Ultra-Low-Powered Aqueous Shear Stress Sensors Based on Bulk EG-CNTs Integrated in Microfluidic Systems

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Abstract—Novel aqueous shear stress sensors based on bulk carbon nanotubes (CNTs) were developed by utilizing MEMS-compatible fabrication technology. The sensors were fabricated on glass substrates by batch assembling electronics-grade CNTs (EG-CNTs) as sensing elements between microelectrode pairs using dielectrophoretic (DEP) technique. Then, the CNT sensors were permanently integrated in glass-polydimethylsiloxane (PDMS) microfluidic channels by using standard glass-PDMS bonding process. Upon exposure to DI-water flow in the micro channel, the characteristics of the CNT sensors were investigated at room temperature under constant current (CC) mode. The specific electrical responses of the CNT sensors at different currents have been measured. It was found that the electrical resistance of the CNT sensors increased noticeably in response to the introduction of fluid shear stress when low activation current (~1μA) was used, and unexpectedly decreased when the current exceeded 5mA. We have shown that the sensor could be activated using input currents as low as 100μA to measure flow shear stress. The experimental results showed that the output resistance change could be plotted as a linear function of the shear stress to the one-third power. This result proved that the EG-CNT sensors can be operated as conventional thermal flow sensors but only require ultra-low activation power (~1μW), which is ~1000 times lower than conventional MEMS thermal flow sensors.

Index Terms—Aqueous Shear Stress Sensors, Carbon Nanotubes, CNT Sensors, Microfluidic System, Ultra-Low-Powered Sensors

I. INTRODUCTION

A fluid flowing past a 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 flow rate, 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].

Wall shear stress sensors are categorized by measurement method into two distinct groups-direct and indirect techniques. MEMS shear stress sensors based on these two principles have already been demonstrated [3, 4]. The well-developed MEMS shear stress sensors are typically micromachined thermal shear stress sensors for aerial and underwater applications [5, 6]. They use polysilicon resistor as the sensing element, and their sensitivity could be improved by an order of magnitude if an underlying vacuum cavity is used.

Actually, minimized size, low operation temperature, and low power consumption are always crucial and decisive for flow sensor applications. With the use of MEMS technology, the size of the shear stress sensors has been greatly reduced, while uniform geometry and consistent performance have been improved in the past decade, 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, it is our long term objective to develop extremely small and low-power-dissipation shear stress sensors that will minimize disturbance to the flow-fields. Since CNTs have unique electrical conductivity, mechanical strength and nanometer scale size, they have drawn intensive attention from worldwide researchers to investigate the possibility of using them as flow sensors. A flow sensor based on SWNT bundles was developed to produce an electrical signal in response to a fluid flow in 2003 [7]. Recently, vertically oriented MWNTs were used to determine the shear...
force of fluid flow by monitoring the polarization and intensity of transmitted light through MWNT mat [8].

In earlier publications in 2003, we have presented the utilization of CNTs for fluidic/thermal measurements, and later as a novel material for pressure sensing element [9-12]. In this study, we will present our recent demonstration of using ultra-low-powered EG-CNTs based aqueous shear stress sensors in microfluidic systems. The operation principle of a thermal transfer principle based shear stress sensor is first introduced, and will eventually be used to explain our experimental observations. Then, we present the fabrication process of the aqueous shear stress sensors, which utilized EG-CNTs as sensing elements and were integrated in PDMS microfluidic channels. The characteristics of EG-CNTs, including electrical characterization, and temperature coefficient of resistance (TCR), are then discussed, which allowed us to characterize the sensors for aqueous flow measurements and to understand the responses of the sensors. We first used constant DI-water flows to validate the sensors’ response to different liquid motions using constant current (CC) activation mode. Then we used different flow rates and activation currents for further investigation. The experimental results were found to be in close agreement with the theoretical prediction, indicating that EG-CNT sensors operate using thermal transfer principles as with conventional thermal shear stress sensors.

II. THERMAL TRANSFER PRINCIPLE AND THEORY

The operating principle of thermal shear stress sensors has been well-discussed [1, 5, 6]. Briefly, a shear stress sensor consists of a sensing element, which is located on the surface of the substrate and possesses a pronounced temperature dependence of resistivity. When current is applied to the sensing element, Joule heating will increase the temperature of the sensing element. And, once the flow is introduced onto the heated element, due to the interaction between the flow and the heated element, the temperature of the heated element will be decreased. Thus its resistance will decrease if the sensing element has a positive TCR, or increase if it has a negative TCR. The rate of heat loss from a heated resistive element to the ambient is dependent on the velocity profile in the boundary layer.

A parameter governing the operation of a thermal principle based sensor is the overheat ratio defined as

$$\alpha = \frac{R_0 - R_S}{R_S} \quad (1)$$

where $R_0$ is the resistance of the sensor at given power input, and $R_S$ is the resistance at reference temperature.

The resistance $R_0$ at temperature $T$ is given by

$$R_0 = R_S[1 + \alpha_T(T - T_0)] \quad (2)$$

where $\alpha_T$ is the temperature coefficient of resistance of CNTs, and $T_0$ is the reference temperature.

Ideally, the relationship between the input power $P$, the temperature of the thermal element and the shear stress $\tau$ for laminar flows can be typically described by

$$i^2R = \Delta T(A\rho \tau)^{1/3} + B \quad (3)$$

where $i$ and $R$ are the activation current and resistance of the sensor at given flow rate input, respectively. $\Delta T$ is the average temperature difference between the heated element and ambient. $A$ is a fluid related constant, $B$ is the heat loss to the substrate, and $\rho$ is the density of the fluid.

The shear stress for a fully developed laminar flow in a duct can be calculated by [13]

$$\tau = 8\phi(n)\frac{\mu v}{D_h w} \quad (4)$$

where $D_h$ is the hydraulic diameter, and $\phi(n)$ is a correction factor that is a function of $h/w$, $h$ and $w$ are the height and width of the channel, respectively, $v$ is the mean flow velocity.

Then, replacing $\tau$ in Eq. (3) with Eq. (4) yields

$$i^2R = \Delta T(A^3v^{1/3} + B) \quad (5)$$

Replacing $R$ in Eq. (5) with $R_0 + \Delta R$ yields

$$i^2(R_0 + \Delta R) = \Delta T(A^3v^{1/3} + B) \quad (6)$$

Note that at CC mode, the operating temperature of sensor is not a constant at different shear stresses. Therefore, the over heat ratio under CC mode is defined at zero shear stress [5]. Hence, at zero shear stress, there exists the following relationship

$$i^2R_0 = \Delta TB \quad (7)$$

Thus, Eq. (6) is simplified to

$$\Delta R = R_0A^3v^{1/3} \quad (8)$$

So, we finally get

$$\frac{\Delta R}{R_0} = A^3v^{1/3} \quad (9)$$

We will show in this paper later that the EG-CNTs do indeed respond to flow rate according to the above equation.

III. EXPERIMENTAL DETAILS

A. Sensor Design and Fabrication

Fig. 1 is the simplified representation of the fabrication process for the CNT flow sensor chip. The microelectrode array was fabricated using a standard 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) by using a sputtering deposition process. Then EG-CNTs (BSI-CNT-016, Brewer Science Inc., USA) were batch assembled between the microelectrodes to serve as the sensing element by utilizing the DEP manipulation technique reported in [10]. Fig. 2 shows the SEM image of DEP manipulated EG-CNTs. As shown, the EG-CNTs were well-assembled between the electrodes. Our later experiments proved that 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 Co., USA) microchannel was fabricated by using SU-8 molding method. The channel is 21mm long with a cross-section area of 500μm x 40μm. Note that to seal the microchannels by using conventional thermal bonding technique has the advantage that the PDMS channels can be easily removed for cleaning; however, the channel may not withstand a high liquid flow pressure and repeated uses. In order to achieve a good seal to withstand the expected flow rate of up to 5ms⁻¹ in our flow experiments, we produced a permanent bond between the PDMS and glass surface 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. 3. Our flow tests presented in the later section of this paper proved that the process of plasma treatment of PDMS and glass substrate before bonding them together enhances the seal quality significantly.

### B. Experimental Setup

The experimental setup was integrated as shown in Fig. 4. 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 sensor was in the fully developed laminar flow region. Resistance measurements were conducted under CC 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.
IV. RESULTS AND DISCUSSION

A. Characteristics of EG-CNTs

1) I-V Curve

First, the I-V characteristics of the CNT sensors were 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. 5. The inset of Fig. 5 shows that EG-CNTs began to exhibit an obvious I-V nonlinearity at ~100μA, with an overheat ratio of ~6.8%. This implies that the EG-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 an oven, whose temperature was controlled from 20°C to 80°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. 6, which has an average negative TCR of ~0.117%°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.

Then the CNT surface temperature change was determined by using the data from Fig. 5 and the measured TCR of EG-CNTs. Hence, the temperature change can be plotted as a function of the input current. As shown in Fig. 7, 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 have 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 stress sensors. Because metal or polysilicon is routinely used as the heating and sensing element, whose resistance is relatively low, and a large biasing current is typically required to produce adequate surface-heating effects.

B. Sensor sensitivity

1) Typical response

Aqueous flow sensing characterizations of the CNT sensors were conducted at room temperature. We measured the electrical resistance of the EG-CNT sensors by cycling the chamber with DI-water from static to dynamic flow. With the activation current of 100μA, output resistance responded linearly and stably for a total volume of 2.5ml of DI-water at a constant flow velocity of 1.8ms⁻¹ inside the microchannel as shown in Fig. 8. The CNT resistance increased ~8.5% over 110 seconds due to flow introduction. After the cessation of flow we observed a time-dependent recovery of the CNT resistance.

We observed the sensor’s response and reproducibility through repeated introduction of DI-water with the same flow rate of 2.1ms⁻¹ for three cycles. In Fig. 9, we show that the sensor responded in the same way for all three cycles. And the value of resistance increase was very repeatable. Resistance
The reversibility of EG-CNTs is quite similar to [14]. The consistent and repeated response in the electrical property of EG-CNTs may indicate that the Au electrode-nanotube contact is not affected when the flow passes over the EG-CNTs, which deserves further study on fundamentally understanding the connection mechanism between EG-CNTs and Au electrodes.

2) The Effect of the Current and Power Consumption

We observed further that different activation currents elicited different resistance responses at the same flow rate. As shown in Fig. 10, 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 larger 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. These phenomena could be explained by the traditional heat transfer theory [15]. When activation current is applied to a sensor, electrical energy is converted into heat energy by Joule heating within the sensor. This heat energy is dissipated in three ways. Part of this thermal energy is lost through the substrate; some of it is transferred to the flow via forced convection; and the rest is stored as internal energy, thus increasing the sensor temperature. Therefore, ignoring the heat lost to the substrate (that is, assuming that heat conduction across the CNTs sensing element is much faster than heat conduction from the CNTs to the substrate), at relatively low activation current i.e., less than 1mA, most of the heat energy is dissipated to the flow, and very little heat energy is left for self-heating. In this case, the response curve is approximately linear. When the activation current is in the range of 5 to 17mA, the applied shear flow can not convect away all of the input heat energy, thus the remaining heat energy is stored in the CNTs for self-heating, and consequently, the resistance decrease for the CNTs with a negative TCR. And, when the current is higher than 20mA, the flow convects the thermal energy away from the CNTs at a higher rate, which causes the sensor’s resistance to increase greatly in a shorter time. However, since the input energy is also increased rapidly in this case, the sensor’s resistance shows an immediately exponential decrease. As a result, we presently activate our sensors at a current smaller than 1mA to avoid the current-induced self-heating. Considering the balance between the sensor working temperature and sensitivity, 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. Under this operating current, the surface temperature of CNTs was ∼63.2°C above the ambient temperature.

3) Shear Stress Sensitivity

The dynamic sensing response of EG-CNT sensors were measured at room temperature upon exposure to DI-water flow with different flow rate ranging from 0.3 to 3.4ms⁻¹. Fig. 11 shows the change of resistance of EG-CNT sensors under different flow velocities. We calculated the sensor response of
\[ \Delta R / R_0 \] 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. 12(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. 12(b)). This result is consistent to our previous theoretical prediction, and from the experimental data, the coefficient that relates shear stress to CNT resistance change ranges from 6.5 to 8.3 for the EG-CNT sensors (from Eq. (9)).

A comparison of EG-CNT sensors with the MEMS shear stress sensors is summarized in Table 1, where \( O \) indicates the order of magnitude. It is clear that EG-CNT sensors have the advantages of small dimensions, low power consumption, and easy fabrication process. However, the EG-CNT shear stress sensors have understandably lower responsivity than polysilicon sensors due to much lower input power. It is interesting to note that (from Table 1) with \( \sim \)1000 times reduction in input power the EG-CNT sensor only suffer \( \sim \)10 times reduction in responsivity.

V. CONCLUSION

In this study, aqueous shear stress sensors that utilize EG-CNTs as sensing elements are integrated in PDMS microfluidic systems using DEP nano-manipulation technique and a MEMS-compatible fabrication process. The electrical and thermal properties of EG-CNTs were measured. Shear stress responsivity of the sensors has been characterized in fully developed laminar DI-water flows inside PDMS microfluidic systems. Upon exposure to DI-water flow, the electrical resistance of the EG-CNTs was found to increase. Furthermore, we have demonstrated an obvious linear relationship between the EG-CNT resistance change and the flow rate to the one-third power. This experimental result and the theoretical prediction based on the thermal transfer principle are in close agreement. Moreover, the reversibility of the electrical property of CNT sensors indicates that the EG-CNTs show a very stable connection to the Au electrodes without any specific protection process. Our results proved the feasibility of using CNTs as aqueous shear stress sensors with ultra-low-power consumption (\( \sim 1 \) to 2\( \mu \)W). Compared with other MEMS-based shear stress sensors, our CNT shear stress sensors possess the unique advantages of ultra low power consumption, low operation temperature, and minimized size. Hence, EG-CNT sensors are promising devices for flow rate, shear force and biomedical sensing applications in micro and nano scales. In order to implement better functions of CNT shear stress sensors, their selectivity based on molecular functional groups, the responsivity for different microfluidic flow media, and reproducibility will be investigated by our group in the future.

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\begin{figure}[h]
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\includegraphics[width=\textwidth]{fig11.png}
\caption{The change of resistance of EG-CNT sensors with DI-water flow of different velocity.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig12.png}
\caption{(a) Output resistance change vs. flow velocity; (b) Output resistance change vs. velocity to the one-third power.}
\end{figure}

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**TABLE I  COMPARISON OF MEMS SHEAR STRESS SENSORS**

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<tr>
<td>Measurement method</td>
<td>Direct</td>
<td>Indirect</td>
<td>Indirect</td>
</tr>
<tr>
<td>Sensing material</td>
<td>Polyimide/Aluminum/Piezoresistive/Silicon</td>
<td>Polysilicon/Platinum</td>
<td>CNTs</td>
</tr>
<tr>
<td>Dimensions of sensing element</td>
<td>L–W–O(100μm)</td>
<td>L–O(1μm)</td>
<td>L–O(1μm)</td>
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<tr>
<td></td>
<td>T–O(1μm)</td>
<td>W–O(1μm)</td>
<td>W–O(1μm)</td>
</tr>
<tr>
<td>Power consumption</td>
<td>×</td>
<td>O(1mW)</td>
<td>~1-2μW</td>
</tr>
<tr>
<td>Complexity of fabrication process</td>
<td>The most complicated</td>
<td>Complicated</td>
<td>Simple</td>
</tr>
<tr>
<td>Responsivity</td>
<td>~0.4kV/Pa</td>
<td>~0.74mV/kPa</td>
<td>~0.06mV/kPa</td>
</tr>
<tr>
<td>Reported application</td>
<td>Gas flow</td>
<td>Gas flow</td>
<td>Gas/liquid flow</td>
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**REFERENCES**


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