Chemical capacitive microsensors for volatile organic compound detection

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Abstract

A low-cost, low-power volatile organic compound (VOC) sensor has been constructed from an array of micromachined parallel-plate capacitors. The sensor has demonstrated detection of many VOCs well below the lower explosive limits and could be used in industrial leak monitoring applications or for homeland defense. In place of a standard dielectric, the individual capacitors were filled with selectively absorbing polymers. Absorption of a target vapor alters the permittivity of the polymers and thereby changes the capacitance of the elements in the array. A variety of polymers have been used, including polyethylene-co-vinylacetate, which was sensitive to nonpolar hydrocarbons, and siloxane-fluoro alcohol, which was highly sensitive to polar VOCs and chemical warfare agent simulants. The response magnitude for each element depends on a combination of different phenomenon such as the dielectric constant of the analyte and polymer swelling. The measured sensitivity of the sensor to most VOCs was found to be in the low parts per million (ppm) range. The response magnitude from one capacitor to the next is reproducible to within 3.2\% at 20°C. The sensor typically responded within a second but frequently required 5–10 min to reach equilibrium. Response times could likely be substantially improved with an optimized capacitor structure that contains a decreased gap between the plates and provisions for more rapid vapor exchange.

Keywords: VOC; MEMS; Capacitance; Permittivity; Polymer; Sensor; Array; Dielectric

1. Introduction

Volatile organic compounds (VOCs) are widely used in industry and can present serious medical, environmental, and explosion dangers. VOC sensors are therefore commonly used to monitor oil-drilling platforms, fuel storage tanks, and military applications, because many chemical warfare agents and explosives can be detected with VOC sensor technology.

The ultimate goal of the present work is to produce distributed wireless sensor networks containing numerous battery-operated, low-cost, low-maintenance VOC point sensors. Such networks would provide faster response times and lower operating costs than manually conducted surveys with handheld sensors. To this end we are developing sensors based on selectively absorbing polymers. The selectivity of these polymers results from functional groups that interact weakly with certain target analytes, imparting some degree of preferred interaction. Each element in the sensor array can contain a different polymer, and the collective information from the responses of all elements of the array is used to identify an unknown analyte. Our polymer-based sensors require relatively simple, low-power detection hardware and can operate unheated at ambient temperatures.

1.1. Polymer-based VOC sensors

The amount of VOC absorbed into a functionalized polymer depends not only on the ambient concentration of the VOC, but also on the chemical properties of the VOC and polymer. The specific chemical properties that affect absorption are reflected in solubility-prediction systems such as the Hansen solubility parameters [1,2] or the linear solvation
sensors can have undesirable cross-sensitivities. In the arena of microfabricated chemical sensors, a number of groups have developed VOC sensors based on polymers that selectively absorb the VOC of interest. These sensors measure one or more polymer properties that change when a VOC is absorbed. For example, researchers have used resistive sensors [4–7] and cantilever stress sensors [8–17] to measure swelling of the polymer; resonating cantilevers, surface acoustic wave (SAW) devices [3,18–20], and flexural plate wave (FPW) sensors [21] to measure mass and viscoelasticity changes; and capacitive sensors [22–29] to measure changes in polymer permittivity.

Surface plasmon resonance (SPR) sensors use phonons created from light reflected off of thin metal surfaces, which can be coated to provide selectivity enhancements similar to other polymer-based sensors [30]. Polymer-based sensors that measure changes in a coating’s index of refraction or fluorescence have also been used to detect VOCs as well as other chemical and biochemical compounds [31,32]. These sensors generally operate on the same solubility-based principles as the microfabricated devices, by measuring the changes in the properties of the coatings, however, they may also be sensitive to the optical properties of the target analyte.

Generally, polymer-based sensors can be used to detect most volatile and semi-volatile organic compounds. Highly volatile chemicals, those that are gases at room temperature and pressure, such as methane, are difficult to detect since they do not absorb into polymers well, while low-volatility, solid compounds such as some explosives produce insufficient vapor-phase material for detection. Polymer-based sensors can detect industrial solvents, liquid fuels, many chemical warfare agents, and certain volatile compounds present in commercial explosives. In addition, polymer-based sensors have been used to detect several non-VOC compounds, particularly water vapor [23–27] and ammonia [28].

Although such sensors have proven successful in a number of applications, various difficulties have prevented the development of low-cost, low-maintenance VOC sensors. Variations in polymer composition or nonuniform polymer films can cause poor unit-to-unit reproducibility, necessitating costly calibration of each unit. In addition, sensor output can drift due to chemical interactions of the polymer with the environment, creating a need for frequent recalibration or for periodic rezeroing with automated exposures to clean air. Finally, because no polymer is perfectly selective, the sensors can have undesirable cross-sensitivities.

Various researchers have developed chemical sensors that contain an array of different polymers and/or other selectively absorbing compounds on a single, microfabricated chip with multiple transducers [6,8–10,21,22]. Some of these “electronic noses” combine such arrays with pattern recognition algorithms to analyze the outputs of the various sensors [18–20,33–35]. Electronic noses can identify complex odors, but are especially sensitive to drift and have to be frequently “trained” (i.e. recalibrated). Examples of commercially available electronic nose instruments include a chemiresistor array from Cyrano Sciences Inc. (Pasadena, CA) and a SAW array from Microsensor Systems Inc. (Browning Green, KY). In addition, the ICAD chemical warfare agent sensor from BAE Systems (Rockville, MD) is essentially an electronic nose also based on polymer-coated SAW sensors.

1.2. Chemicapacitive sensors

A “chemicapacitive” sensor (or “chemicapacitor”) is a capacitor that has a selectively absorbing material, such as a polymer, as a dielectric: chemicals absorb into the dielectric, alter its permittivity, and thereby raise or lower the capacitance of the sensor. Polymer-based chemicapacitors in particular are a promising platform for low-cost, low-power sensors, since unlike most other VOC sensors they do not require heating, mechanical excitation, or light sources. However, chemicapacitors are not limited to polymer dielectrics. Other materials have been used to broaden the range of detectable chemicals—sol–gel chemicapacitors, for example, can detect carbon dioxide [23]—although such materials often have to be heated to achieve optimal performance.

To date, chemicapacitors have been developed in two geometries. Interdigitated electrodes [22–25,29] consist of a single layer of metal deposited on a substrate to form two meshed combs. Polymer is deposited on top of the combs. In one example [22], the combs have a periodicity of 1.6 μm, overall dimensions of 0.8 mm × 0.8 mm, and capacitance of about 1 pF. General Eastern (Woburn, MA) offers a humidity sensor based on this technology. Parallel-plate sensors [25–29] consist of a layer of metal deposited on a substrate, followed by a layer of polymer and finally a second, porous layer of metal on top of the polymer. This technology cannot generally produce an array of capacitors on a single sensor chip, since etching the final metal layer would destroy many polymers. Honeywell Inc. (Freeport, IL) and Vaisala (Helsinki, Finland) offer humidity sensors based on this technology, and Vaisala also offers an ammonia sensor [28] based on similar technology.

We have developed a micromachined parallel-plate chemicapacitor (Fig. 1) that, unlike previous parallel-plate chemicapacitors, has a fixed gap width and makes it possible to construct an array of different capacitors on a single chip. These micromachined chemicapacitors are significantly smaller than previous chemicapacitors, so more of them can be integrated on a single sensor chip. In addition,
the response characteristics of our micromachined chem-
icapacitors are highly reproducible from device to device
since the amount of polymer deposited is not critical; ex-
cess polymer lies outside the gap and does not affect sensor
performance [25,29]. Parallel-plate capacitor sensors should
have twice the sensitivity of interdigitated-electrode sensors,
since in the latter, half of the electric field lines pass en-
tirely through the substrate rather than the polymer. On the
other hand, parallel-plate capacitors are more complex to
manufacture, and they have a longer response time since the
polymer is partially covered with the capacitor plate, reduc-
ing the surface area available for vapor exchange [25,29].

In this paper we describe the sensitivity and selectivity
characteristics of these micromachined parallel-plate chem-
icapacitors. We also discuss how the novel response char-
acteristics of these sensors could be used to enhance the
selectivity of VOC sensor systems.

2. Experimental

2.1. Sensor elements

Sensor chips (Fig. 2) were fabricated in the Multi-User
MEMS Process (MUMPs; JDS Uniphase, Research Triangle
Park, NC). All sensor chips measured 5 mm × 2 mm and had
10 parallel-plate capacitors that could each receive a differ-
cent coating. The capacitor plates were made of conductive
polycrystalline silicon consisting of a 0.5 µm-thick bottom
plate resting on the substrate, an air gap that was filled with
polymer subsequent to the MEMS fabrication process, and
a 2 µm-thick ventilated top plate. The capacitor plates were
either circular or square. Circular plate capacitors measured
360 µm in diameter, with a 2 µm gap and a base capaci-
tance of about 0.5 pF. Square plate capacitors measured 300
or 150 µm on a side, with a 0.75 or 2.0 µm gap and base
capacitances of 0.1–1.0 pF.

To minimize flexing of the top plate when the polymer
absorbed VOCs and swelled, the top plate was anchored to
the substrate with posts at approximately 60 µm intervals.
The top plate also had 2 µm diameter etch holes separated
by about 30 µm. These holes were required for removal of
a sacrificial silicon oxide layer during fabrication but also
allowed analyte vapors to pass through.

The gap between the plates was filled with a polymer
 injected either from the edge of the device, through the etch
holes, or through larger fill holes in the top plate. Various
each and fill hole patterns were tested to optimize ease of
coating. For example, some capacitors had many small fill
holes instead of one large fill hole to ensure complete filling
of the device.

2.2. Readout electronics

The readout electronics have been described in a previous
publication about capacitive cantilever-based sensors [16].
The readout electronics were identical for the cantilevers
and the fixed-plate capacitors, since both devices had the
same base capacitance. Also as with the cantilevers [16] the
fixed-plate capacitors are intrinsically explosion-safe; the
electrical power available to and stored in the sensor chip is
insufficient to cause a VOC explosion.

In brief, a charge/discharge readout circuit [8,16] (Fig. 3)
measured the capacitance of each sensor array using a
10 kHz charge/discharge drive voltage, and produced a
The relative permittivity, or dielectric constant of the sensor, was 2.3. Polymers

Polymers and synthesis precursors were purchased from Polysciences Inc. (Warrington, PA), Scientific Polymers Inc. (Ontario, NY), Gelest Inc. (Morrisville, PA), or Sigma–Aldrich (St. Louis, MO). Polymers were selected based on their ability to selectively form weak reversible chemical interactions (hydrogen bonds, van der Waals bonds, and dipole–dipole interactions) with a particular analyte [1–5,36–40]. In general, liquid polymers or polymers with a low modulus of elasticity were preferred because they absorb analytes more quickly than rigid polymers [3].

Experiments were carried out with eight liquid and solid polymers (Table 1), all of which have been used in previous VOC sensors [3,4,18,19]. Polymers were chosen based on the findings in the literature applying these solubility parameters. Other factors in choosing the polymers included stability, ease of acquisition, solubility in a suitable solvent, and detection range. While increasing $\Delta V_{osc}$ could increase the sensitivity of the capacitors, it could also increase the noise level if $\Delta V_{osc}$ was near 1 V. The timing of the reset and sample voltage waveforms, required to make the sensor capacitance measurement, was set by the microcontroller, and the measured output was an analog voltage.

The sensor and readout chips were packaged in a custom-made dual inline package (DIP) with the readout chip in a hermetically sealed cavity and the sensor chip in a second, exposed cavity. The remainder of the readout circuit [16], or “motherboard” (Fig. 4) contained a socket for the DIP, a microcontroller and a 12 bit ADC to digitize the output of the readout chip, and could be made quite small. For each data point acquired, 256 ADC readings were averaged together to achieve effective 16 bit resolution. The smallest measurable capacitance change was 0.25 fF.

The output voltage of filled and unfilled capacitors in the absence of VOCs:

$$\varepsilon_{polymer} = \frac{(C_{sensor})_{filled}}{(C_{sensor})_{unfilled}}$$

where $(C_{sensor})_{filled}$ is the capacitance of the capacitor with polymer in the gap, and $(C_{sensor})_{unfilled}$ the capacitance with polymer in the gap. Assuming that $C_{feedback}$ and $\Delta V_{osc}$ are constants, solving Eq. (1) for $C_{sensor}$ and substituting into Eq. (2) leads to:

$$\varepsilon_{polymer} = \frac{(V_{out} - V_{mid})_{filled}}{(V_{out} - V_{mid})_{unfilled}}$$

The relative permittivity, or dielectric constant of the selectively absorbing polymer was calculated by comparing the output voltage of filled and unfilled capacitors in the absence of VOCs:

$$\varepsilon_{polymer} = \frac{(C_{sensor})_{filled}}{(C_{sensor})_{unfilled}}$$

where $(C_{sensor})_{filled}$ is the capacitance of the capacitor with air in the capacitor gap, and $(C_{sensor})_{unfilled}$ the capacitance with polymer in the gap. Assuming that $C_{feedback}$ and $\Delta V_{osc}$ are constants, solving Eq. (1) for $C_{sensor}$ and substituting into Eq. (2) leads to:

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Experiments were carried out with eight liquid and solid polymers (Table 1), all of which have been used in previous VOC sensors [3,4,18,19]. Polymers were chosen based on the findings in the literature applying these solubility parameters. Other factors in choosing the polymers included stability, ease of acquisition, solubility in a suitable solvent, and ease of coating application. The fluoroalcohol SXFA [36] has an affinity for hydrogen-bonding bases and is useful for the detection of chemical warfare agents such as Sarin. SXFA was synthesized in house from methods.
Table 1
Polymer properties and deposition parameters

<table>
<thead>
<tr>
<th>Polymer Phase</th>
<th>Polymer phase (at 50% RH, 20°C)</th>
<th>Analytes</th>
<th>Measured dielectric constant (in dry N₂, 20°C)</th>
<th>Solvent</th>
<th>Concentration (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyisobutylene (PIB; 1350 MW)</td>
<td>Liquid</td>
<td>Nonpolar hydrocarbons</td>
<td>2.1 ± 0.09</td>
<td>Toluene</td>
<td>1.0</td>
</tr>
<tr>
<td>Polymethyl siloxane (PDMS; 100,000 cSt)</td>
<td>Liquid</td>
<td>Nonpolar hydrocarbons</td>
<td>2.5 ± 0.07</td>
<td>Chloroform or toluene</td>
<td>1.0</td>
</tr>
<tr>
<td>Polycarbonate urethane (PCUT)</td>
<td>Solid</td>
<td>Low polarity hydrocarbons</td>
<td>4.0 ± 0.26</td>
<td>Chloroform</td>
<td>1.0</td>
</tr>
<tr>
<td>Polyethylene-co-vinylacetate with 40% acetate content (PEVA 40%)</td>
<td>Solid</td>
<td>Low polarity hydrocarbons; aromatics</td>
<td>4.1 ± 0.04</td>
<td>Toluene or chloroform</td>
<td>0.75</td>
</tr>
<tr>
<td>Siloxanefluoro alcohol (SXFA) Liquid</td>
<td>Polar VOCs; hydrogen-bonding bases</td>
<td>7.3 ± 0.08</td>
<td>Chloroform</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Polypeichlorohydrine (PECH; 700,000 MW) Gum</td>
<td>Low polarity hydrocarbons; chloroethers</td>
<td>7.4 ± 1.1</td>
<td>Chloroform</td>
<td>0.2</td>
<td></td>
</tr>
<tr>
<td>Cyanopropyl methyl phenylmethyl silicone (OV-225; 9000 cSt)</td>
<td>Liquid</td>
<td>Nitro-aromatics; aromatic VOCs</td>
<td>11.0 ± 0.5</td>
<td>Toluene or chloroform</td>
<td>1.0</td>
</tr>
<tr>
<td>Dicyanoallyl silicone (OV-275; 20,000 cSt)</td>
<td>Liquid</td>
<td>Nitro-alkanes; polar VOCs</td>
<td>33.1 ± 3.7</td>
<td>Acetone</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Table 1. Both the voltage waveform applied to the inkjet (controlling the droplet size) and the amount of time between droplets, were optimized for each combination of capacitor geometry and polymer type.

Sensor capacitance was monitored during deposition, and each capacitor was coated until its capacitance stopped changing, i.e., until the entire gap was filled. Typically, this entailed depositing 100–300 droplets of a solution containing 1.0 wt.% dissolved polymer. Since the droplets were so small, most of the solvent would evaporate before the next drop landed. The sensors were subsequently dried in an oven at 110°C for 30–60 min to completely remove the solvent. After drying, the sensors were visually inspected under a microscope and their capacitance levels were measured to determine if additional polymer was needed to fully fill the gap between the plates. The drying process was repeated after each application of polymer. A small amount of material would remain on top of the top capacitor plates.

Coatings were typically applied to seven “signal” capacitors, leaving one uncoated “reference” capacitor and two unused, unconnected capacitors. The uncoated capacitors did not respond to chemical exposures or changes in relative humidity or flow rate. To reduce systematic common-mode electronic noise, the reference output was subtracted from each of the seven signal outputs. Typically this noise level was reduced from near 1 mV to approximately 0.3 mV, when ΔV_avg was 0.2–0.7 V.

2.4. Polymer deposition

Polymer solutions (Table 1) were deposited onto the sensors with a manually positioned piezoelectric inkjet head (Microfab Technologies MJ-AB01-030 30- or 80-micron dispenser with CT-M3-01 inkjet controller). The inkjet head could deposit individual droplets (30–100 μm diameter) of solution, each of which dried in about 1 s.

Solutions were mixed to a concentration that did not clog the inkjet nozzle (indicating that the solution was too concentrated) or wash away previously deposited polymer (too dilute). The solution concentrations ranged from 0.015 to 2% by weight of polymer in solvent (optimal values are in Table 1). Both the voltage waveform applied to the inkjet (controlling the droplet size) and the amount of time between
treated with the Silcosteel® process (Restek Corporation, Bellefonte, PA) to prevent chemical contamination. A recirculating chiller that pumped water through cold plates or water jackets around the chamber regulated the temperature of each chamber. Each chiller had a closed-loop temperature control system regulated by a four-wire RTD inside the chamber. Humidity inside the chamber was monitored with a Honeywell HIH-3602-C humidity sensor (Honeywell Inc., Freeport, IL).

All VOCs were purchased commercially (Sigma–Aldrich; Alfa Aesar, Ward Hill, MA; Fisher Scientific Inc.; or VWR International Inc., West Chester, PA) and used as received. VOC vapors were generated by bubbling nitrogen or air through gas-washing bottles (bubblers) containing liquid VOCs. To control the VOC concentration, the glass bubblers were immersed in a water bath; the temperature of the bath could be adjusted to vary the vapor pressure of the VOC. Furthermore, the output of the bubblers was diluted with air or nitrogen metered by mass-flow controllers (MKS Instruments, Andover, MA). Humidity was similarly controlled by passing a metered percentage of the nitrogen or air flowing into the test chamber through a bubbler containing water. A LabVIEW (National Instruments, Austin, TX) program varied the temperature, humidity, and concentration of up to six analytes inside the chamber while recording the output from the sensor units.

2.6. Sensor response time

To gauge the differences in response times between liquid and solid polymers, a set of tests was performed with the coated sensors placed perpendicular to the outlet of a diluted vapor stream immediately after a four-way switching valve. For this experiment, OV225, SXFA and PEVA were chosen as representative liquid and solid polymers. Each sensor chip was tested individually, and placed approximately 1 mm from the outlet tube whose inner diameter was 1/16 of an inch. The vapor concentration was 10% of P_{sat} with the bubblers held at 15 °C, and the total flow rate was 500 sccm, which resulted in a vapor plug that could arrive at the sensor in approximately 0.01 s. The electronic four-way switching valve was alternated between dry nitrogen and the diluted analyte vapor and took less than 2 s to switch fully. The coated capacitors did not respond to the switching of the valves, when there was no analyte present. Vapor generation was started 10 min prior to exposure to allow the bubbler temperature to stabilize. During the experiments the uncoated capacitors did not respond to the chemical exposures.

3. Results and discussion

3.1. Sensitivity and response

The chemi capacitor sensitivity to most VOCs is generally comparable to that of other polymer-based VOC senors. We have observed that the chemi capacitors typically have sufficient sensitivity to detect common industrial solvents below 100 parts per million (ppm), and below 1 ppm for the chemical warfare agents simulants and explosives taggants [42]. Table 2 shows the lowest concentration of various analytes—including industrial solvents, chemical warfare agent simulants, and nitro-organics—that we have detected with polymer-filled capacitors. Many of the values in Table 2 reflect the lowest concentration that our test system can deliver, rather than the lowest concentration the sensor can detect. Theoretical limits of detection (LOD) have been calculated using a signal-to-noise limit of 3:1. One can see from the LOD values, that SXFA is more sensitive to the more polar analytes. Four polymers were compared for the detection of CEE; SXFA and OV275 were significantly more sensitive than either PECH or PEVA.

As an example of the data from which Table 2 was generated, Fig. 5 shows the response of an SXFA-filled capacitor to various concentrations of dimethyl-methyl-phosphonate (DMMP, a simulant for sarin) in dry and humid air. Both the DMMP and water vapor cause an increase in the measured capacitance; however the sensitivity of SXFA to DMMP vapor is significantly greater than to water vapor. Exposure to 800 ppb of DMMP causes a change in capacitance that is more than twice that of a change in 40% relative humidity.

The signal-to-noise ratio generally increases with increasing polymer and analyte polarity, an effect that will be discussed further in Section 3.3. In our experience, at a given analyte concentration, the signal-to-noise of a chemi capacitor is roughly proportional to its capacitance. Thus, signal-to-noise improves as plate area increases or as gap width decreases. Although larger capacitors are undesirable because they occupy more area on the sensor chip and therefore cost more to fabricate, smaller gaps have no such disadvantage. In addition, smaller gaps lead to thinner polymer films and therefore faster response. Thus, the ideal gap dimension is the smallest that can be filled with polymer.

We have successfully filled capacitors with 0.75 μm gaps, which is the narrowest gap achievable with the MUMPs process. Over a testing period of several weeks, we have not observed differences in the stability of capacitors with a 0.75 or 2 μm gap.

Although chemi capacitors may exhibit a linear response, particularly when used to measure humidity [24,26,29] in general their response is nonlinear (Fig. 6). McGill et al. [3] have suggested that the response is nonlinear because as the films absorb an analyte, their ability to interact with the analyte changes, generally reducing their capability to absorb more of the same analyte. The ability to absorb other analytes may also be affected; for example, the presence of humidity reduces the sensitivity of an SXFA film to DMMP (Fig. 5). A possible factor contributing to the nonlinear response may be that as the polymer swells, the reduced polymer density between the plates causes a decrease in permittivity. This swelling effect counters the effect of adding a
Fig. 5. Response of an SXFA-filled capacitor to low concentrations of DMMP in a background of dry nitrogen and in nitrogen with 40% relative humidity. The sensor was at 30°C and the oscillation amplitude $\Delta V_{osc}$ was 0.1 mV. The response to 800 ppb of DMMP was approximately 167 and 200 mV with and without the background humidity, respectively, and the peak-to-peak noise level was 0.5 mV.

Table 2

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Lower explosive limit (LEL) (ppm)</th>
<th>Immediate dangerous to life and health (IDLH) (ppm)</th>
<th>Lowest detected concentration (ppm)</th>
<th>S:N</th>
<th>LOD (ppm)</th>
<th>Polymer used</th>
<th>$\Delta V_{osc}$ (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>25000</td>
<td>2500</td>
<td>11</td>
<td>17</td>
<td>2</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>50000</td>
<td>500</td>
<td>25</td>
<td>10</td>
<td>8</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Benzene</td>
<td>12000</td>
<td>500</td>
<td>142</td>
<td>5</td>
<td>85</td>
<td>OV225</td>
<td>0.2</td>
</tr>
<tr>
<td>Bromobenzene</td>
<td>5000</td>
<td>N/A</td>
<td>7</td>
<td>50</td>
<td>0.4</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>20000</td>
<td>2000</td>
<td>37</td>
<td>20</td>
<td>6</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Ethyl alcohol</td>
<td>33000</td>
<td>3300</td>
<td>63</td>
<td>12</td>
<td>16</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Isopropyl alcohol</td>
<td>20000</td>
<td>2000</td>
<td>84</td>
<td>18</td>
<td>14</td>
<td>SXFA</td>
<td>0.2</td>
</tr>
<tr>
<td>Methyl alcohol</td>
<td>60000</td>
<td>6000</td>
<td>21</td>
<td>9</td>
<td>7</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Octane</td>
<td>14000</td>
<td>1000</td>
<td>49</td>
<td>5</td>
<td>29</td>
<td>OV225</td>
<td>0.2</td>
</tr>
<tr>
<td>Tetrahydrofuran (THF)</td>
<td>20000</td>
<td>2000</td>
<td>7</td>
<td>15</td>
<td>1</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Toluene</td>
<td>11000</td>
<td>500</td>
<td>61</td>
<td>3.3</td>
<td>55</td>
<td>OV225</td>
<td>0.2</td>
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</table>

Chemical warfare agent simulants

<table>
<thead>
<tr>
<th>Chloroethyl ether (CEE)</th>
<th>N/A</th>
<th>N/A</th>
<th>1</th>
<th>3</th>
<th>1</th>
<th>PECH</th>
<th>0.5</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.4</td>
<td>3</td>
<td>0.4 PEVA</td>
<td>1.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>10</td>
<td>0.06 SXFA</td>
<td>0.4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.2</td>
<td>10</td>
<td>0.07 OV275</td>
<td>0.075</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Diisopropyl methylphosphonate (DIMP)</th>
<th>N/A</th>
<th>N/A</th>
<th>0.1</th>
<th>100</th>
<th>0.003</th>
<th>SXFA</th>
<th>0.4</th>
</tr>
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<tbody>
<tr>
<td>Dimethyl methylphosphonate (DMMP)</td>
<td>N/A</td>
<td>N/A</td>
<td>0.18</td>
<td>300</td>
<td>0.002</td>
<td>SXFA</td>
<td>0.4</td>
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</tbody>
</table>

Explosives byproducts and impurities

<table>
<thead>
<tr>
<th>Nitro-benzene</th>
<th>N/A</th>
<th>N/A</th>
<th>0.02</th>
<th>13</th>
<th>0.005</th>
<th>SXFA</th>
<th>0.2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.44</td>
<td>10</td>
<td>0.1</td>
<td>OV275</td>
<td>0.075</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitro-propane</td>
<td>N/A</td>
<td>N/A</td>
<td>5</td>
<td>25</td>
<td>0.6</td>
<td>SXFA</td>
<td>0.4</td>
</tr>
<tr>
<td>Nitro-toluene</td>
<td>N/A</td>
<td>N/A</td>
<td>0.001</td>
<td>23</td>
<td>0.0001</td>
<td>SXFA</td>
<td>0.2</td>
</tr>
</tbody>
</table>

The responses (Fig. 6) of the SXFA films show that humidity has little influence on the acetone response at low acetone concentrations, however above 15,000 ppm of acetone, the high humidity background causes a larger response from the capacitors. Again, these data are presented as changes from the baseline at each relative humidity. In typical commercial applications, such as industrial leak detection, we expect that an embedded microcontroller will take measurements from a temperature sensor and humidity-sensitive chemicapacitor in addition to the analyze-sensitive chemicapacitor, and then use a multiple-parameter calibration table or pattern recognition algorithm to determine the analyte concentration.
due to vapor absorption can take the form of changes in the volume fraction of polymer and analyte. In addition, as long as the capacitors are completely filled, their topology will not affect the capacitor’s capacitance. This is an important advantage of this form of chemcapacitor, potentially eliminating the need to individually calibrate sensors and thereby reduce production costs.

3.3. Response time

Many factors influence a polymer coated sensor’s response time, including mass transport limitations, vapor concentration, the stiffness, crystallinity and thickness of the polymer and the molar volume and diffusivity of the analyte [43]. In general, large analyte molecules will diffuse slower through polymers than small molecules. Solid polymers will reduce the rate of analyte diffusion compared to liquid polymers, and polymers that have a high amount of crystallinity will also have slower responses compared to rubbery or amorphous polymers. In fact, it has been shown that the diffusion constant of molecules can decrease by several orders of magnitude for relatively small increases in the molecules size [43]. Results in the literature suggest that thin layers of liquid polymers should produce the fastest sensors, especially with smaller analyte molecules.

The response time of our sensors, as shown in Table 3, generally conforms to this pattern. Both SXFA and OV225 are liquid polymers, while PEVA with 40% vinyl acetate is a rubber-like polymer with approximately 10% crystallinity (the crystallinity of PEVA 40% was estimated from data in [44]). For the three circular plate capacitors that were tested; the onset of response occurs within 1 s for the four vapors tested for SXFA and OV225, and within 3 s for PEVA.

Table 3 shows that the time to achieve 10% of full response ($t_{10}$) to all four vapors for the two liquid polymers is only a few seconds to about 20 s, while $t_{10}$ for the solid PEVA films is typically longer. The time to achieve 90% of full response ($t_{90}$) ranges from 20 s for SXFA and acetone, to many minutes depending on the stiffness of the polymer and the diffusion of the analyte. In all cases, the liquid polymers’ $t_{10}$ absorption and desorption response was faster than the solid polymer’s responses. In addition, it is apparent that desorption can be slower than absorption, especially if the polymer interacts strongly with the analyte. SXFA in particular exhibits slower desorption than adsorption, presumably due to strong acid–base interactions with the analyte.

Polymers may “relax” [43] when responding to certain analytes and especially at large concentrations, i.e. the signal reaches a maximum and then begins to decline during an exposure to a constant concentration of analyte. This behavior could occur because the polymer expands and begins to seep out of the capacitor gap. Relaxation artificially lowers the 0–90% response time, since the maximum response occurs before the sensor has finished responding to the analyte. Another factor that could effect the measured response time for the acetone exposures is evaporative cooling; where the

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**Fig. 6. Average response of four SXFA-filled capacitors to varying concentrations of acetone in dry nitrogen at 20, 30 and 40 °C (upper graph). Average response of the same sensors to varying concentrations of acetone at four different relative humidity levels in nitrogen at 20 °C (lower graph).**

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3.2. Reproducibility

To test the reproducibility of the deposition process, four identical SXFA-filled capacitors were produced on each of six sensor chips. These chips were separately given 30 min exposures of 10% $P_{sat}(15\degree C)$ acetone in dry nitrogen at 20 and 40 °C, and their response magnitudes were measured. The coefficient of variation for the four capacitors on any single chip averaged 3.2% at 20°C and 1.9% at 40°C. The chip-to-chip variation averaged 9% at 20°C and 6% at 40°C.

We hypothesize that capacitor-to-capacitor variability decreases at the higher temperature because the sensors settle faster. That is, at higher temperatures, acetone diffuses more quickly into the polymer, and a capacitor reading is more likely to be made under a steady-state condition. This reduces or removes one source of variability, i.e. differences in diffusion speed between different capacitors and between sensor chips.

Thus, inkjetting methods can make chemcapacitors that vary in response by only a few percent. Unlike most other polymer-based sensors, the structure of the capacitors fixes the volume being sensed. Changes in the dielectric material

---

**Table 3**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>$t_{10}$</th>
<th>$t_{90}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>10 s</td>
<td>20 s</td>
</tr>
<tr>
<td>SXFA</td>
<td>1 s</td>
<td>3 s</td>
</tr>
<tr>
<td>PEVA</td>
<td>3 s</td>
<td>6 s</td>
</tr>
</tbody>
</table>
Table 3
Time to reach 10% \((t_{10})\) and 90% \((t_{90})\) of maximum response at 20–25°C and 0% humidity; average response ± standard deviation; analyte concentration = 10% of \(P_{atm}\) at 15°C

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Analyte</th>
<th>Absorption</th>
<th>Desorption</th>
<th>Absorption</th>
<th>Desorption</th>
<th>Response</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>(t_{10}) (s)</td>
<td>(t_{90}) (s)</td>
<td>(t_{10}) (s)</td>
<td>(t_{90}) (s)</td>
<td>(mV)</td>
</tr>
<tr>
<td>PEVA</td>
<td>Toluene</td>
<td>14.3</td>
<td>18.6</td>
<td>366</td>
<td>510</td>
<td>-90.8 ± 4.6</td>
</tr>
<tr>
<td></td>
<td>Acetone</td>
<td>21.1</td>
<td>10.0</td>
<td>312</td>
<td>378</td>
<td>40.8 ± 6.6</td>
</tr>
<tr>
<td></td>
<td>DMMP</td>
<td>65</td>
<td>49.2</td>
<td>1548</td>
<td>1812</td>
<td>3.9 ± 0.3</td>
</tr>
<tr>
<td></td>
<td>Water</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>Negligible response</td>
</tr>
<tr>
<td>SXFA</td>
<td>Toluene</td>
<td>2.6</td>
<td>3.9</td>
<td>318</td>
<td>690</td>
<td>172.8 ± 12.8</td>
</tr>
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<td></td>
<td>Acetone</td>
<td>4.2</td>
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<td>19.8</td>
<td>228</td>
<td>750.0 ± 23.7</td>
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<tr>
<td></td>
<td>DMMP</td>
<td>15.8</td>
<td>11.0</td>
<td>138</td>
<td>3084</td>
<td>1170.8 ± 3.5</td>
</tr>
<tr>
<td></td>
<td>Water</td>
<td>3.7</td>
<td>9.12</td>
<td>636</td>
<td>2016</td>
<td>78.3 ± 10.3</td>
</tr>
<tr>
<td>OV225</td>
<td>Toluene</td>
<td>8.6</td>
<td>13.8</td>
<td>168</td>
<td>396</td>
<td>-27.0 ± 1.4</td>
</tr>
<tr>
<td></td>
<td>Acetone</td>
<td>2.1</td>
<td>2.2</td>
<td>84</td>
<td>84</td>
<td>44.8 ± 5.0</td>
</tr>
<tr>
<td></td>
<td>DMMP</td>
<td>2.9</td>
<td>6.7</td>
<td>558</td>
<td>402</td>
<td>66.8 ± 5.7</td>
</tr>
<tr>
<td></td>
<td>Water</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>Negligible response</td>
</tr>
</tbody>
</table>

\*4.3% relaxation in 1 h.

3.4. Response mechanism

As Eq. (3) illustrates, the output of a chemicapacitor reflects the permittivity of its selectively absorbing dielectric. The sensor responds to chemicals when the dielectric absorbs an analyte, causing its permittivity to change. Hierlemann et al. [22] have previously hypothesized that a complex combination of response phenomena occurs and that the degree of change depends on four factors: (1) dielectric chemical structure modification from reversible weak interactions (hydrogen, dipole, van der Waals) with the analyte; (2) the amount of swelling that the dielectric undergoes upon absorbing the analyte (swelling reduces the density of dipole moments within the polymer, and therefore the permittivity of the polymer); (3) the permittivity of the analyte being absorbed; and (4) the amount of analyte that absorbs into the dielectric.

Unlike the interdigitated design of Hierlemann et al. [22], in the present design, the fixed parallel-plate structure that we have employed causes all of the electric field lines to pass through the polymer between the plates. Consequently, the measured capacitance is only dependent on the properties of the material between the plates, which is a fixed volume. Upon vapor absorption the polymer can swell through both the fill holes and vent holes, and the electric field will then pass through a polymer with lower density than in its neat state. Therefore, swelling alone would cause the capacitance to decrease. In contrast, the absorption of any analyte without concurrent swelling, whether it has a higher or lower dielectric constant than the polymer will increase the capacitance of the device, because it is an additive effect. The capacitance of the device upon vapor exposure can thus either increase or decrease, depending upon whether the swelling or chemical absorption dominates.

Two capacitors on the same chip with different gaps were coated with PIB and tested against octane and toluene vapors. The results, shown in Fig. 7, show a larger response from the 0.75 μm gap device versus the 2.0 μm gap device. The noise level during the experiment was ±0.15 mV for both devices. This suggests that the sensing gap can be manipulated if needed to achieve greater signal-to-noise levels. The response of PIB to stepped concentrations of octane and toluene shows a change in direction associated with the permittivity of the each analyte compared to the polymer. For octane swelling dominates the sensor response, and the overall capacitance decreases, while with toluene, which has a slightly higher permittivity than octane, the capacitance increases. This effect has been observed in several polymers with a variety of permittivity values, resulting in a relative sensitivity pattern that shifts direction depending upon the polarity of the polymer and analyte and on the polymer’s ability to swell. In this example, PIB has a solubility parameter [39] \(\delta = 15–16.5 \text{MPa}^{1/2}\) and a dielectric constant \((\varepsilon = 2.1–2.4)\) that are between the values for octane \((\delta =\)
Fig. 7. Response of two PIB-filled chemicacpacitors on the same sensor chip to octane and toluene vapors in dry nitrogen at 20 °C (lower graph). The capacitor that produced larger responses (both positive and negative voltage shifts from baseline) has a 0.75 µm gap, while the other has a 2 µm gap. Concentrations are given as a percent of the saturated vapor pressure of the VOCs at 15 °C (upper graph). Both capacitors were measured with the same electronic settings.

14.1–15.5 MPa^{1/2}, \( \varepsilon = 1.95 \)) and toluene (\( \delta = 18.3 \) MPa^{1/2}, \( \varepsilon = 2.4 \)). From these solubility parameter values, both octane and toluene would be expected to cause a large amount of swelling in the PIB coating [1,4,5].

To further illustrate the response characteristics of polymer-filled chemieapacitors, sensor response is plotted as a function of polymer permittivity and analyte permittivity in Fig. 8. It is apparent that, in general, high-permittivity analytes produce larger responses than low-permittivity analytes, and low-permittivity analytes such as octane can produce negative responses. The negative sensor responses (decreases in relative permittivity) arise when the permittivity of the absorbed analyte is insufficient to compensate for the swelling that the polymer undergoes upon absorbing the analyte. In general, low-permittivity analytes are difficult to sense because they only produce small responses when they are absorbed.

For example, previously reported swelling and mass-uptake experiments [3,4,38,40,46] indicate that the polymers PIB and PDMS should absorb octane, benzene, and toluene more readily than they absorb THF. However, in PIB and PDMS-filled chemicacpacitors, octane, benzene and toluene actually produce much smaller responses than THF. This is presumably because the dielectric constant of THF is about three times higher than that of octane, benzene, or toluene.

Fig. 9 shows the data from Fig. 8 for the PIB-filled capacitor with comparable data from the literature for a PIB coated chemiresistor [47]. Both data sets have been normalized to show them on the same scale. The differences in the data and PDMS-filled chemicacpacitors, octane, benzene and toluene more readily than they absorb THF. However, in PIB and PDMS-filled chemicacpacitors, octane, benzene and toluene actually produce much smaller responses than THF. This is presumably because the dielectric constant of THF is about three times higher than that of octane, benzene, or toluene.

Fig. 8. Sensor response as a function of polymer and analyte permittivity. Each data point was generated from between three and six identically prepared sensors, each of which was exposed to three-five doses of analyte. The data points represent averaged values and the error bars reflect the standard deviation of these values. The concentration of all analytes was 20% of the saturation vapor pressure at 15 °C. All measurements were taken at 0% humidity and 20 °C.
Electric constants above 22 produce small signals because their permittivity is high. Finally, analytes with dielectric constants below 3 produce large signals even though they may not absorb as well, due to the added permittivity due to the absorbed analyte. Polymers with relative permittivity values under 3 produce small or negative signals because their permittivity is low and the absorbed amount is insufficient to counteract the large amount of swelling. On the other hand, we observe that analytes with dielectric constants between 3 and 21 produce large signals, even though they may not absorb as well, because their permittivity is high. Finally, analytes with dielectric constants above 22 produce small signals because their permittivity is high.

In contrast, the high-polarity polymer OV275 absorbs high-polarity analytes well, producing large signals in response to methanol and water. Although SXFA also has a high polarity, its acidic hydrogen-bonding character results in low absorption of water and methanol. Neither OV275 nor SXFA absorbs enough octane vapor to produce a negative response from swelling. PEVA, PCUT, PECH, and OV225 exhibit larger negative responses to benzene than to octane, presumably due to greater swelling in the presence of benzene.

While this study is by no means a comprehensive study of the wide variety of polymers available for testing, the results show that a simple array of such coatings could satisfy the needs for a wide range of VOC detection applications.

4. Conclusion

We have shown that micromachined, parallel-plate capacitors can be filled with polymers and used to detect VOCs, chemical warfare agent simulants, and explosive taggants. Chemicapacitors are uniquely selective among polymer-based VOC sensors in that they offer selectivity due to the characteristic electrical properties of the analyte in addition to the selective sorption due to the polymer/analyte interactions. This unique sensitivity of chemicapacitors to analyte permittivity can potentially lead to sensor systems with improved selectivity. In particular, varying response characteristics can be ensured by contrasting the different detection modalities of a polymer-filled chemicapacitor with that of a mass-sensitive transducer, such as a microcantilever, coated with the same polymer. Because both transducers can be fabricated on the same chip and coated in a single process, such a combination would be cost-effective and compact.

Acknowledgements

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References


[41] OV225 is a stationary phase used by OSHA to separate and verify the responses to organic vapors, Anal. Chem. 65 (1993) 2055–2066.


Biographies

Sanjay Patel received a BS in chemical engineering from the University of California at Berkeley, in 1992. He received his MSE and PhD in chemical engineering from the University of Michigan, in 1993 and 1997, respectively. His PhD research was on the interactions of combustible gases with metallic and semiconducting thin films on microfabricated hotplates, for use as automotive exhaust gas sensors. In 1997, Dr. Patel became a postdoctoral appointee at Sandia National Laboratories, where he developed chemiresistive polymer-composite films and sensors for the detection of volatile organic compounds; sensor platforms for low-power chemical detection of solvents; and novel diffusion barriers for hydrogen sensors. He joined Graviton Inc., in July 2000 to develop microcantilever-based gas sensors, and helped to develop the chemiresistive sensor technology. In 2005, Dr. Patel co-founded Seacoast Science Inc., a sensor development company, located in Carlsbad, CA. Dr. Patel has authored or coauthored several journal articles in the field of chemical sensing and MEMS.

Todd Mlsna graduated from Albion College in Albion, Michigan, in 1985 with a degree in chemistry. He received his PhD from the University of Texas at Austin in 1990, where he studied organofluorine chemistry, and subsequently became a visiting assistant professor at Clemson University. From 1994 until 1998 he worked at the Naval Research Laboratory (NRL), developing polymer-film-based chemical sensors. His paper “Aromatic Fluoroalcohol Coatings for Explosive Detection” won the Best Paper award at the International Sensor Conference in Beijing in 1998, and the NRL recognized his work on pulsed laser deposition of polymer films in 2000 as one of the year’s best patent applications. Following his time at NRL, he became director of analytical chemistry at Sensor Research and Development Corporation, where he developed polymer-based surface acoustic wave sensors. He joined Graviton as a senior staff scientist in August 2000, where he developed polymer formulations for microcapacitor sensors. In February 2003, Dr. Mlsna co-founded Seacoast Science Inc., in Carlsbad, CA. Dr. Mlsna has over 60 presentations and publications.

Berndt Fruhberger has worked in the areas of surface chemical reactions, thin film deposition and spectroscopic characterization of surfaces for over 10 years. He received a diploma in chemistry from the University of Heidelberg in 1989, for studies of oscillating surface reactions, and a PhD in chemistry, also from the University of Heidelberg, for research on the interface chemistry of metal oxide gas sensors. As a postdoctoral fellow at Exxon’s Corporate Research Laboratory, he modified transition metal surfaces to tailor their reactivities in hydrocarbon surface reactions. He then held positions as a senior scientist at the BOC Group Technical Center, where he modified metal surfaces for use in corrosive environments, and as a senior staff scientist at Sensor Research and Development Corporation, where he developed thin film metal oxide sensors. Dr. Fruhberger’s scientific work has led to over 25 publications in peer-reviewed journals, and numerous presentations at national and international conferences. He joined Graviton in June 2000 where he developed palladium films for microcantilever hydrogen sensors. In 2003 he joined the University of California at San Diego, as a staff researcher where he develops microfabricated sensors and chemically sensitive coatings.

Erno Klaassen’s expertise is in the areas of MEMS and analog integrated circuit design. He received a BS in electrical engineering from the University of California at Berkeley in 1992, and MS and PhD degrees in electrical engineering from Stanford University in 1993 and 1996, respectively. His PhD dissertation, Micromachined Instrumentation Systems, describes a set of fully integrated microsystems built using a novel micromachining process based on a commercial CMOS process. Presentations on this work received Best Paper awards at the IEEE Solid State Circuits Conference and at the International Semiconductor Device Research Symposium. Dr. Klaassen has held positions as analog IC designer at Analog Devices; as research staff member at the IBM Almaden Research Center, where he developed MEMS and electronics for magnetic data storage applications; and as senior MEMS and analog designer at Optical Micromachines, where he was lead designer for a family of bulk-micromachined MEMS actuators for optical switching applications. Dr. Klaassen joined Graviton in August 2001 and is currently a senior engineer at Intel.

Sabina Cemalovic was born and raised in Sarajevo, Bosnia. She graduated from the Third Gymnasia Chemistry Technical School in 1989 and the University of Technical Science at Sarajevo in 1993. During these years she was also a volunteer for the Red Cross in Sarajevo. After spending 1995 in Zagreb, Croatia, in transit to the USA, in 1996 she joined the American Technology Group in Monrovia, CA. In 1999 she joined Cyano Sciences Inc., in Pasadena, CA, where she helped to develop a hand-held electronic nose. She worked at Graviton from 2000 to 2003 and was responsible for fabricating and characterizing Graviton’s microcapacitor sensors. In May 2003, she joined Seacoast Science Inc., as a researcher to develop chemical sensors.

David Baselt received an MS in chemistry in 1988 from the University of California at Berkeley, and a PhD in physical chemistry in 1993 from the California Institute of Technology. As a graduate student, he designed and constructed one of the first multimode atomic force microscopes, which was subsequently commercialized by Topometrix Inc. (now ThermoMicroscopes). Following a postdoctoral fellowship in video microscopy at Yale University, he worked at the Naval Research Laboratory developing microcantilever- and magnetoresistor-based biosensors that use magnetic forces to measure the strength of intermolecular bonds. This work resulted in several patents that were later licensed by Graviton; presentations including a talk at the 1998 Gordon Conference on Bioanalytical Sensors and a keynote talk at Biosensors’98; and awards including Best Paper at Biosensors’98, the Berman award for best applied research paper in NREL’s chemistry division at the NREL Technology Transfer award.