Optically Transparent Microfluidic Systems Integrated with Carbon Nanotube Sensors

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***Abstract*-In this paper, we present carbon nanotube (CNT) based thermal shear stress sensors integrated inside optically transparent Polymethylmethacrylate (PMMA) microfluidic systems. The sensors were fabricated on PMMA substrates by batch assembling multi-walled carbon nanotubes (MWNTs) as sensing elements between microelectrode pairs using AC dielectrophoretic (DEP) technique. PMMA chambers were fabricated using SU-8 molding/hot-embossing technique. Then, the PMMA substrate with a micro chamber and vortex micropump was bonded to the other PMMA substrate embedded with the MWNT sensor array to form a closed flow chamber. Experiments showed that the CNT sensors could detect volumetric air flow rate in the order of $10^{-8}m^3/s$ inside this microchannel system. We have also proved that upon exposure to constant liquid (DI-water) flow, the electrical resistance of the CNT sensor was found to increase linearly at low activation current of 100μA. And a linear relation between the change of output resistance and one-third power of flow rate was observed for flow rate from 0.3 to 2.3m/s. This result proved that the CNT sensors work with the same principle as conventional MEMS based thermal shear stress sensors, but only require ultra-low activation power (~μW) to achieve comparable sensitivity, which is three orders of magnitude lower than conventional MEMS polysilicon based flow sensors.**

I. INTRODUCTION

Accurate measurement of time-resolved wall shear stress in flows plays an important role in a broad application spectrum that ranges from fundamental scientific research to industrial process control and biomedical applications. In particular, the measurement of flowrate 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 [1].

Wall shear stress sensors are categorized by measurement methods into two distinct groups — direct and indirect techniques. MEMS shear stress sensors based on these two principles were already demonstrated by [2, 3]. The former directly measure the shear force acting on a solid surface. This is typically achieved by employing a "floating element" balance, which is attached to either a displacement transducer or is part of a feedback force-rebalance configuration. Therefore, the fabrication process of these sensors is extremely complicated, inefficient, and time consuming. Meanwhile, the sensors suffer from complex configuration, unacceptably large chip size (several millimeters on a side), and unstable mechanical properties due to environmental condition (e.g., moisture) variations [4-6].

On the other hand, indirect techniques require an empirical or theoretical correlation, typically valid for very specific conditions, to relate the measured property to the wall shear stress. The MEMS community has produced a variety of indirect transduction schemes such as hot-film sensors and micro-optical systems to measure near-wall velocity gradients. Among them is the thermal shear stress sensor which measures flow-imposed surface shear stress based on the amount of convective heat transfer from a heated polysilicon sensing element to the surrounding fluid flow. A MEMS thermal shear stress sensor is typically 200μm×200μm in size with polysilicon sensing element of approximately 150μm long, 3μm wide and 0.5μm thick. The large length-to-width ratio is necessary to ensure sensitivity preference in the direction normal to the length. Also, to minimize conduction heat loss to the substrate, a vacuum cavity is usually included underneath the sensing element. The MEMS thermal shear stress sensors have been proven to be effective in both air [7, 8] and aqueous flows [9]. 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.

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 stimulated the utilization of CNTs as a novel sensing material for pressure, thermal, gas, and flow sensors [10]. Therefore, in the past few years, CNTs have drawn attention from world-wide researchers to investigate the possibility of using them as micro shear force/flowrate sensors. Voltage in the order of mV had been observed to be generated by an aqueous flow over the single-walled carbon nanotube (SWNT) bundles [11] and multi-walled carbon nanotube (MWNT) thin film [12]. These work showed the potential for nanotubes as sensitive flow sensors. And, a vertically oriented MWNTs based fluid flow/shear shear sensor was developed to determine the shear force of fluid flow by monitoring the polarization and intensity of the transmitted light through the MWNT mat [13].

In earlier publications we have presented the utilization of CNTs for pressure and thermal measurements [14-16]. In this paper, we will demonstrate our latest results of integrating CNT sensors inside optically transparent microfluidic systems for shear stress detection.

II. CNT SENSOR FABRICAITON

Fig. 1 shows the fabrication process of CNT shear stress sensor chips. First, the microelectrode array was fabricated by standard sputtering, lithography, and wet chemical etching process. Parylene C was used to strengthen the adhesion of Au microelectrodes to the PMMA substrate. Then, MWNTs were batch assembled between the microelectrode pair to serve as sensing element by using the AC dielectrophoretic (DEP) manipulation technique reported in [17]. Fig. 2 is the SEM image of MWNT bundles between Au microelectrodes. After that, another layer of Parylene C was deposited to fix the MWNTs onto the electrodes. Meanwhile, a PMMA chamber was fabricated using a customized SU-8 molding/hot-embossing process. The chamber is 7mm long with a cross-sectional area of 4mm × 300μm. PMMA was chosen because it is optically transparent, electrically and thermally insulating, biocompatible, and low-cost. The details of the vortex micropump fabrication process can be found in our previous paper [18]. Finally, the PMMA substrate with a micro chamber and vortex miropump was bonded to the other PMMA substrate embedded with MWNT sensors array to form a closed flow chamber by using standard PMMA-PMMA bonding process. A prototype MWNT sensor chip is shown in Fig. 3.

III. TESTING AND DISCUSSION

The I-V characteristics of the CNT sensors were measured at room temperature. The tested sensor has a room temperature resistance of 150kΩ. A typical I-V curve of the CNT sensors is shown in Fig. 4. The results show that MWNTs exhibited an obviously nonlinear I-V property above an input current of ~2μA. This nonlinearity of the I-V curve proved that the MWNTs experienced a pronounced temperature rise due to Joule heating. And the current required to induce the heating effect of bulk MWNTs is in the order of μA, which implies the CNT sensors could be used as thermal flow sensors with as little as a few μW of

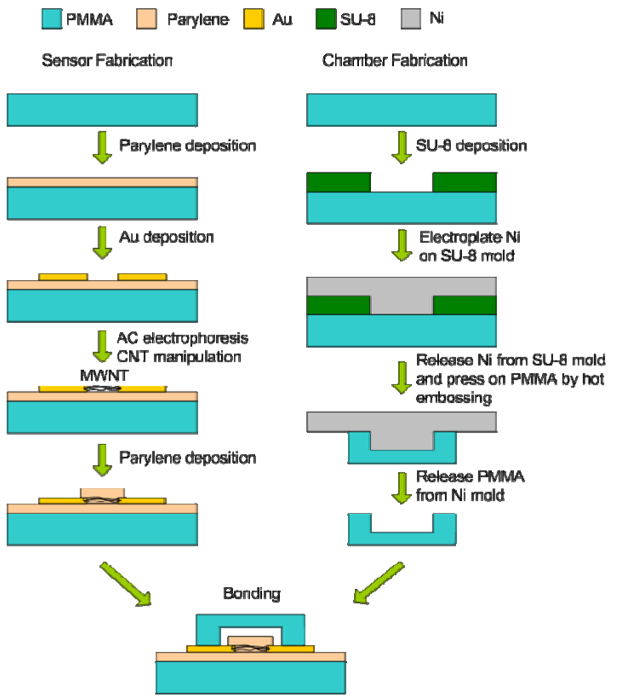

Fig. 1. Simplified fabrication process for CNT based shear stress sensors integrated in PMMA microfluidic systems.

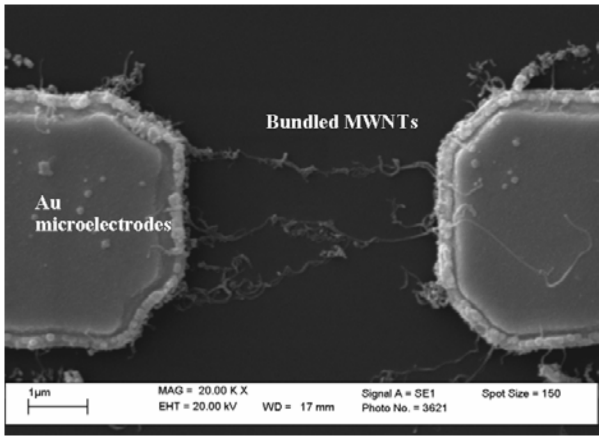

Fig. 2. SEM image of MWNT bundles (~5μm in length) between Au microelectrode pair (gap=5μm) after DEP manipulation.

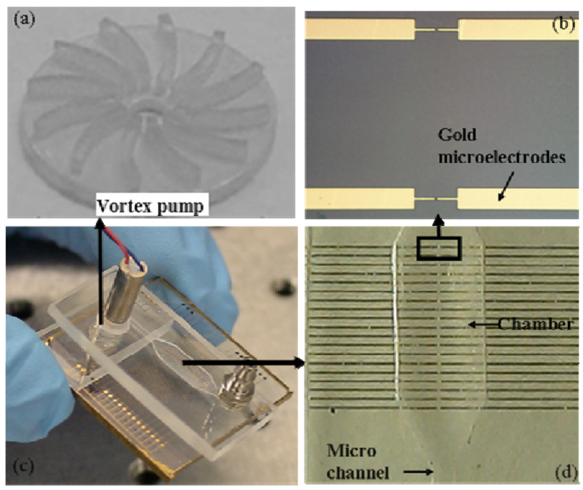

Fig. 3. (a) Photography of a vortex pump; (b) optical microscope image of two pairs of microelectrodes with CNT bundles; (c) photograph of a shear stress sensor chip; (d) optical microscope image of an array of MWNT sensors embedded inside a PMMA microfluidic system.

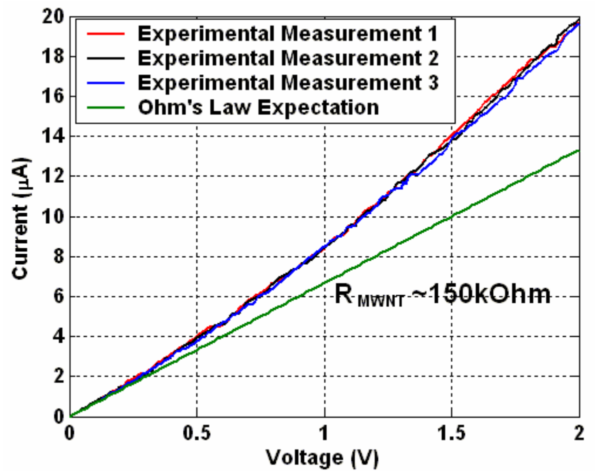

Fig. 4. Typical I-V characteristics of a MWNT sensor with a room temperature resistance of 150kΩ.

input power.

Fig. 5 shows the integrated experimental setup for flow detection. A vortex micropump connected to the vortex micropump was used to control the flow rate and direct the flow into the CNT sensor chip by changing the power supply of the DC motor. While a constant current mode circuit was used to generate the operating current to activate the sensor and also to provide the measured voltage values to the computer via an ADC interface. Prior to each measurement, the sensor was activated and saturated with the flow medium for ~20mins to eliminate the influence of sudden change of flow impact. After each measurement, adequate time delay (~30mins) was allowed for the resistance to recover to its original state.

A. Air flow sensitivity

Experiments were performed to validate the air flow sensing ability of the MWNT sensor array in the flow chamber at room temperature. As shown in Fig. 6, the CNT sensors could detect volumetric air flow rate in the order of 10^{-8} m^3/s inside the microfluidic system. When the air flow rate exceeded $5\times10^{-8}m^3/s$, there was a linear relation between the output voltage and the flow rate. While under $5\times10^{-8}m^3/s$, the sensor showed little responsivity.

B. Aqueous flow sensitivity

We also conducted aqueous flow tests on the CNT sensors. Under a constant activation current, the output resistance responded linearly and stably for a total volume of 2.5ml of DI-water to a constant flow introcution of $1.3ms^{-1}$ inside the microchannel as shown in Fig. 7. The CNT resistance increased ~ 10.8% over 110 seconds due to flow introduction. After the flow was stopped, we observed a time-dependent recovery of the CNT resistance. Note that the CNTs used in this experiments were electronics grade CNTs (EG-CNTs) provide by Brewer Science Inc., Missouri.

Then, the dynamic sensing response of CNT sensors was measured upon exposure to DI-water flow with different flow rate ranging from 0.3 to 3.4m/s. Fig. 8 shows the change of resistance of CNT sensors under different flow velocities. When the velocity was larger than 2.3m/s, the CNT sensors showed very little responsivity. However, it is

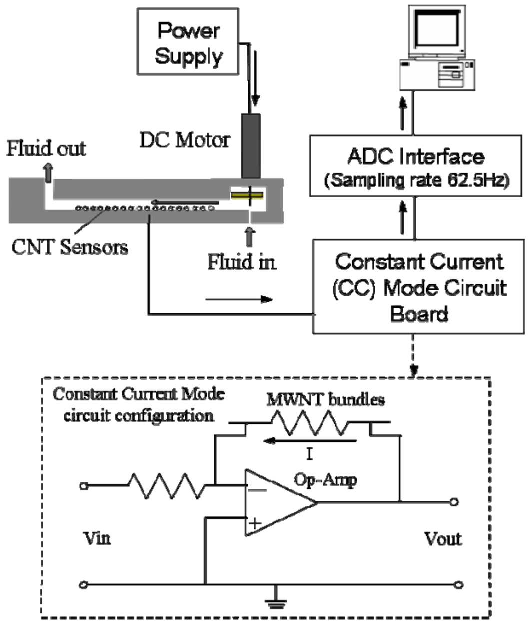

Fig. 5. Schematic diagram of the experimental setup for fluid flow test in the integrated microchannel and micropump system. The CNT sensors are placed parallel to the direction of the fluidic flow.

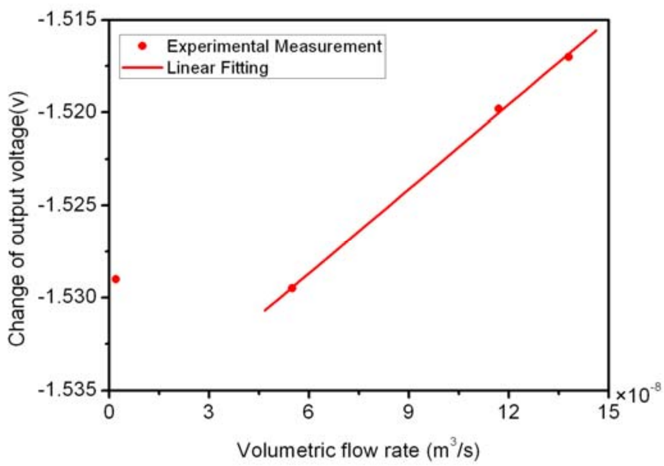

Fig. 6. The change of voltage with different air flowrate generated by the vortex micropump.

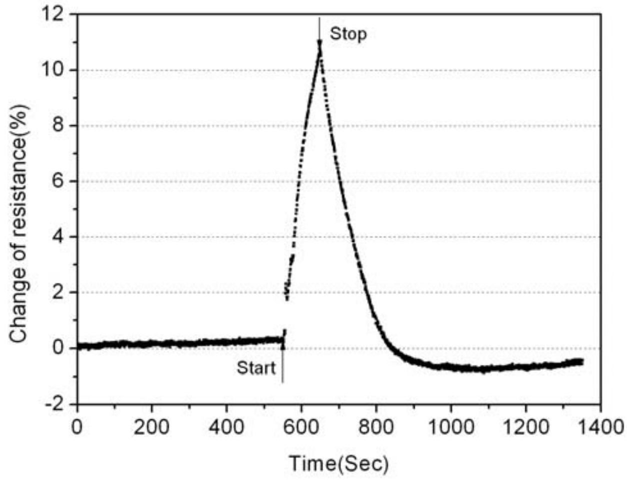

Fig. 7. Typical output resistance change with the introduction of DI-water flow of 1.3m/s under CC activation mode.

evident that the change of resistance can be plotted as a linear function of the flow velocity to the one-third power

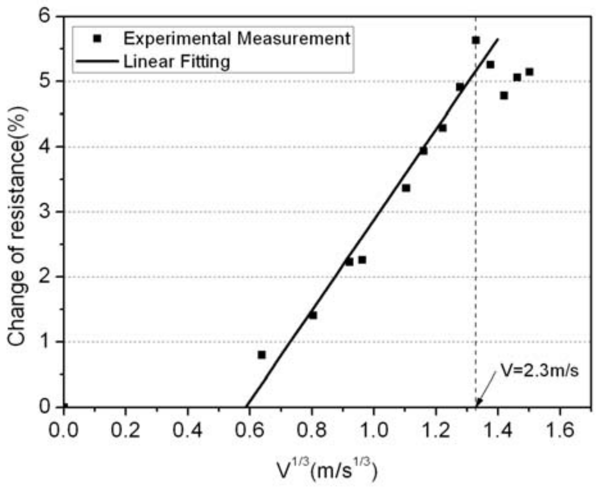

Fig. 7 Output resistance change vs. flow velocity to the one-third power.

when velocity was under 2.3m/s. This result indicates that our sensors work with the same principle as conventional MEMS based thermal shear stress sensors, but with much less (~1000 times less) power input to achieve comparable responsivity.

Ⅳ. CONDLUSION

In this study, we have demonstrated MWNT sensors integrated inside PMMA optical transparent mcirofluidic systems. The fabrication process was developed by combining a MEMS-compatible fabrication process with DEP nano-manipulation technique. The nonlinearity of I-V curve of MWNTs indicates the heating effect and the current required to induce this nonlinear behavior is in the order of μA, which implies the CNT sensors could be used as thermal flow sensors with as little as a few μW input power. Upon exposure to both air flow and DI-water flow, experiments were performed to validate the shear force sensing ability of the CNT sensors. The results showed that the CNT sensors could detect volumetric air flow rate in the order of 10^{-8} m^3/s inside this microchannel system. And a linear relation was observed between the output resistance change and the one-third power of DI-water flow rate ranging from 0.3 to 2.3m/s. Our results revealed that the CNT sensors can be integrated in PMMA microfluidic systems and operated as thermal shear stress sensors in μW range, a power range that is three orders of magnitude lower than conventional MEMS polysilicon based flow sensors.

Ⅴ. AKNOWLEDGEMENT

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