduction for the highest tested velocity. As shown in Fig. 9 (a), when the velocity exceeded 2.3 ms^{-1}, the CNT sensors showed very little responsivity. However, it is evident that the change of resistance can be plotted as a linear function of the flow velocity to the one-third power when v is under 2.3 ms^{-1} (Fig. 9(b)), which is consistent to the thermal transfer principle of conventional thermal shear stress sensors[1].

IV. Conclusions

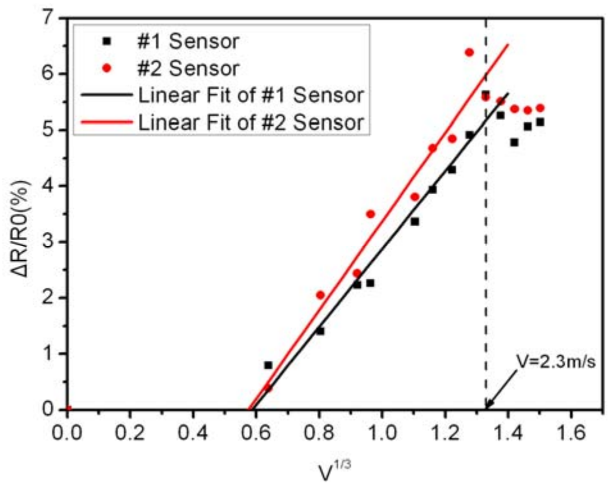
In conclusion, we demonstrated aqueous shear stress sensors based on bulk aligned EG-CNTs in PDMS microfluidic channel. The electrical resistance of the sensor was found to increase linearly with the introduction of DI-water flow. Upon exposure CNTs to different flow rates, the experimental results show that a one-third power relationship exists between the change of resistance and the flow velocity. Our results proved the feasibility of using EG-CNTs as aqueous shear stress sensors with ultra-low-power consumption (~1 to 2μW), which is ~1000 times less than that of conventional thermal shear stress sensors. Compared with other MEMS-based shear stress sensors, EG-CNT shear stress sensors possess the unique advangages of ultra-low activation power, low operation temperature, and minimized size. Hence, CNT sensors are promising devices for flow rate, shear force and biomedical sensing applications in micro and nano scales. In the future, we will focus on further investigation of CNT sensors' selectivity based on molecular functional groups, responsivity to different microfluidic flow media, and reproducibility.

Acknowledgment

The authors acknowledge support from the Hong Kong Research Grants Council, Hong Kong Innovation and Technology Fund and the National Natural Science Foundation of China.



(a)



(b)

Figure 9. (a) Output resistance change vs. flow velocity; (b) Output resistance change vs. velocity to the one-third power.

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