A polymer gate FET sensor array for detecting organic vapours

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Abstract

Here we describe a novel ChemFET sensor array that utilises carbon black composite polymers for the detection of organic vapours. Three vapour-sensitive composite polymers have been used as the gate materials of FET devices. A shift in the threshold voltage to toluene is observed with a linear dependence to concentration (Henry's law) and a voltage sensitivity of up to 2.8 \( \mu \text{V/ppm} \). The effects of humidity and temperature have also been examined and the magnitude of the response shows little dependence to humidity but an exponential fall with increasing temperature. We believe that this reduction in response is primarily due to a modification in the bulk solubility of the polymer. The shift in device threshold voltage is may be caused by a change in the work function of the polymer composite, a swelling effect or a charge transfer effect. A generalised linear model is proposed here for the response of a ChemFET to an organic vapour at different humidities. \( \copyright \) 2001 Elsevier Science B.V. All rights reserved.

Keywords: ChemFET; Polymer; Gas sensor; Electronic nose

1. Introduction

The use of FET devices as gas sensors has been of interest to researchers since first reported by Lundström [1]. These sensors utilise catalytic metals, such as palladium, as the gate material and show a high sensitivity to hydrogen and other hydrogen-containing gases [2,3]. These so-called GasFETs normally operate between 150 and 200°C and are fabricated using a non-standard CMOS silicon process. More recent research has concentrated on combining conducting polymers with FET devices. Such conducting polymer materials are attractive due to their ease of deposition, variety of available polymer combinations and ability to operate at room temperature [4,5]. Lately there has interest in carbon black polymer composite materials, since their discovery by Lundberg et al. [6]. These materials consist of conducting carbon black spheres dispersed in an insulating polymer and have been found to be sensitive to a broad range of organic compounds. Present theory, for resistive devices, suggests a swelling effect, in which exposure to the analyte expands the polymer thus increasing the gap between the carbon spheres and increasing the resistance of the polymer composite film [7–9]. Therefore, the combination of conducting polymers with MOSFET technology is attractive for handheld instrumentation.

Research into polymer/FET-based sensors (polFETs) has previously concentrated on electrochemically deposited conducting polymers and techniques based on spin coating [10–15]. Both of these techniques have fashioned sensors with sensitivity to organic compounds, such as ethanol and isopropanol. Here we report on a novel conducting polymer gate FET sensor, based upon carbon black polymer composite films as the gate electrode. We also present the sensors response to two analytes, toluene and ethanol and the sensors’ dependence to humidity and temperature.

2. Device fabrication and polymer deposition

The device comprises an array of four n-type enhanced MOSFET sensors together with a p–n thermodiode, fabricated by the Institute of Microtechnology (IMT at the University of Neuchatel, Switzerland). The MOSFET channel, with typical dimensions of 10 \( \mu \text{m} \times 300 \mu \text{m} \) (length/width), is meandered to reduce the electrical resistance of the polymer gate and to reduce the overall physical size of the device. The NMOS devices are fabricated using a p-well technology [16]. The devices are initially fabricated with a solid gold electrode (Au: 120 nm/Ti: 30 nm) over the channel area covered by a passivation made of a PECVD oxinitride or nitride film. The solid gold gate electrode protects the silicon oxide gate insulator (80 nm) when the passivation film is etched away over the gate area. Then the
gold is either etched away to expose the gate oxide (opened) or left solid (closed). Such a configuration allows the sensing polymer to be deposited over the gate oxide or gold film. A photograph of the cell before polymer deposition is shown in Fig. 1 with two opened and two closed channels. The four MOSFETs are configured with a common gate and drain and are operated in the saturated region by a constant current. A schematic of the connectivity and operation of this device is shown in Fig. 2.

Three different polymer composite films have been deposited, poly(ethylene-co-vinyl acetate), poly(styrene-co-butadiene) and poly(9-vinylcarbazole) each mixed with a 20% carbon black loading by weight. The polymers were sprayed onto the device, by Cyrano Sciences (USA), using a BioDot XYZ Platform (BIODOT, Irvine, USA), which combines a motion control unit with AirJet 2000™ dispenser. The polymer films were deposited at room temperature (21°C) by spraying from a height of 12.4 cm above the device at 10 PSI with toluene as the solvent. This forms a circular covering over the FET devices.

The thickness of the polymer film was measured using a Wyko™ NT-2000 interferometer. This instrument uses optical phase shifting and vertical scanning interferometry to create a three-dimensional surface profile of the polymer. Fig. 3 shows a 2D top view image of poly(ethylene-co-vinyl acetate) deposited onto the FET structure. The polymer thickness in the sensing area has been shown to have only a small deviation (≈10%). These results were verified using a Nanostep 2010 (Rank Taylor-Hobson, UK) that uses a stylus to produce a profile of the film. Typical polymer thicknesses of the three polymers are given in Table 1.

Further to these polFET sensors, a standard resistive sensor was used to benchmark the responses. The resistive device comprised a simple passivated silicon substrate with two gold electrodes, 50 μm apart deposited on top. A coating of poly(styrene-co-butadiene) was deposited over these two electrodes to form the chemoresistor.

3. Experimental set-up

The sensors were placed within an automated flow system, consisting of a PC running National Instruments™ LabVIEW software, solenoid valves and mass flow controllers. This was used to deliver various concentrations of analyte and water in air over the sensors, based on bubbling...
of purified air through a fine sinter in an analyte. A Dreschel vessel, containing the analyte, sits in a re-circulator cooling bath NESLAB RTE-300. By altering the temperature of this cooling bath, the maximum vapour pressure of analyte can be controlled. This carrier gas containing analyte is diluted by a dry/wet air line combination at various flow rates to achieve the required water and analyte concentrations, with a total flow rate of 300 ml/min. Further details are published elsewhere [15].

The sensors were placed in a DRI-BLOCK® (DB-2D) heater for a period of 24 h to equilibrate before testing, and operated at a constant current of 10 μA with the gate/drain (V_{GDS}) voltage monitored. Each sensor was exposed for 60 min at a particular humidity to create a stable baseline, then exposed for a period of 1500 s to a selected analyte followed by 1500 s recovery time at the initial humidity level. The sensors were tested at six different concentrations of toluene and ethanol (605, 847, 1210, 2663, 3995 and 5691 ppm of toluene and 2665, 3732, 5331, 11729, 17593 and 25058 ppm of ethanol) and at four different humidity levels (3000, 4500, 6750 and 9853 ppm of water, typically 10–45% RH and 20°C). Fig. 4 shows a typical response of a the polyFET sensor with a poly(styrene-co-butadiene) coating to toluene at fixed temperature and humidity (37°C/4500 ppm water in air).

### 4. Results

Initial testing at a fixed humidity and fixed temperature showed all three polymers exhibiting a linear change in threshold voltage to toluene concentration, as shown in Fig. 5. The voltage sensitivity coefficients of the polyFETs to toluene was found to be 2.8 μV/ppm for poly(9-vinylcarbazole), 1.7 μV/ppm for poly(styrene-co-butadiene) and −0.48 μV/ppm for poly(ethylene-co-vinyl acetate). Experiments performed using ethanol, showed much smaller responses with sensitivity values of 0.34 μV/ppm poly(styrene-co-butadiene), 0.13 μV/ppm for poly(9-vinylcarbazole) and −0.07 μV/ppm poly(ethylene-co-butadiene) also exhibiting a linear dependence. Typical time responses of the sensors was in the order of 300 s, though showed a diffusion limited response not a reaction limited response.

The effect of humidity on the magnitudes of the response and sensor baseline was also investigated. The effect of humidity on the baseline is shown in Fig. 6 for two of the polymers. Here the V_{GDS} value has been subtracted from an estimated V_{GDS} at a humidity concentration of 0 ppm (estimated values of poly(styrene-co-butadiene) is 0.685 V, poly(ethylene-co-vinyl acetate) is 0.556 V) and the results fitted to a Langmuir isotherm. Typical device sensitivity to water, at 37°C, was measured for poly(styrene-co-butadiene) at −2.0 μV/ppm and for poly(ethylene-co-vinyl acetate) at −3.1 μV/ppm. The baseline of poly(9-vinylcarba-

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Maximum thickness of polymer (μm)</th>
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<tbody>
<tr>
<td>Poly(ethylene-co-vinyl acetate)</td>
<td>2.5 ± 0.20</td>
</tr>
<tr>
<td>Poly(styrene-co-butadiene)</td>
<td>3.7 ± 0.25</td>
</tr>
<tr>
<td>Poly(9-vinylcarbazole)</td>
<td>1.9 ± 0.15</td>
</tr>
</tbody>
</table>

Fig. 4. Typical response of poly(styrene-co-butadiene) FET to toluene to six concentrations at 37°C.
bazole) coated FETs showed no measurable response to humidity on its baseline value during our test. Also measured was the effect of temperature on humidity response, which showed a reduction in the humidity dependence with increasing temperature; typical values of 1.5 μV/ppm poly(ethylene-co-vinyl acetate) and 2.6 μV/ppm for poly(ethylene-co-vinyl acetate) at 67°C were observed. Further to this, all the polymers showed no significant effect of humidity on the magnitude of the response to the analytes. Fig. 7 shows the response of poly(styrene-co-butadiene) at three fixed analyte concentrations with humidity. It was also found that the chemoresistor showed no effect on either its baseline value or the magnitude of its response to the humidity level.

The temperature dependence of the response and of the baseline was measured for the polFET sensors for both the solid gold and polymer gate devices. The gold gate FET showed an exponential increase in baseline value with increasing temperature, while the polymer devices show a significant linear decrease in baseline for all the polymers. Typical voltage sensitivity values for the gold gate were 13.14 μV/°C and for the poly(styrene-co-butadiene) gate −2.8 mV/°C. Fig. 8 shows the effect of temperature on the baseline of a poly(styrene-co-butadiene) and solid gold FET device. Further experiments showed a reduction in the magnitude of the response, at fixed concentration and humidity, with increasing temperature following an expo-

![Fig. 5. Typical response of three polymer FET devices to toluene vapour in air.](image1)

![Fig. 6. Effect of humidity on the baseline of two FET devices at different temperatures.](image2)

![Fig. 7. The effect of humidity on the response of poly(styrene-co-butadiene) coated FET at three different concentrations of toluene vapour.](image3)
5. Analysis and discussion

To analyse the results, it is first useful to examine the theoretical equations that characterise FET device. The standard equation for a standard solid metal gate n-type MOSFET in the saturated regime is

\[ i_{DS} = g_m(V_{GDS} - V_T)^2 = \kappa \]  

(1)

where \( V_{GDS} \) is the gate/drain voltage commoned to source, \( V_T \) the threshold voltage, \( \kappa \) a constant here and \( g_m \) is given by

\[ g_m = \mu C_o \frac{W}{L} = a \]  

(2)

where \( W/L \) are the channel width and length, \( C_o \) the oxide capacitance per unit area, \( \mu \) the carrier mobility and \( a \) is constant in our circuit. A change in \( i_{DS} \) in terms of \( \kappa \) may be written as

\[ \frac{\partial i_{DS}}{\partial \kappa} = \frac{\partial V_T}{\partial C} \frac{\partial g_m}{\partial V_T} \]  

(3)

Since these polFET sensors are operating at constant current and so \( V_{GDS} \) is measured then from Eqs. (1) and (3)

\[ \frac{\partial V_{GDS}}{\partial C} = \frac{\partial V_T}{\partial C} \frac{\partial g_m}{\partial V_T} \]  

(4)

Thus, from this basic equation, we can define the expected effects of changes in concentration, humidity and temperature on changes in \( V_{GDS} \) for the active sensor.

6. Effect of gas concentration on response

From the results section we have seen that the response of the FET sensor, at fixed temperature and humidity follows a linear function. As the parameter \( g_m \) is independent of concentration, then Eq. (4) simply becomes

\[ \frac{dV_{GDS}}{dC} = \frac{dV_T}{dC} \]  

(5)

and so the change in gate/drain voltage equals the change in threshold voltage. Assuming that the threshold voltage is a linear function of \( C \), then

\[ V_T = V_{T0} + kC_P, \quad \Delta V_T = V_T - V_{T0} = kC_P \]  

(6)

where \( C_P \) is the concentration of molecules of analyte in the polymer and equals product partition coefficient and analyte concentration and \( k \) a sensitivity coefficient. Results support

Table 2
Tabulated coefficients for polFET sensors

<table>
<thead>
<tr>
<th>Polymer</th>
<th>( \frac{dV_{GDS}}{dC} )</th>
<th>( \frac{dC}{\Delta V_T} )</th>
<th>( \frac{dV_{GDS}}{dC} )</th>
<th>( \frac{dC}{\Delta V_T} )</th>
<th>( \frac{dV_{GDS}}{dT} )</th>
<th>( \frac{dT}{\Delta V_T} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly(ethylene-co-vinyl acetate)</td>
<td>-0.48</td>
<td>-0.07</td>
<td>-3.1</td>
<td>-3.0 ( \mu )V/( ^\circ )C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Poly(styrene-co-butadiene)</td>
<td>1.70</td>
<td>0.34</td>
<td>-2.0</td>
<td>-2.8 ( \mu )V/( ^\circ )C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Poly(9-vinylcarbazole)</td>
<td>2.8</td>
<td>0.13</td>
<td>0</td>
<td>-1.0 ( \mu )V/( ^\circ )C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
of concentration, we have

\[ \Delta V_f = kC_p = k_pC_A. \]

Previous work with electrochemical deposited polymers [10], indicates that this shift in the threshold voltage is brought about by a modulation in the work function at the polymer and semiconductor interface, through the flat-band potential, i.e. \( \Delta V_f \approx \Delta \phi_{ps}. \) We believe in this case that the change in threshold has two possible further mechanisms. Firstly it could be due to a partial charge transfer from the analyte to the polymer, with the polymer behaving as an acceptor or donor for electrons. Secondly it could be due to a swelling effect of the polymer, that increases the distance between the conducting carbon spheres, hence modulating average work function of the carbon balls and the polymer. This swelling may be thickness-dependant because the layers above the polymer/insulator interface may restrict the movement of the polymer at this interface.

It is probable that the response of the sensors shows a combined work function (electronic) and swelling effect (mechanical). For a swelling effect, the shift in the composition of the carbon (\( \phi_C \)) and the polymer (\( \phi_p \)), hence

\[ \Delta \phi_{PC} = f_c\phi_C + (1 - f_c)\phi_p \]  

Obviously the change in work function due to partial charge transfer is also a function of coverage and by combining the two effects and differentiating, as a function of concentration, we have

\[ \frac{d\Delta \phi_{PC}}{dT} = \frac{df_c}{dT}\phi_C - \frac{df_p}{dT}(1 - f_c)\phi_p + (1 - f_c)\frac{d\phi_p}{dT} \]  

\[ \frac{d\Delta \phi_{PC}}{dT} = \frac{df_c}{dT}(\phi_C - \phi_p) + (1 - f_c)\frac{d\phi_p}{dT} \]  

It should be noted that if \( \phi_p \approx \phi_C \), then Eq. (9) reduces to

\[ \frac{d\Delta \phi_{PC}}{dT} \approx (1 - f_c)\frac{d\phi_p}{dT} \]

where the average shift in simply reduced by a constant of \((1 - f_c)\).

7. Effect of humidity on magnitude of response and baseline

From various humidity tests, we can state with a degree of confidence that poly(ethylene-co-vinyl acetate) and poly(styrene-co-butadiene) are hydrophilic materials and poly(9-vinylacte) is a hydrophobic material. The effect of humidity on the hydrophilic sensors has been shown to follow a Langmuir isotherm, which suggests that there is a limited number of absorption sites for the water molecules to bind. The threshold voltage with humidity at zero or constant analyte concentration, can be written as

\[ \Delta V_T|_{C_A} \approx \frac{2KC_H}{1 + KC_H} \]  

where \( C_H \) is the concentration of water and \( K \) the reaction coefficient and \( z \) a constant. We postulate here the observed response may be due to water molecules forming a dipole layer with the dangling oxygen molecules on the surface of the gate oxide. This adds to the potential already applied to the gate shifting the threshold voltage of the polFET sensor. This is supported by the observation of Langmuir isotherm response as water vapour increases then the occupancy of sites at the oxide interface increases, thereby increasing the dipole filed and hence the threshold voltage. The hydrophobic polymer does not allow water to reach this polymer/oxide interface and so the baseline is not affected by humidity. Furthermore, such humidity dependence is not observed with the resistive device which operates simply on a bulk swelling model. This undesirable sensitivity to water of the polFET may be removed by using an alternative gate insulator, such as silicon nitride.

The total effect of analyte and water vapour on the sensor is a combination of these two effects and because no cross sensitivity term was observed, the FETs follow

\[ V_T = V_{T0} + aKC_T + \frac{bKC_H}{(1 + KC_H)} \]  

\[ \Delta V_T = aKC_T + \frac{bKC_H}{(1 + KC_H)} \]

where \( C_T \) is the concentration of toluene and \( K_T \) is the partition coefficient.

8. Effect of temperature

The effect temperature on both the magnitude of the response of the sensor and the baseline for a solid gate device has been shown to follow an exponential function. The effect of temperature can be investigated by solving

\[ \frac{dV_{GDS}}{dT} = \frac{dV_T}{dT} - \frac{(V_{GDS} - V_T)}{2g_m} \frac{dg_m}{dT} \]

If we first consider the effect of temperature on the baseline for a standard solid gate MOSFET, without a vapour sensitive layer, then the effect of the temperature on the threshold voltage is minimal \((dV_T/dT \approx 0)\) and so the temperature only effects the \( g_m \) term. Separating this term out we obtain

\[ \frac{dg_m}{dT} = \frac{W}{2L} \left[ \mu \frac{d\rho_n}{dT} + \mu' \frac{d\rho'}{dT} \right] \]

\[ \frac{dg_m}{dT} = \frac{W}{2L} \left[ \mu \frac{d\rho_n}{dT} + \mu' \frac{d\rho'}{dT} \right] \]
Since the change in capacitance per unit area is not very temperature-dependent then, and using Eq. (2), Eq. (17) reduces to

$$\frac{dG_m}{dT} \approx \frac{g_m}{\mu_n} \frac{d\mu_n}{dT}$$

(16)

Then,

$$\frac{dV_{GDS}}{dT} \approx -(V_{GDS} - V_T) \frac{d\mu_n}{dT}$$

(17)

From well-known FET properties, we can state that the majority of the shift in $V_{GDS}$ is due to a modification in the carrier mobility (exponential function), as observed with the solid gate FETs. If this was the only effect then the shift of the solid gate devices would be the same as the polymer device. Results have shown that temperature effect of the gas-sensitive devices are a magnitude greater than the solid gate sensors, furthermore this does not account for the reduction in the magnitude of the response of the sensor to analyte with temperature. Hence, any further effect therefore must be due to a shift in the threshold voltage for devices with gas-sensitive layers. A possible mechanism for the reduction in response at constant concentration was described by Hang et al. [18]. He showed that the bulk solubility and so concentration of a solvent within a polymer reduces with increasing temperature. Hence, even if the concentration of solvent or water is constant in the gas phase, the increase in temperature will reduce the concentration within the polymer and so reduce the magnitude of the response. Thus, for a coefficient $k_{p,g}$ defined as gas volume dissolved in 1 cm$^3$ at a partial pressure $c_i$ for molecules $i$, the ratio of the concentration of solvent in the polymer to the concentration in the same volume of gas can be expressed as

$$k_{p,g} = \frac{c_i^{poly}}{c_i^{gas}}$$

(18)

This was shown to be equal to

$$\log(k_{p,g}) = \frac{4.4T_b}{T} - 4.4 + \frac{0.43 S_0^{el}}{R}$$

(19)

where $T_b$ is the boiling point of the solvent, $S_0^{el}$ the entropy and $R$ the gas constant. This assumes an ideal mixing of molecules and entropy of evaporation of 85 J K$^{-1}$ mol$^{-1}$. Thus, we can define the effect of temperature to be

$$\log(k_{p,g}) \propto \frac{1}{T}$$

(20)

Since the concentration of analyte in the gas above the polymer is constant, the shift in the threshold voltage can be expressed as

$$V_T = V_{T0} + k^* \frac{C_g}{k_{p,g}}$$

(21)

where the shift in the threshold voltage is a function of the concentration of analyte in the polymer and $k^*$ is the temperature dependent term for the bulk solubility. From the previous section we can state that the effect of analyte on the response of the sensor is a linear function, hence the magnitude of the response will decrease as an exponential function of $1/T$. Plotting response for $1/T$, with a logarithmic scale, shows a linear dependence verifying this first-order effect, see Fig. 10. Differentiating in terms of $k_{p,g}$, with temperature dependence we have

$$\frac{dk_{p,g}}{dT} = k_{p,g} \left( \frac{4.4T_b}{T^2} \right)$$

(22)

If we then assume that the shift ($dk_{p,g}$/$dT$) is equal to the shift in the threshold voltage ($dV_T$/$dT$). Then the final effect of temperature on the magnitude if the response ($dV_{GDS}$/$dT$) will be a combination of the shift in the threshold voltage ($dk_{p,g}$/$dT$) and the change in the conductance, brought about by a modification in the carrier mobility ($d\mu_n$/$dT$). The same effect is also observed with for temperature on humidity concentration, as shown in Fig. 6, where an increase in temperature reduces the shift in the threshold voltage from the RH = 0 level, due to a reduction of water molecules in the polymer.

The effect of temperature on the baseline shows a linear reduction in value with increasing temperature at constant humidity and zero analyte concentration. If the shift in this baseline was simply an effect of a change in the bulk solubility to water we would expect an increase of the baseline with temperature. Furthermore, the magnitude of this shift is significantly greater than the effect of humidity or analyte on the sensor, thus further effect is observed. A possible explanation is due to linear thermal expansion of the carbon black/polymer composite material, where temperature is swelling the polymer and so increasing the distance between the carbon spheres. This will again alter the average work function of the composite polymer, shifting the device threshold voltage. To an extent this is supported by results from the resistive devices, where the base resistance of the device increases with temperature. This may also be due to a
thermal expansion of the polymer increasing the resistance of the film.

9. Conclusions

Here we report on a new type of polymer-based ChemFET sensor employing a carbon black/polymer composite gate material. Results have shown a linear dependence to analyte concentration, which we believe is associated with a combination of swelling effect and change in the work function of the polymer and the analyte. Furthermore, it has been found that humidity does not affect the magnitude of the response, though does affect the baseline of the sensor, though the polymer with the highest sensitivity to toluene poly(9-vinylcarbazole) shows no baseline dependence to humidity. This is possible due to the formation of a dipole layer at the oxide/polymer interface. The polymers were also shown to exhibit significant temperature dependence both on the baseline value and in the magnitude of the response, and typically follow an activated function. This we believe is due to thermal linear expansion and a change in the bulk solubility of the polymer.

The ease of film deposition and the many available polymers make these materials attractive for gas sensors. This deposition technique means that these polymers can be deposited onto almost any substrate, thus it is possible to combine these materials with standard CMOS technology to create, low cost, repeatable sensor structures for the low power hand-held electronic nose market.

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References


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D. Briand received his BEng degree and MSc degree in engineering physics from École Polytechnique in Montréal in 1995 and 1997, respectively. His master degree was done in collaboration with the Laboratoire des Matériaux et du Génie Physique (INPG) in Grenoble, France. He is currently a PhD student at the Sensors, Actuators and Microsystems Laboratory (samlab) of the Institute of Microtechnology in Neuchâtel, Switzerland. His present research interests include chemical sensors, micromachining technologies and integration of microsystems.

N.F. Rooij received a PhD degree from Twente University of Technology, The Netherlands, in 1978. From 1978 to 1982, he worked at the Research and Development Department of Cordis Europa N.V., The Netherlands. In 1982, he joined the Institute of Microtechnology of the University of Neuchâtel, Switzerland (IMT UNI-NE), as professor and head of the Sensors, Actuators and Microsystems Laboratory. Since October 1990 till October 1996, he was acting as director of the IMT UNI-NE. Since 1987, he has been a lecturer at the Swiss Federal Institute of Technology, Zurich (ETHZ), and since 1989, he has also been a professor at the Swiss Federal Institute of Technology, Lausanne (EPFL). His research activities include microfabricated sensors, actuators and microsystems.