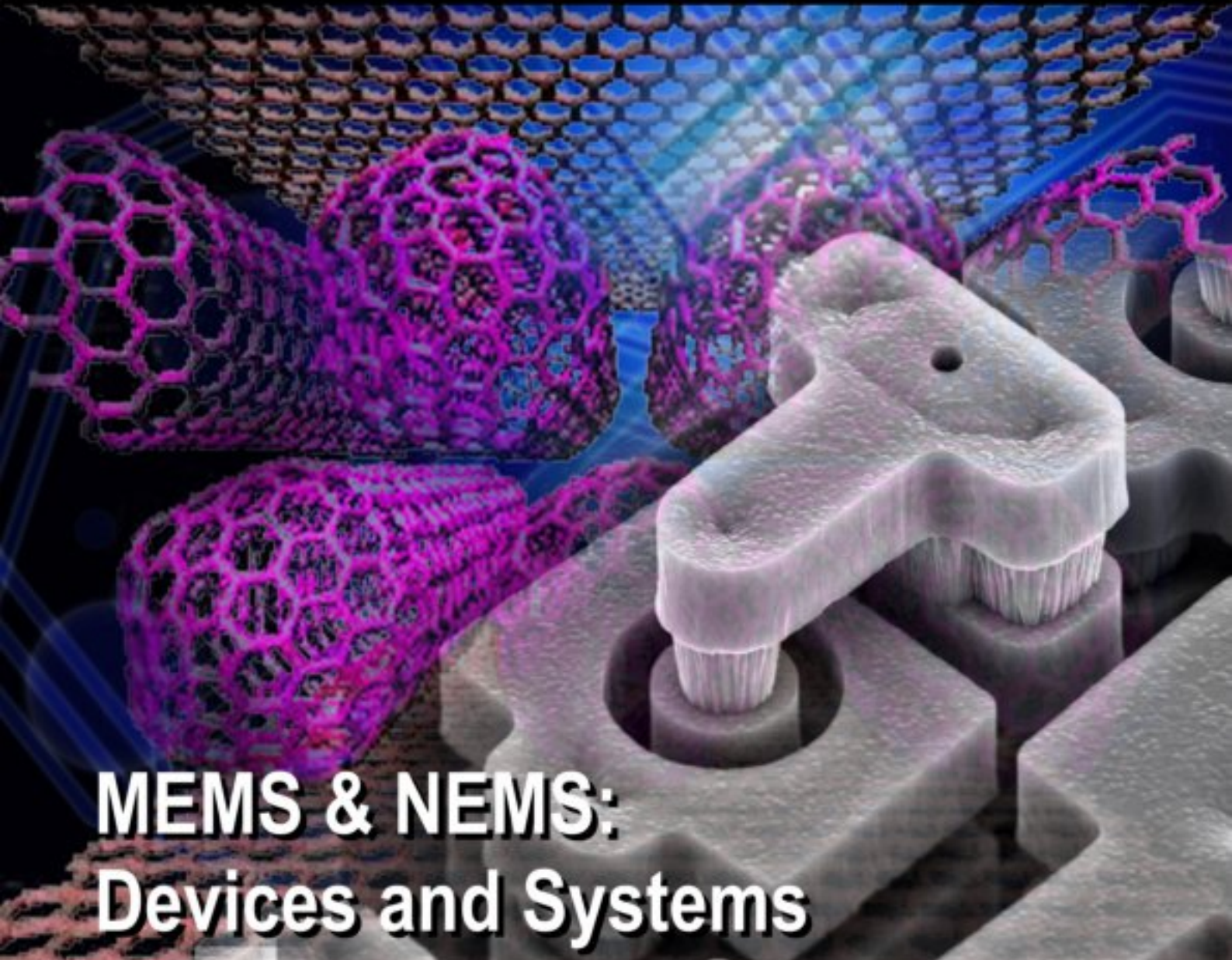


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Please visit journal's webpage with preparation instructions: <http://www.sensorsportal.com/HTML/DIGEST/Submission.htm>

Foreword

The 10th annual NSTI Nanotech Conference and Trade Show was held this year during 20-24 May at the Santa Clara Convention Center, in Santa Clara, California. The conference has grown this year to host 3000 attendees and 250 exhibitors, while the resulting proceedings boasts over 3000 pages of peer-reviewed micro and nanotechnology research.

A number of authors publishing in the Joint Electronics and Microsystems Symposia track were invited to submit a revised version of their papers to this special issue. Papers were selected from a number of symposia within the track, including: MEMS & NEMS, Sensors & Systems, Micro & Nano Fluidics, and MSM – Modeling Microsystems. These symposia brought together researchers from a number of disciplines to discuss topics ranging from theoretical developments, to design and fabrication, through to industrial applications of MEMS and NEMS sensors, devices and systems.

The joint symposia are motivated by the dream of smarter, smaller, and more complex systems that integrate micro and nano system technologies with intelligence, power and communication ability at the same micro or nano scale. The resulting increase in complexity poses an enormous challenge to engineers when designing, modeling, and fabricating such integrated micro and nano systems. The joint symposia aimed at bringing together researchers from different disciplines to exchange ideas about how to best develop such systems.

As with the joint symposia, this special issue includes papers ranging from those with a higher level focus to those covering low-level physical aspects of MEMS and NEMS devices and their modeling and fabrication. Four of the papers presented in this special issue correspond to invited talks: Sanna et al., examine miniaturization trends in preventative medicine and include some results from the EU project ANGEL; Adams et al., describe the results of the NASA funded GEMSTONE project, which involved creating and field-testing a small system of atmospheric probes; French and Yang explore the opportunities and pitfalls of scaling, whilst Nieva presents a number of new trends for using MEMS sensors in harsh environments.

We are very thankful both to the NSTI directors and Nanotech chairs (Dr. Matthew Laudon and Dr. Bart Romanovicz) and to the *Sensors & Transducers Journal* for offering the opportunity to publish this special issue.

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The Development of Chemical Nanosensors

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Abstract: This paper presents a study of the chemical nanosensors (CNS) for space and environmental applications, safety alert devices, etc. The high-resolution nanosensors are applied to detect the rocket fuel hydrazine leak. The CNS detects changes in the electrical conductivity response during the chemical species presence. When the hydrazine is leaked into air, it immediately dissociates into NO₂. As a result, we are actually detecting the NO₂ gas in the trace amount from the fuel leakage. In more detail, we will discuss the sensor chips preparation and process control in terms of the resistance range control while depositing the nanomaterials on the sensors. Furthermore, there will be detailed studies of the CNS response to the dry NO₂ in the ambient conditions. The inter-digitized electrode sensors are characterized to the variables of NO₂ concentration and nanomaterials. *Copyright © 2007 IFSA.*

Keywords: Chemical nano sensors, 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 space and environmental applications. For example, we can apply CNS to detect the electrical signal during the chemical species presence.

Fig. 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 further data processing.

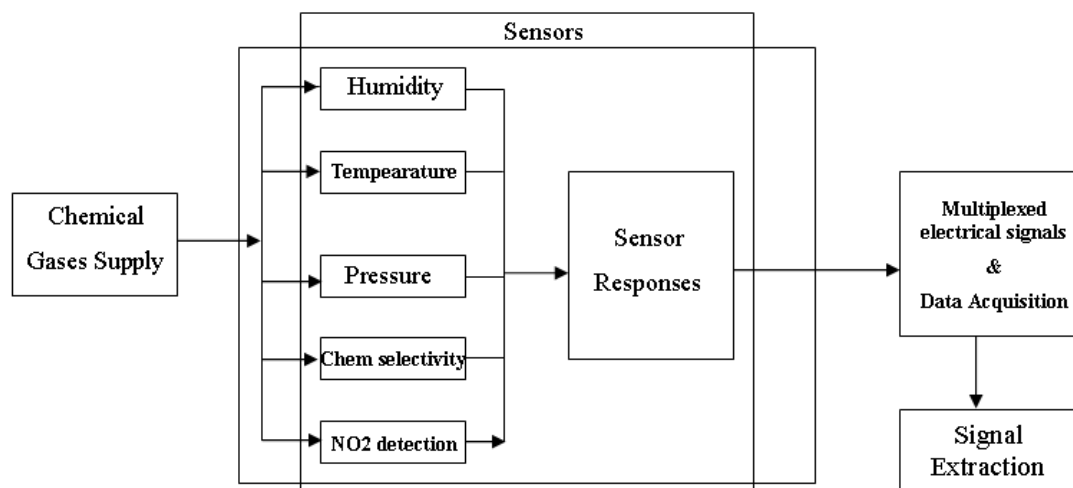


Fig.1. The schematic shows the conceptual CNS NO₂ experiments.

The purpose of our CNS project is to monitor the trace amount of NO₂ composed from the leakage of one fuel component, hydrazine. The liquid hydrazine (N₂H₄) is an efficient rocket propellant. When the N₂H₄ is leaked into air, it immediately dissociates and produces NO₂.

As published in previous literature [1], carbon nanotubes (CNT) is very sensitive to NO₂ 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 NO₂ response in relationship to various NO₂ concentrations. In terms of the dry NO₂ 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, including - 1) CGNT, 2) CGNT+MPC, 3) CGNT+polymer. The nanomaterials were discussed in Section 2.2, where CGNT is the CVD grown nanotubes. The MPC is monolayer-protected gold clusters (MPC). The polymer is cellulose hydroxypropyl.

We employ the carbon nanotubes (CNT) as the base nanomaterials in the form of the CVD growth. Illustration of Fig. 2 procedures is aimed at preparing a sensor chip before its sensor application.

2. Sensor Studies and Optimization

2.1. IV Characterization

The IV characteristics of several nanomaterials are studied in the voltage windows and in the reversal voltage as well. Current and voltage characteristics are measured with a semiconductor parameter analyzer.

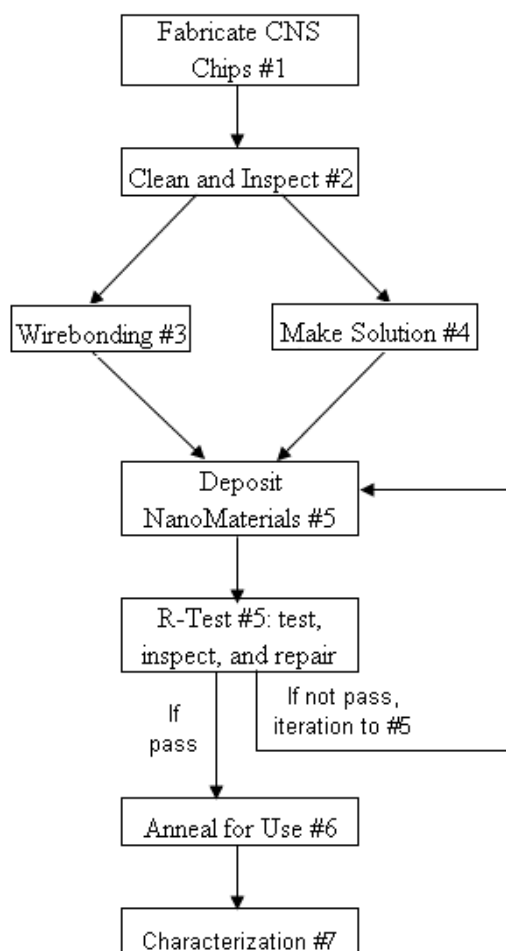


Fig. 2. The flow chart shows preparation procedures of the clean CNS chip.

We conducted the current measurement with the DC voltage sweep from -2 V to 2 V with 0.01 V increment and employed the HP4155B semiconductor parameter analyzer. Some typical IV curves are shown in Fig. 3. For example, we investigated the IV sweep curves from typical sensors with a SWNT/MPC nanomaterial (on the left), a SWNT/cellulose nanomaterial (at middle), and the cast-SWNT nanosensors (on the right). We have plotted a variety of IV curves in Fig. 3 (a), 3 (b), and 3 (c) for the $4\ \mu\text{m}$ feature gap of three typical interdigitated electrode (IDE) 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 studied the bias effects by applying different DC bias values. As stated at above, the nanosensors with different nanomaterials show different IV electrical response. The electrical resistance of the CNS may be nonlinear. Therefore, we chose several different dc-bias voltages to measure the sensor response curve at various NO_2 concentrations (Fig.4).

When the gas flow is the pure air, we only detect the baseline without any 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.

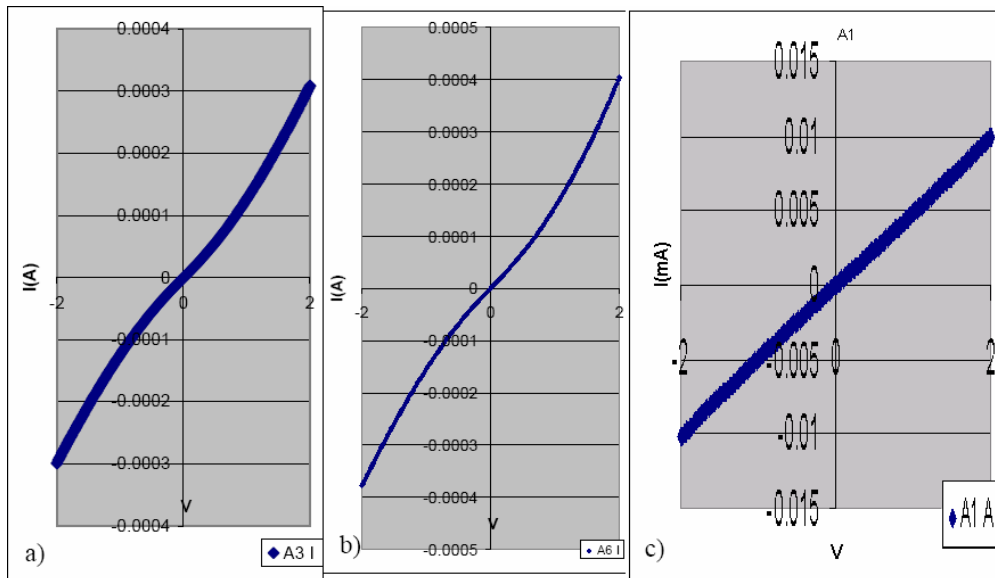


Fig.3. The IV curves of the nanosensors are characterized for three nanomaterials. (a) CNT/MPC, (b) CNT/polymer, (c) CNT.

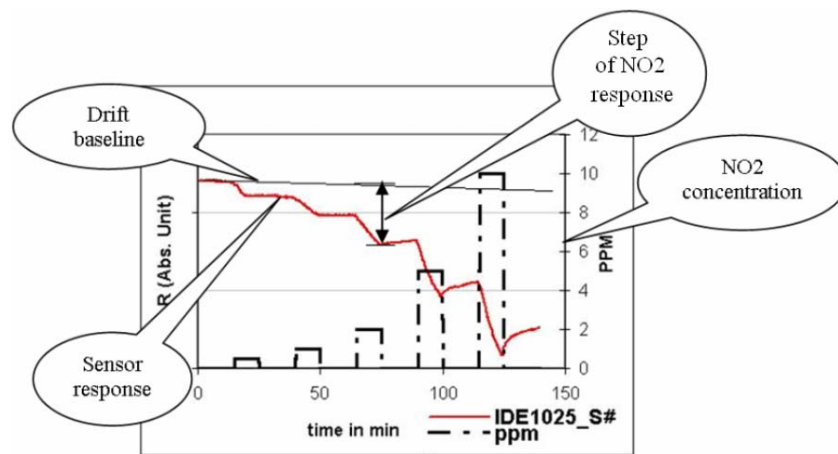


Fig.4. Typical data trace of the CNS response to the dry NO₂ chemical at various concentrations. The influence of the various environmental factors will be discussed later.

After having enough purge time, go back and iterate the exposure-purge steps until the sensing process finish. Here is a typical recipe where different gas flow is sequentially applied to the sensor:

15' Air → 10' Exposing_x (x = 0.5-, 1-, 2-, 5-, or 10-ppm) → 15' Air... where the time unit is minutes, and the unit of exposure concentration is ppm or parts per million. A typical recipe may contain many exposure-purge cycles by iterating through a given set of concentrations.

Fig. 5 is a typical KAC31 run, where the chip KAC31 is a sensor chip with nanomaterials of CGNT-only, CGNT and MPC composite, and CGNT and polymer composite, respectively. Sixteen sensors are shown to have strong response. We analyzed 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) Using linear regression method, extrapolate the baseline as a function of time;
- 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 Fig. 3.2a, 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 Fig. 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 1. Tabulated is a typical recipe of the dry NO_2 chemical sampling. The sensors chip is conditioned and tested by this recipe. The standard flow rate in total is 400CCM for the most tests. Note that every concentration is diluted by pure air.

Step number	1	2	3	4	5	6	7	8	9	10	11
Mode (F-flow, C-concentration)	F	C	F	C	F	C	F	C	F	C	F
Time (minutes)	15'	10'	15'	10'	15'	10'	15'	10'	15'	10'	15'
NO_2 (ppm)	0	0.5	0	1	0	2	0	5	0	10	0

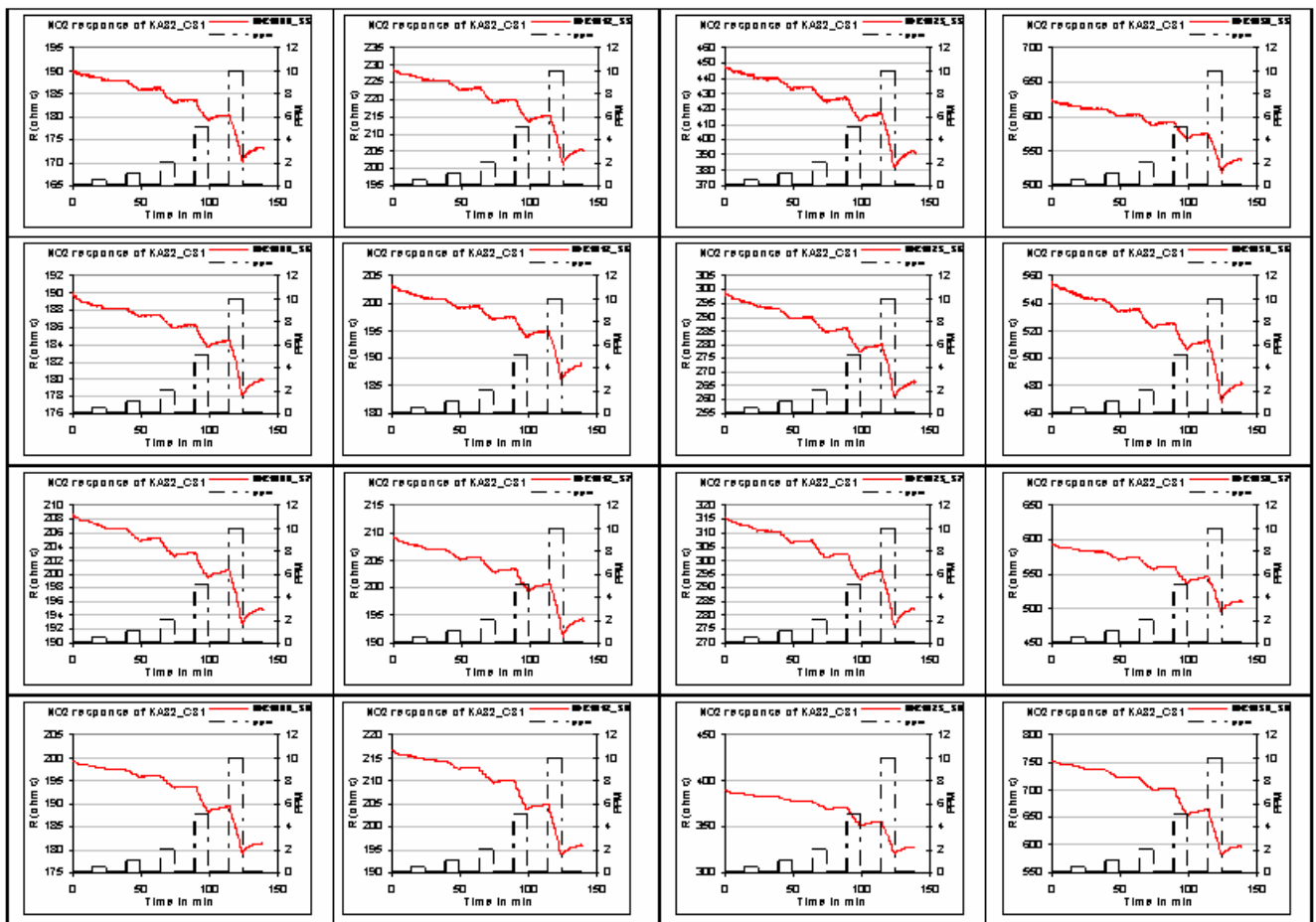


Fig.5. Every trace at above shows an individual sensor of KAC31 chip to the NO_2 chemical diluted in air at 400 CCM total flow rate and at ambient conditions. The sensors are labeled. The coordinates are at below: x-axis is the minutes of time; y-axis at left is the electrical signal; the secondary y-axis at right indicates NO_2 levels at 0-, 0.5-, 1-, 2-, 5-, 10-ppm concentrations.

Furthermore, the analysis yields the dR and dR/R_0 dependence upon NO_2 concentration. By extracting the dR/noise ratio for every sensor at all concentrations, we calculate the sensitivity function and plot this function in Fig. 5 versus both the sensor and the NO_2 concentration.

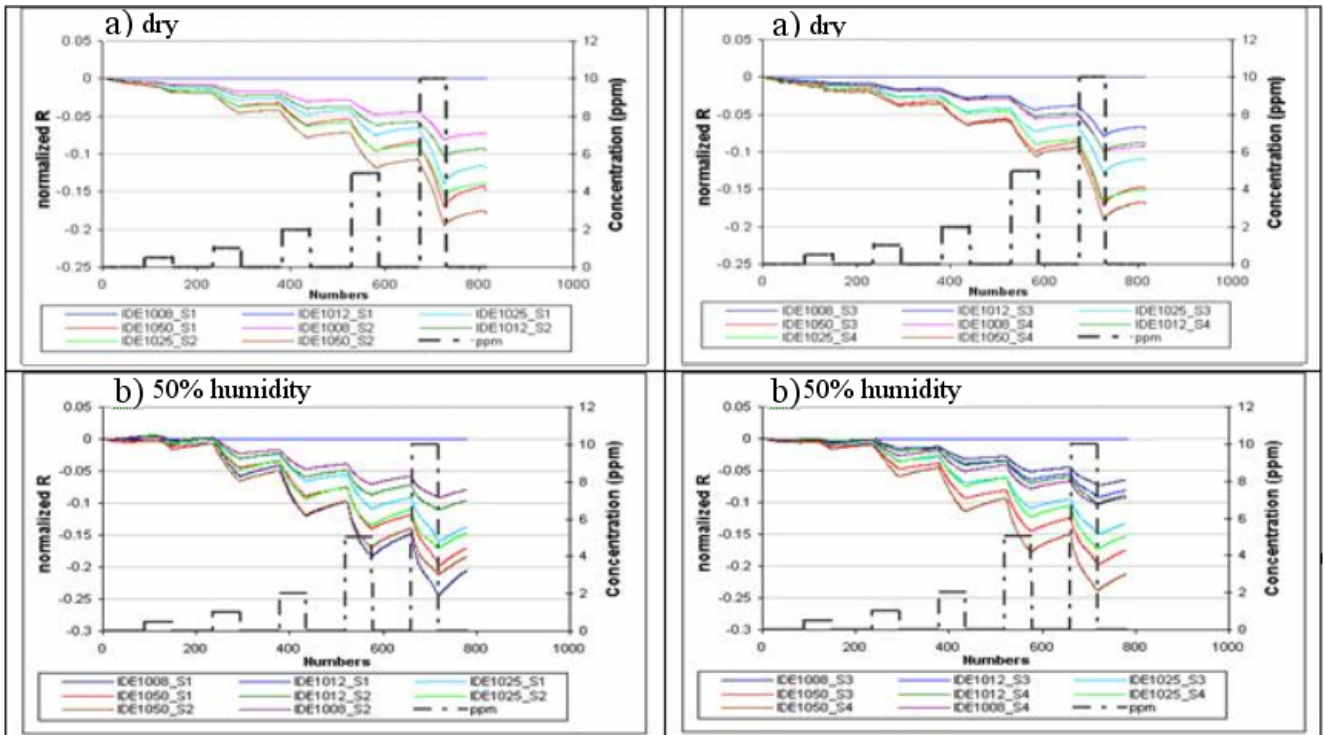


Fig.6. These charts show the relative response traces of the KAC31 chip with two RH levels: (a) 0% or dry, (b) 50%. The NO_2 concentration varies as shown at 0.5-, 1-, 2-, 5-, and 10-ppm.

We have studied the sensor's responses at various humidity values. The humidity tests were set up with the CNS characterization system. The humidifier was calibrated by the factory. 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 small effects on the nanosensor response. The NO_2 response increases with the increasing at a humidity level of 50 % and greater.

3. Conclusion

In summary, the CNS has significantly high resolution. We have studied the trace concentration of NO_2 in sub 1-ppm regime. Further studies are in the progress to characterize the life-expectance of CNS and the effect of temperature, pressure, etc.

Acknowledgment

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Guide for Contributors

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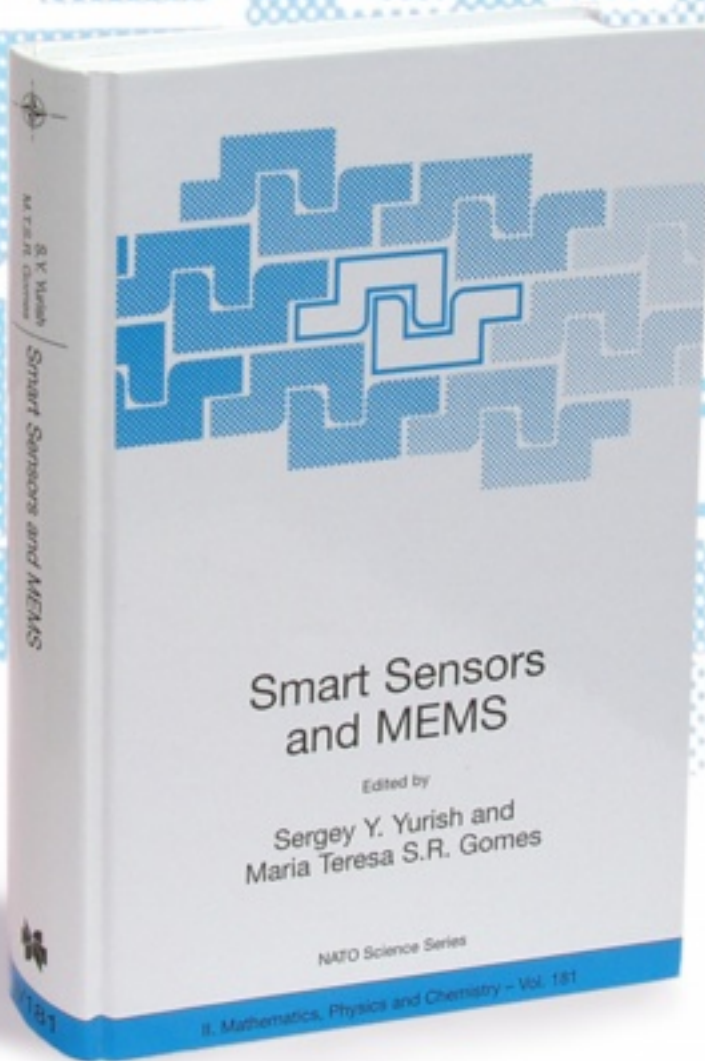
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