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An electric impedance based microelectromechanical system flow sensor for ionic solutions

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Abstract

Microfluidic devices with channel cross sections measuring $4 \times 10 \mu\text{m}^2$ instrumented with gold microelectrodes were used to sense flow rates of ionic solutions on the basis of electric impedance (EI) measured perpendicular to the flow. Negative pressures were applied to access ports of the microdevices to generate flow of saline solutions (physiologic concentrations 0.9%) through the micro-EI recording zone with flow rates between 2.4 and $4.8 \mu\text{l min}^{-1}$. The EI spectra (100 Hz–20 MHz) recorded under flow conditions were compared with the no-flow condition. Changes in the magnitude of EI (at 350 Hz) for flow rates as low as $2.4 \mu\text{l min}^{-1}$ were statistically significant compared with the no-flow condition. The observed dependence of EI on flow rate is attributed to the relative difference between the rate of migration of charge-balancing electrolyte ions to the electrode surface and the rate of removal of the same ions by forced convection. An electrochemical convection–diffusion model was used to study the observed dependence on flow. Simulations support the conceptual model that passing DC current from the gold electrodes into the ionic solution results in an increase in ionic concentration near the electrode surface (due to the inward migration of counter-balancing ions). When the fluid flow rates increase, these counter-balancing ions are replaced by the bulk solution, thereby lowering the average ionic concentration within the recording zone. This local concentration drop results in an increase in the real part of the impedance.

Keywords

microfluidics; MEMS; flow; sensor; ionic solutions; microflow; electric-impedance; microelectrode; electrochemistry; electrochemical; diffusion layer; conductivity; electrolyte

1. Introduction

Microfluidic systems are beginning to provide a platform for new technologies with a wide range of applications in engineering and life science. Measuring and controlling flow rates for these microsystems is often a basic requirement for device function and performance. Recent efforts have been made to integrate conventional flow sensors into microelectromechanical system (MEMS) devices. Numerous groups are attempting to miniaturize thermal, anemometer style liquid flow meters by integrating them into microchannels (Wu *et al* 2000, Rasmussen and Zaghloul 1999, Lyons *et al* 1998, Ashauer *et al* 1999). Other attempts to create on-chip flow sensors include static turbine concepts that convert fluid flow into torque (Svedin *et al* 2001), differential pressure sensors using piezoresistive elements (Richter *et al* 1999), estimates of flow using drag forces exerted by the fluid flowing over a cantilever (Gass *et al* 1993) and a mass flow sensor based on the Coriolis force principle (Enoksson *et al* 1996).

Despite these promising developments, commercially available microfluidic systems still use conventional, off-chip sensors and positive displacement pumps to control the fluids. This work describes the initial efforts towards developing a simple, low-power, on-chip sensor capable of measuring flow rates of ionic solutions using EI as the measured parameter.

Previous investigations into the electrochemical behaviour of microelectrodes in solutions of low ionic strength (i.e. solutions in which the concentration of supporting electrolyte is significantly lower than the concentration of the redox-active species) have demonstrated increases in net ionic concentration close to the electrode surface (Amatore *et al* 1988, Drew and Wightman 1991, Gao and White 1995). This increase in near-surface electrolyte conductivity is the result of the oxidized species creating a depletion layer surrounding the microelectrode. The electrical charge associated with the newly formed cation is balanced by a local increase and decrease in the concentrations of the electrolyte anions and cations respectively. The migration of counter-balancing ions to the electrode surface results in an electrolyte conductivity that is lower than the bulk conductivity. Rotating microdisc electrochemical experiments conducted in solutions with low ionic concentrations demonstrated a decrease in voltametric currents with increasing angular disc rotation (Gao and White 1995). Gao and White's data suggest that the build-up of near-surface ionic concentration is washed out by the presence of fluid flowing over the surface of the electrode as the speed of the disc increases. This ionic build-up/washout phenomenon is the basis for a new surface-mounted EI based flow sensor described herein.

The microflow sensors used for this work were constructed on glass wafers and consisted of fluid channels having cross-sectional dimensions of $4 \times 10 \mu\text{m}^2$ (Ayiliffe *et al* 1999). Pairs of gold microelectrodes were integrated into the sidewalls of the microchannel and used to interrogate EI spectra (100 Hz–20 MHz) during flow and no-flow conditions. Since our primary interest was in bio-MEMS applications, the fluid consisted of a physiologic saline solution (0.9% NaCl). EI spectra were also collected in the presence of a DC bias to increase device sensitivity and investigate the electrochemical ionic build up/washout phenomenon. Results suggest that the system is capable of measuring flow in the low microlitre per minute range (2.4 and $4.8 \mu\text{l min}^{-1}$) at relatively low interrogation frequencies (350 Hz) superimposed on a 50 mV DC bias.

To better understand the electrochemistry and physics involved in the EI based microflow sensor, a two-dimensional, electrochemical convection–diffusion model was developed. The model supports the hypothesis that the dependence of EI on flow was due to local changes in net ionic concentration. At the low voltages used, below the half-cell potential for AgCl (on the order of 500 mV), oxidized or reduced solution trace elements are the most likely source of charged molecules to drive the migration of counterbalancing ions to the electrode surface. Applying a DC bias to the electrodes is believed to cause the reaction to occur at a new or different location on the cyclic voltametric curve (DeRosa and Beard 1977, Ragheb and Geddes 1991, Schwan 1967). Shifting the relative reaction potential is thought to correspond to a change in device sensitivity by altering the concentration and/or species involved in the oxidation/reduction processes. The model supports the hypothesis that when fluid flow is increased, the solution with the higher ionic concentration is partially washed out and replaced by the bulk solution (having a lower average ionic concentration). This exchange results in an increase in the real part of the impedance with increased flow rate. The ability to simply measure flow using the EI of ionic solutions (on-chip) may prove to be a valuable addition to future microfluidic systems such as microflow cytometers and electrophoresis detectors.

2. Methods

2.1. Experimental methods

Gold-electrode instrumented microfluidic channels were fabricated on glass wafers following Ayliffe *et al* (1999). Microdevices were cleaned prior to each recording by flushing the microchannels with a 10:1 solution of 10 molar HCl in DI water for 5 min. The HCl solution was replaced by DI water and vigorously flushed through the microchannels five times. During the precleaning, each device was positioned under an inverted Nikon confocal microscope (Diaphot 200, Melville, NY) to assure complete filling and flushing of the recording zone.

The glass wafers containing the microdevices were visually aligned and securely fixed to the stage of the inverted light microscope. Electrical connection was made from the gold contact pads on the microdevice to the computer-coupled (GPIB, IEEE 488, National Instruments) network analyser (HP4194A, Hewlett Packard) via a 1 m long extension probe (HP16433A, Hewlett Packard). The extension probe was positioned and held in contact with the microdevice gold contact pads using a micromanipulator (Narishige, Japan). The network analyser was appropriately calibrated just prior to making the electrical connection with the extension probe in place. This initial calibration compensates for the measurement lead wires such that the data include only the combined characteristics of the microdevice and the material in the recording zone. The raw data measured between the device contact pads are termed the 'device EI' for simplicity.

Flow EI measurements began by adding a 10 μ l aliquot of saline to the source reservoir of a clean and dry microdevice. Slight negative pressure was initially applied to the sink reservoir to pull the solution completely into the microchannel. Complete filling of the device-recording zone was confirmed by visual inspection. A timer was started and a 'no-flow' EI measurement was recorded from 100 Hz to 15 MHz. EI data were downloaded to a personal computer (GPIB, IEEE 488, National Instruments; Igor Pro, Wave Metrics; Apple Macintosh). A custom pressure system was used for all of the flow experiments to apply suction to the sink reservoir. The system consisted of a calibrated syringe, a water-manometer and flexible tubing to connect to the microdevice. To begin the flow of solution in the microchannel, the syringe was withdrawn to a known volume and the syringe stopcock was closed. The manometer was monitored during the flow measurements to ensure that the applied pressure remained constant. Once the pressure was applied, the EI spectrum was recorded. The negative pressure was then released by opening the system to atmosphere and a second EI spectrum in the no-flow condition was recorded. The flow/no-flow records were recorded repetitively using this technique about seven times over a time course of 15 min. Three sets of experiments were conducted for all of the different conditions tested.

The flow experiments were designed to characterize the fundamental relationship between device EI and the bulk flow of an ionic solution. One series of experiments was conducted, on three different microdevices, to examine correlates between device EI and flow rate. All of these experiments used a physiologic concentration of saline (0.9%) as the reference solution. A second set of experiments was performed to investigate the effects of a DC bias added to the 500 mV sinusoidal AC interrogation signal. For these experiments, the same 0.9% saline solution was used and EI spectra were recorded at the highest flow rate. Finally, the effects of different saline concentrations in flow-dependent EI were quantified. In these studies, the EI of 0.45, 0.9 and 1.8% saline concentrations was measured at various flow rates.

Following each saline flow experiment, the microdevices were flushed with deionized (DI) water and allowed to dry in air. A series of seven experiments were then conducted to relate the volume of air displaced in the syringe to the flow rate through the microchannel. A 10 μ l aliquot of DI water was added to the source reservoir of each microdevice tested and negative

pressure was applied to the sink reservoir using a known volume in the syringe and applied pressure. The time between the application of suction and the draining of the microchannel was recorded. This time was determined by visually inspecting the EI recording zone to determine when the last of the DI water ran through the channel. The average flow rate and standard deviation for each flow condition was calculated using the seven measured times and known sample volumes.

2.2. Modelling methods

The flux $\vec{J}_n$ (molecules $\text{cm}^{-2} \text{s}^{-1}$) of each ionic species n in the microchannel was modelled using classical electrochemical diffusion of the form

$$\vec{J}_n = -D_n \left(\vec{\nabla} C_n - \frac{z_n q}{kT} C_n \vec{E} \right),$$

where D is the diffusion coefficient, C is the molecular concentration, z is the apparent valence, q is the unit charge, k is the Boltzmann constant, T is the absolute temperature and $\vec{E}$ is the electric field vector. Conservation of ions within the non-reacting solution requires for each ionic species n

$$\frac{\partial C_n}{\partial t} + \vec{V} \cdot \vec{\nabla} C_n = \vec{\nabla} \cdot \vec{J}_n,$$

where $\vec{V}$ is the fluid velocity vector and t is time. The electric current density I arising from redox reactions at the metal electrodes was modelled as an injection of an ionic species i just outside the double layer. For each electrode

$$I = z_i q (\vec{J}_i \cdot \hat{n}),$$

where $\hat{n}$ is the outward unit normal from the electrode surface. The flux was set to zero at all non-reacting surfaces and concentrations were set to the initial background levels at the two extreme ends of the channel. Electrostatics and ideal space charge neutrality were assumed. Accordingly the divergence of the electric field was set to zero

$$\vec{\nabla} \cdot \sigma \vec{E} = 0,$$

where σ is the conductivity tensor taken here to be a constant in space. Simulations were carried out by lumping all anions into one group ' $n = 1$ ' and all cations into a second group ' $n = 2$ '. The fluid velocity was assumed to be fully developed laminar flow with a parabolic profile. Numerical solutions were obtained by successive over-relaxation for the steady case (partial time derivatives were set to zero).

3. Experimental results

The photograph in figure 1(A) depicts the complete experimental set-up used for all of the characterization studies. The inverted light microscope, Hewlett-Packard computer controlled network analyser and video-imaging equipment can be seen in the figure. In figure 1(B), a

glass wafer containing several micro-EI devices is seen on the stage of the inverted light microscope with the probe from the network analyser being held in position with a micromanipulator.

The magnitude and phase of the raw EI data from one of the flow experiments is shown in figure 2. The thick solid curve is the average ($n = 7$) for the no-flow condition while the average ($n = 7$) for the flow condition is the thick dashed curve. Standard deviations were calculated at 12 frequencies and are shown as error bars in the figure. The differences in the magnitude between the two flow conditions was statistically significant at all frequencies below 10 kHz (Student's t -test, 100 Hz, 350 Hz, 1 kHz). The maximum difference in the magnitude for the majority the flow experiments occurred at ~ 350 Hz. The effects of flow on the measured EI phase were found to be statistically significant at frequencies as high as 100 kHz.

The magnitude and phase data at 350 Hz for the three flow rates tested (2.40 ± 0.12 , 3.49 ± 0.13 , and $4.77 \pm 0.15 \mu\text{l min}^{-1}$) are shown in figure 3. The differences in EI magnitude between the flow and no-flow conditions were statistically significant at all three flow rates (Student's t -test, $\alpha < 0.05$). No statistically significant differences were found between the device EI phase at this low frequency.

To normalize the average EI data for the three flow rates tested in one device, the complex-valued impedance recorded in the flow condition was divided by the corresponding no-flow impedance. The relative magnitude and phase are presented in figure 4. The dependence of EI on solution flow rate is demonstrated to occur primarily at lower frequencies. As the flow rate increases, the magnitude of impedance increases and the difference in phase increases. In addition, the higher flow rates result in an upward shift in the maximum difference in phase. Both the magnitude and phase effects of flow can be seen at higher frequencies as the flow rate increases. Above 200 kHz, however, the spectra are nearly devoid of any effects of bulk flow.

All of the EI measurements were conducted with the network analyser set to a constant-voltage mode and linear analysis were used to calculate the impedance. Interrogation waveforms were not recorded and therefore results cannot address any nonlinearities and/or signal rectification. It is quite possible that some rectification and DC offset did occur. Rectification would be expected to increase the concentration of oxidized or reduced species and drive the migration of counter-balancing ions, thereby increasing the net ionic concentration in the recording zone (Amatore *et al* 1988, Drew and Wightman 1991, Oldham 1988, Gao and White 1995, Ayiliffe 1999). Bulk flow through the recording zone could potentially wash out this solution and replace it with fresh solution from the fluid reservoir, which has a lower net ionic concentration (Gao and White 1995). To further investigate this hypothesis, the experiments were repeated in the presence of a DC bias current.

The same 15 min flow/no-flow protocol described previously was used. EI spectra of the device filled with 0.9% saline solution were measured using both a 10 and 50 mV DC bias applied to the 500 mV AC signal. Based on the $8 \mu\text{m}$ electrode spacing, this bias generated electric field strengths of ~ 1.25 and 6.25 kV m^{-1} respectively. Differences between the flow ($2.48 \pm 0.06 \mu\text{l min}^{-1}$) and no-flow EI data for the magnitude and phase at both 10 and 50 mV were statistically different at 350 Hz (Student's t -test, $\alpha < 0.05$). Figure 5 shows the averages of the raw bias data ($n = 7$) and the standard deviation at 350 Hz.

The data for the 10 and 50 mV bias flow experiments were normalized by dividing the complex-valued flow impedance by the no-flow impedance. The relative magnitude and phase data are shown in figure 6. Superimposing a DC bias on the interrogation signal increased the sensitivity of the device to fluid flow at low frequencies (< 10 kHz). No correlation between EI and DC bias was found at higher frequencies. These data are consistent with the hypothesis that a DC

bias results in a net increase in local ionic concentration and that convective washout within the recording zone results in a change in resistive component of the impedance.

4. Model predictions

A two-dimensional electrochemical convection–diffusion model was used to investigate the basis of the dependence of EI on flow rate. The equations are quasistatic and are provided in the methods section. In the model, charged redox reaction products generated at the electrode surface sequester counterbalancing ions from the bulk solution to form a cloud of anions and cations near each electrode. Figure 7 shows the voltage (top panel) and concentration (second panel) predicted by the model for the no-flow condition. The cloud, induced by DC current, increases the conductance of the solution between the electrodes. As fluid flow rate increases from zero (figure 7, second to fifth panels) the ion cloud is washed out and replaced with ionic solution having a lower concentration. This results in a positive correlation between the real part of the impedance and flow rate. Predictions of this relatively simple 2D model for changes in the real part of the impedance due to convection of the ion cloud are consistent with the data.

5. Discussion

Results demonstrate statistically significant changes in device EI for the flow versus no-flow conditions of physiological saline (0.9%) at rates as low as $2.4 \mu\text{l min}^{-1}$. The greatest sensitivity to changes in the flow rate in the microchannel occurred at 350 Hz (figure 4). At this frequency, the relative impedance during flow was ~ 1.5 times higher than recorded in the static no-flow condition. In general, the magnitude of impedance was found to increase as a function of flow rate. In addition, the dependence of EI on flow rate extended to higher frequencies as the flow rate increased.

The data and model point to a significant electrochemical effect that alters the ionic strength within the microdomain of the electrodes and underlies the dependence of EI on flow rate. Since all of the experiments used voltages below the half-cell potentials for Au, solution trace elements such as dissolved oxygen are likely to be species being oxidized and reduced. The addition of a DC bias across an Au interface is known to result in a shift in the cyclic voltametric curve and an increase in rate of oxidation and reduction of ions (Ragheb and Geddes 1991, Gao and White 1995). The oxidation (or reduction) of ions at the electrode surface would be expected to generate a depletion layer which is charge-balanced by the migration of anions and cations from the bulk solution. For oxidation, the charge compensation would occur primarily by migration of Cl^- toward the electrode (reduction via Na^+ migration toward the opposite electrode). Due to the confined electrode/microchannel geometry, the absence of flow results in a build-up of ionic concentration surrounding the recording zone (during EI interrogation) and a corresponding increase in overall conductance. The computer model depicts this steady-state condition for no flow in figure 7, second panel. As the flow rates increase, the bulk solution, having a lower ionic concentration, begins to wash out the solution within the recording zone. As the model predicts, an increase in flow rate corresponds to a decrease in solution conductance (i.e. an increase in the real part of the measured device impedance).

Both the data and the model indicate that direct DC bias or asymmetric rectification occurring at the two electrodes (thus generating a DC component during pure AC sinusoidal interrogation) resulted in an increase in local ionic concentration, leading to EI sensitivity to changes in flow rates. Comparison of DC bias data to AC alone indicates that a rectified DC signal having a magnitude near 5% of the AC rms signal was present. This rectification may account for differences in flow sensitivity observed for AC interrogation signals in different devices—differences owing to relatively small changes in electrode geometry and symmetry (i.e. two electrodes are not perfectly identical).

The observed increases in the real part of the EI of the measuring device with increasing flow rates is believed to be a phenomenon associated directly with the size of the metal electrodes and the fluid channel (Gao and White 1995, Amatore *et al* 1988, Drew and Wightman 1991, Oldham 1988, Aylyffe 1999). Macroscale studies with rotating disc electrodes show that increasing disc rotation in solutions with low ionic strength correlate with a *decrease* in the real part of measured impedance (i.e. an opposite correlation to microelectrodes) (Pleskov 1976). On a macroscale, hydrodynamic motion facilitates the diffusion of ions into and out of the depletion layer, thereby decreasing the overall impedance near the electrode surface. It is therefore expected that macroscale EI flow sensors for ionic solutions would not display the same correlation with flow and would therefore not function as a flow sensor.

These preliminary data suggest the feasibility of implementing EI based flow sensors into microfluidic devices with biological and/or biochemical applications. Although the constituents and concentration of ions in the vicinity of the electrodes are altered during EI interrogation, the primary change in concentration is due to the sequestering of ions from the bulk solution to the electrode surface. A small fraction of the net effect results from the generation of anions and cations when electrons transfer into or out of solution. In practice, fluid flow could be measured periodically (e.g. every second) using pulses at the frequency with the highest sensitivity (350 Hz). A pulsed EI interrogation would greatly reduce the number of oxidized or reduce impurities created and minimize the ionic redistribution within the bulk solution.

6. Summary

An EI based microfluidic system is reported that has the capability of measuring flow rates of ionic solutions. Physiologic concentrations (0.9%) of saline were forced to flow between pairs of gold electrodes at rates between 2.4 and 4.8 $\mu\text{l min}^{-1}$. EI spectra were recorded at 500 mV between 100 Hz and 20 MHz with and without the addition of a DC bias (up to 50 mV). Maximum sensitivity was at 350 Hz interrogation with a DC bias of 50 mV.

An electrochemical convection–diffusion model was used to study the origin of the effect. The model predicts that passing a DC current between the gold electrodes results in a change (increase) in ionic concentration in the microdomain between the electrodes. This change is due to the inward migration of counter-balancing ions to the electrode surfaces required to maintain space-charge neutrality. When fluid flow rates increase, this higher ionic concentration is washed out and replaced by the bulk solution, thereby lowering the average ionic concentration within the recording zone. This decrease in the net concentration of ions available to carry current between the electrodes results in a change (decrease) in the real part of the impedance. The net affect suggests feasibility that EI could form the basis for on-chip, microscale ionic flow sensors.

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Figure 1.

The experimental set-up. (A) Depicts the complete experimental set-up used for all of the characterization studies. Seen in the figure are the inverted light microscope, Hewlett-Packard computer-controlled network analyser and video-imaging equipment. (B) Depicts a glass wafer containing several micro-EI devices on the stage of the inverted light microscope with the probe from the network analyser being held in position with a micromanipulator.

Figure 2.

The EI magnitude and phase are plotted for 0.9% saline in the no-flow and high-flow conditions. The thin grey curves depict individual EI spectra. The solid thick black curve is the average in the no-flow condition ($n = 7$). The thick dashed curve is the average in the high-flow condition ($n = 7$, $4.77 \pm 0.15 \mu\text{l min}^{-1}$). Error bars depict $\pm$ one standard deviation. EI magnitudes in the flow and no-flow condition are significantly different ($\alpha = 0.05$) at all frequencies less than 10 kHz.

Figure 3.

The EI magnitude (A) and phase (B) while flowing saline (0.9%) through the microchannel at three different flow rates along with controls in the no-flow condition are shown. Values for both bar graphs are taken from figure 2 at 350 Hz. Error bars depict ± 1 standard deviation. Student's *t*-tests demonstrated that the difference in magnitude between the flow and no-flow conditions is statistically significant ($\alpha = 0.05$). Differences in EI phase are not statistically significantly at this frequency (350 Hz).

Figure 4.

The magnitude and phase data for the flow condition after the solution EI spectra were divided by the no-flow spectra. The maximum effect in the magnitude occurs at ~350 Hz. The corresponding phase peaks occur at much higher, and shifted, frequencies. The dependence of flow on the EI spectra is absent at frequencies above 200 kHz. Data are consistent with an increase in the resistance of the saline media associated with convective washout of the Nernst diffusion layer.

Figure 5.

Results from flow experiments measuring the EI of 0.9% saline using two different DC biases. (A) and (B) represent the average ($n = 7$) EI magnitude and phase respectively. Values for both bar graphs were taken at 350 Hz. The same flow rate ($2.8 \pm 0.06 \mu\text{l min}^{-1}$) was used for all of the flow condition data presented in these figures. Error bars represent ± 1 standard deviation. The differences in the data shown here were found to be statistically significantly ($\alpha = 0.05$) using paired Student's t -tests.

Figure 6.

The EI magnitude and phase data for the DC bias experiments after the solution EI spectra were divided by the no-flow EI spectra. The difference between the two curves is the greatest at low frequencies. The dependence on bias is absent from both spectra at frequencies above 30 kHz and complete cancellation of the electrode polarization impedance is accomplished.

Figure 7.

A two-dimensional electro-chemical convection-diffusion model depicting the distribution of voltage (top panel) for zero flow and ionic concentration for flow rates from 0 to 7.8 nl s^{-1} (bottom panels).