The Chemical Nano-Sensor Development and Characterization

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ABSTRACT

The nanotechnology will be critical for the near future success of the US economy. In this paper, I will present study of the chemical nanosensors for the space and environmental applications, the safety alert devices, etc. I will demonstrate a high-resolution CNS that is applied to the rocket fuel hydrazine leak detection. The CNS detects the changes in the electrical conductivity response during the chemical species presence. When the hydrazine is leaked into air, it immediately dissociates into NO\textsubscript{2}. Detailed works in this paper are as follows. I will discuss the sensor chips preparation and process control. Furthermore, detailed studies of the CNS show responses to varying NO\textsubscript{2} concentration and nanomaterials.

Keywords: chemical nano sensor, hydrazine, nano technology, nanosensor stability

1 BACKGROUND

Nanoscience and nanotechnology, through the exploration and control of the nanomaterials at the nanometer scale, is considered as one of the key research areas for the future growth of US economy. Many sensor devices are part of our everyday life. More sensor improvements are needed for small size, great sensitivity and selectivity, fast response, minimal power consumption, and reliability demands, etc. Due to the well organized structure in atomic level of nanomaterials and their large surface-to-volume ratio, nanosensors are becoming very attractive for the next-generation of the sensing devices. Chemical nanosensors (CNS) are fabricated for the space and environmental applications. For example, we can apply the CNS that detects the electrical signal during the chemical species presence.

Figure 1 is a conceptual diagram where the sensor is placed so that the physical and chemical environment can be monitored and controlled. These conditions include the total gas flow rate, chemical concentration, humidity, chemical interface, temperature, pressure, etc. When chemical gases pass by CNS, the nanomaterials in the sensor platform respond correspondingly. The sensor response by electrical conductivity change is a result of the chemical sensing. Each sensor response is monitored electronically and is recorded in the computer as a sensor signal and for the further data processing.

The purpose of our CNS project is to monitor the trace amount of NO\textsubscript{2} composed from the leakage of one fuel component, hydrazanine. The liquid hydrazine (N2H4) is an efficient rocket propellant. When the N2H4 is leaked into air, it immediately dissociates and produces NO\textsubscript{2}.

As published in the previous literatures [1], carbon nanotubes (CNT) is very sensitive to NO\textsubscript{2} and it is therefore a very promising CNS to be employed as a commercial sensor product. The sensor development in this study will focus on the CNS and its NO\textsubscript{2} response in relationship to various NO\textsubscript{2} concentrations. In terms of the dry NO\textsubscript{2} analyte response, we will investigate the...
CNS on the effects of various variables such as nanomaterials and gap size, etc.

We surveyed many sensing nanomaterials that include: 1) CGNT, 2) CGNT+MPC, 3) CGNT+polymer. The nanomaterials are as follows. The CGNT is the CVD grown nanotubes [2]. The MPC is monolayer-protected gold clusters (MPC) [3]. The polymer is cellulose hydroxypropyl [1,3].

We employ CGNT as the base nanomaterial in the form of the CVD growth. Illustration of Figure 2 procedures shows the nano coating to prepare a sensor chip before its sensor application.

2 IV CURVE, BIAS OPTIMIZATION

2.1 IV characterization

The IV characteristics of several nanomaterials are studied in the voltage windows and in the reversal voltage as well. The typical

![Figure 2: The flow chart shows preparation procedures of the clean CNS chip.](image1)

IV curves are shown in Figure 3. For example, we investigate the IV sweep curves from typical sensors with a CNT/MPC nanomaterial (on the upper left), a CNT/cellulose nanomaterial (on the upper right), and the CNT nanosensors (on the lower left). We have plotted a variety of IV curves in Fig. 3a, 3b, and 3c) for the 4µm feature gap of three typical inter-digitated electrode sensors. The non-linearity of the IV curves is also very interesting in order to identify the optimal sensors operating regime.

2.2 Bias voltage optimization

Moreover, we study the bias effects by applying different DC bias values. As stated at above, the electrical resistance of the CNS may be nonlinear. Therefore, we choose several different dc-bias voltages to measure the sensor response curve at various NO$_2$ concentration.
3. SENSOR STUDIES

We detect the baseline for the gas flow with pure air that shows no signal. Then we expose a sensor by applying on a sensor the chemical/gas flow with a concentration programmed in the same total flow. The change in the electrical signal is measured and the response is extracted from the sensor. Following this step, the sensor is purged to recover. After having enough purge time, go back and iterate the exposure-purge steps until the sensing process finish.

Figure 4 is a typical KAC31 run, where the chip KAC31 is deposited with nanomaterials of CGNT-only, CGNT plus MPC composite, and CGNT plus polymer composite, respectively. The sensors show strong response.

We analyze the data and the relationship between the resistance change and the chemical flow as follows:
1. Make a linear fit to the drift baseline (on the initial 15’ conditioning);
2. The baseline resistance is taken near the end of the recipe step-1, that is the end of the initial conditioning;
3. The baseline is extrapolated as a function of time by using a linear regression method;
4. The response $dR$ is calculated as the difference between the resistance signal and the baseline at the time immediately after the exposure step. As shown in Figure 4, the CNS response steps are extracted for every concentration.

Furthermore, the analysis yields the $dR$ and $dR/R_0$ dependence upon NO$_2$ concentration. By extracting $dR$/noise ratio for every sensor at all concentrations, we calculate the sensitivity function and plot this function in Figure 5 versus the sensors. As a remark, the sensor’s resolution in terms of the sensitivity limit, $S/N$, can also be derived by an extrapolation method.

<table>
<thead>
<tr>
<th>Step Number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
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<tr>
<td>Time (minutes)</td>
<td>15’</td>
<td>10’</td>
<td>15’</td>
<td>10’</td>
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<td>15’</td>
<td>10’</td>
<td>15’</td>
<td>10’</td>
<td>15’</td>
</tr>
<tr>
<td>NO$_2$ (ppm)</td>
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<td>0.5</td>
<td>1</td>
<td>0</td>
<td>2</td>
<td>0</td>
<td>5</td>
<td>5</td>
<td>0</td>
<td>10</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 2: The table shows a typical recipe of the NO$_2$ sampling with total flow rate 400CCM.

![Figure 5](image)

Figure 5: These charts show the relative response traces of the KAC31 chip with two RH levels: a) 0% or dry, b) 50%. The NO$_2$ concentration varies as shown at 0.5-, 1-, 2-, 5-, and 10-ppm.

We studied the sensor’s responses at various humidity values. We observed that, for the response at low humidity range of 0% to 30% RH, the relative sensor response shows that the humidity in this range has quite little effects on the nanosensor response. The NO$_2$ response increases with the increasing humidity at a value of 50% and greater.

4. SUMMARY

In summary, We have studied the trace concentration of NO$_2$ below 1-ppm regime. Further studies are in the progress to characterize
the life-expectance of CNS and the effect of temperature, pressure, etc.

References:
[1]: J. Li, Y. Lu, Q. Ye, M. Cinke, J. Han, and M. Meyyapan, NanoLett. 3, 929 (2003).