A Variable-Potential Amperometric Hydrocarbon Sensor

Masoud Aliramezani, Charles Robert Koch, and Ron Patrick

***Abstract*— Using the understanding of an inexpensive production NO_x sensor, the operating parameters are changed to enable hydrocarbon measurement using the same sensor. A limiting-current-type amperometric hydrocarbon sensor for rich conditions (in the absence of O_2) is developed in this work. To do this, an inexpensive three-chamber amperometric sensor with three separate electrochemical cells is parameterized to measure propane concentration. The sensor is tested using a controlled sensor test rig at different propane concentrations. The inputs to the sensor electrochemical cells have been modified to determine the best HC measurement parameters (HCMPs) for measuring propane at different concentrations. First, the transient performance and stability of the sensor are optimized by changing the sensor temperature, the reference cell potential, and the stabilizing cell potential at a high propane concentration (5000 ppm - balanced with nitrogen). Over the range tested, the sensor has the longest stable output duration at the temperature of 1009 K, the reference cell potential of 0.67 V and the stabilizing cell potential of 0.45 V. Using these sensor inputs for sensor temperature, reference cell potential and stabilizing cell potential, the sensor steady state behavior is studied to find the diffusion-rate-determined operating region. The sensor is shown to have a linear sensitivity to propane concentration from 0 to 3200 ppm. Finally, the sensor response time to different step changes from 0 up to 5000 ppm propane concentration are studied. It is shown that propane stepsize does not have a significant effect on the sensor response time. Consequently, using the working principles of an existing production amperometric NO_x sensor and changing the sensor operating parameters, an amperometric hydrocarbon sensor that works in diffusion rate determining operating region is developed.**

***Index Terms*— Gas sensor, hydrocarbon sensor, emission measurement, amperometric sensor, propane.**

I. Introduction

AMPEROMETRIC gas sensors (AGS)s belong to a promising group of electrochemical gas sensors that play a pivotal role in a wide range of industrial applications, including the automotive industry [1], [2], medical [3], [4], and environmental monitoring [5], [6]. In an AGS, the sensor output signal is equal to the current generated by oxidation or reduction of species over the electrode-electrolyte interface. This current is typically measured at a fixed cell potential to obtain stable current value [7], [8]. Variable-potential AGS are also used in some applications such as wide-band O_2 sensors [9] to increase the sensor operating range.

The AGS current signal generally depends on the sensing cell potential, sensor temperature and the concentration of the measuring species [10], [11]. When the sensor temperature and the potential of an AGS sensing cell is set high enough, then the reaction rate in the cell becomes much faster than diffusion rate of species through the sensor diffusion barriers. In this case, the sensor current output is limited only by the diffusion of species. These sensors are called limiting-current type amperometric sensors. Limiting-current-type AGSs have much higher resolution than other types of electrochemical sensors and do not depend on chemical equilibrium at the electrode/electrolyte interface [12], [13].

Defining the appropriate value of sensing cell voltage is essential to achieve the most stable sensor response. On the one hand, the sensing cell potential must be high enough to keep the reaction rates within the diffusion-rate-determining region [14], otherwise the sensor output signal will not be proportional to the concentration of the measuring species reducing the sensor accuracy. Conversely, if the sensing cell voltage is too high, electrolyte decomposition takes place [15] which will not only reduce the sensor accuracy but can also damage the sensor. The value of optimal cell potential is also affected by manufacturing tolerances and sensor aging.

These drawbacks of fixed potential amperometric sensors can be solved by implementing a variable-potential sensing cell. Implementing a variable-potential sensing cell facilitates controlling the partial pressure of the measuring gas species inside the sensing cell using a reference cell [16]. In this case, the sensing cell potential is changed to keep the reference cell potential at a constant value [17]. This is used in wide band lambda sensors where the oxygen concentration in the engine exhaust system is compared to the stoichiometric condition represented by the reference chamber set-point potential [18].

In this work, a limiting-current-type hydrocarbon sensor is developed to measure propane over a wide range propane concentration in a rich (no oxygen) environment. The variable potential approach, which has not been used so far for amperometric HC sensors, ensures adjustment of the sensing cell potential to extend the diffusion rate determining (DRD) operating region. Unlike the other types of electrochemical HC sensors [8], [19], [20], the output of the developed sensor is linearly dependant on the concentration of hydrocarbons thanks to the extended DRD operation. In addition, the HC

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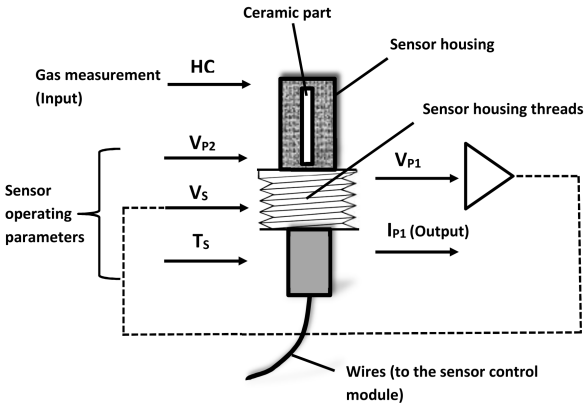
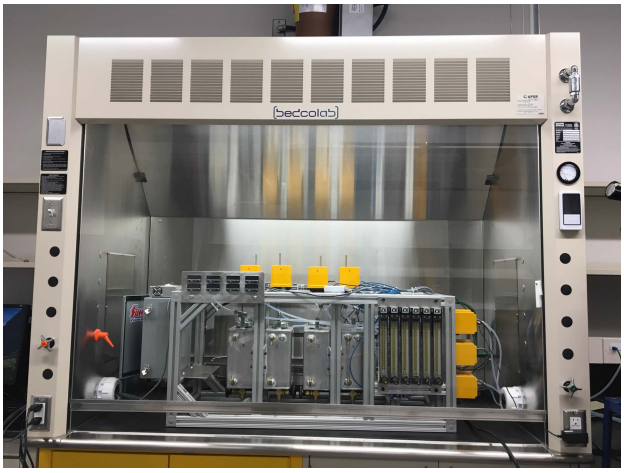


(a) Amperometric HC Sensor operating parameters and input-output schematic



(b) Sensor test rig setup in fume hood

Fig. 1. HC sensor schematic and the sensor test rig setup.

sensor is developed by only changing the parameters of an existing production NO_x sensor which allows the use of a low cost readily available commercial sensor. A low cost mass produced amperometric O_2-NO_x sensor (Bosch PN: 0281006643) is used as the base sensor and then the operating conditions are modified to measure propane concentration. This inexpensive production sensor is widely used in the automotive industry to reduce diesel engine emissions [2]. The main objective of this study is to develop an amperometric HC sensor based on a production electrochemical sensor. The number of electrochemical cells of this production O_2-NO_x sensor (three cells) provides a sufficient number of degrees of freedom to use the sensor to measure another species by only changing the sensor operating parameters. All of the main electrochemical inputs of the sensor have been modified to improve the sensor performance for measuring propane at different concentrations. The three electrochemical sensor cells in this case are: HC sensing, reference and stabilizing. The effect of sensor temperature (controlled by a closed loop heater), the reference cell potential, and the stabilizing cell potential on sensor performance are first studied to improve the transient performance and stability of the sensor. The sensor inputs and outputs are shown schematically in Fig. 1a and the sensor test rig is shown in Fig. 1b. Next, the sensor steady state behavior is experimentally investigated to find the diffusion-rate-determined operating region. Finally, the sensor transient response is studied for step changes in propane concentration.

II. Amperometric Sensor for HC Measurement

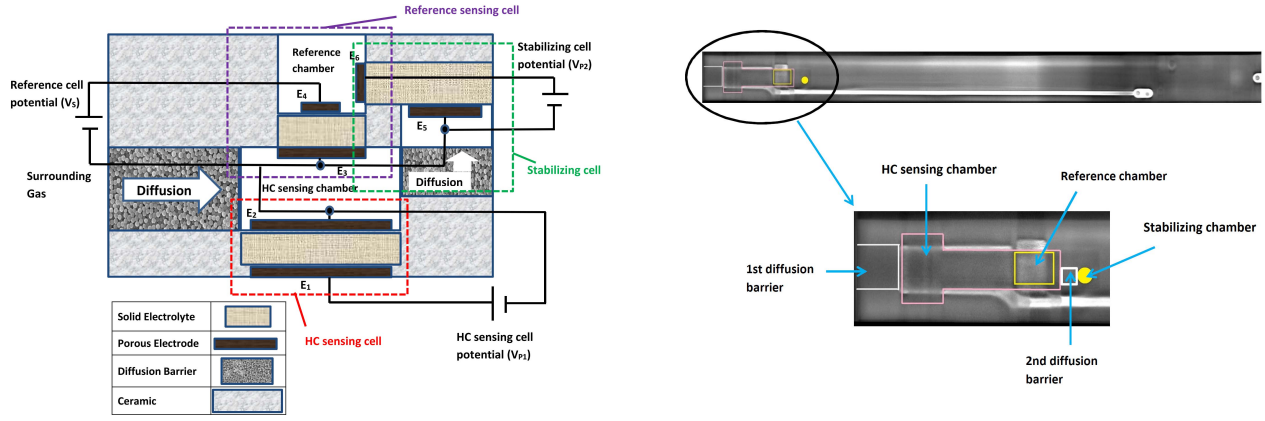
A conventional NO_x sensor has been used as the base sensor and modified to measure propane at different concentrations. The sensor, shown schematically in Fig. 2a, consists of three electrochemical cells: the HC sensing cell, the reference cell and the stabilizing cell. An X-ray image of the sensor is shown in Fig. 2b. The sensor inputs are set to develop the capability of a limiting-current type amperometric HC sensor. In the limiting-current operating region, HC oxidizes in the first chamber and produces a cell current proportional to HC concentration. Higher resolution and easier calibration of this sensor are the main advantages in comparison with the other amperometric HC sensors [7], [8], [21].

The HC sensing cell is a variable-potential electrochemical cell. An internal controller adjusts the HC sensing cell potential (V_{P1}) to keep the reference cell potential (V_S) at a constant value. V_S corresponds to the potential difference between the HC sensing cell electrode (E3) and the reference cell electrode (E4). When the HC concentration increases in the surrounding gas and therefore diffuses into the first chamber, then the HC sensing cell voltage decreases to oxidize all the HC inside the first chamber and therefore to keep the reference cell potential at a constant value. It will be experimentally shown that a positive fixed value of the stabilizing cell potential increases the sensor stability at high hydrocarbon concentrations. The best value of the reference cell potential and the stabilizing cell potential have been determined experimentally as explained in the next section.

III. Experimental Setup

A custom built sensor test rig, shown in Fig. 1b, was developed and used to test the sensor at different propane concentrations. The test rig consists of six externally-controlled 2-way valves, three externally-controlled 3-way valves, four humidifying tanks and six CCR MKS-GE50A mass flow controllers, connected to different known gas cylinders. Tube heaters are installed to keep the gas mixture at the desired temperature and to avoid any undesired water condensation. More details about the sensor test rig are available in [22].

All of the test rig actuators are externally controlled via an ethernet interface to a computer PC. Custom MATLAB code is used to control the sensor test rig actuators and to communicate with the mass flow controllers. The test rig is connected to the computer via an Arduino interface. The sensor test rig setup is schematically shown in Fig. 3. In this work, only propane and nitrogen cylinders are used and the water tanks are all bypassed.



(a) HC Sensor Schematic (b) X-ray tomography of the sensor ceramic (top view)

Fig. 2. Inside the HC sensor ceramic part.

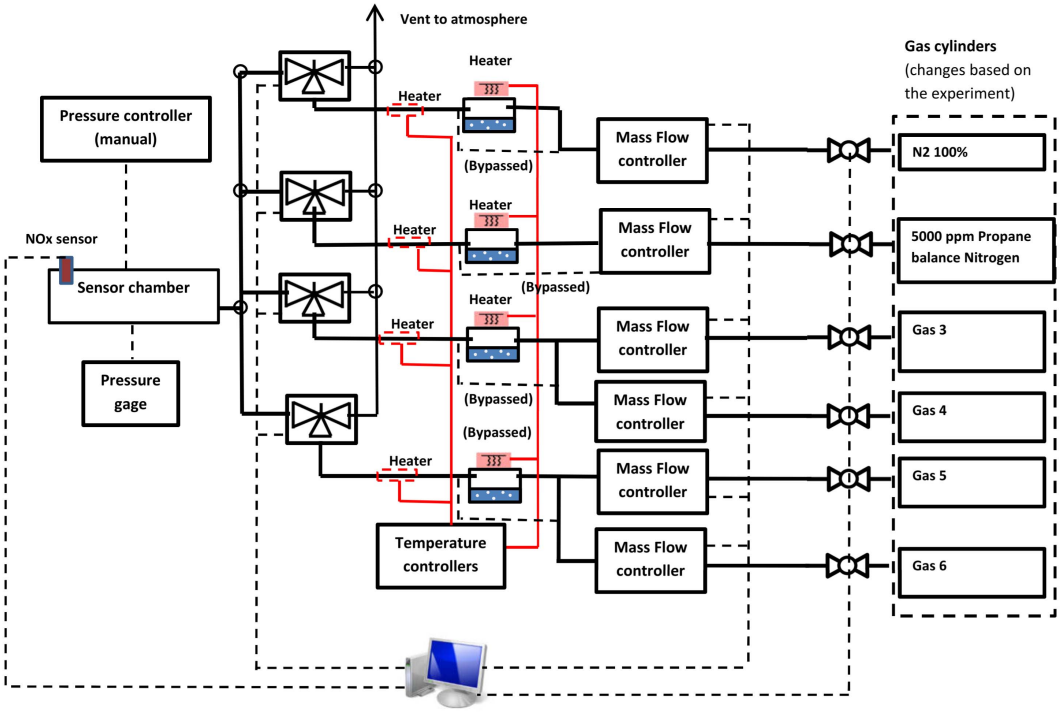


Fig. 3. Sensor test rig schematic.

The sensor operating parameters, including the sensor temperature; the reference sensing cell potential and the NO_x sensing cell potential, are modified using an aftermarket sensor control module (ECM 02-07) [23]. This module is connected to a computer via a Controller Area Network (CAN bus) interface.

IV. Determining HC Measurement Parameters (HCMPs)

The sensor temperature, T_S, the reference potential, V_S and the stabilizing cell potential, V_{P2}, are the main inputs that affect the sensor performance by affecting the diffusion of HC (propane) through the sensor as well as the oxidation rate of HC over the sensing electrode. The main objective of this optimization process is investigating the effect of these factors on sensitivity and stability of the sensor.

To achieve high resolution and accuracy, the sensor should work in Diffusion Rate Determining (DRD) conditions. To keep the sensor at the DRD condition, the diffusion rate of the measuring species through the sensor diffusion barrier should be lower than the reaction rate of the species on the sensing electrode. The sensor inputs (shown in Fig. 1a) are modified to extend the working range of the sensor by expanding the DRD operating region.

The reaction rate of species over the sensing electrode is a function of sensor temperature and the cell potential [24], [25]. The diffusion rate directly varies with the sensor temperature, the concentration of species in the surrounding gas and the concentration of species inside the sensor chambers [11], [24] according to the equations explained in section V-B.

The diffusion rate increases with increasing the hydrocarbon concentration in the surrounding gas. If the hydrocarbon concentration goes higher than a certain high value, the diffusion

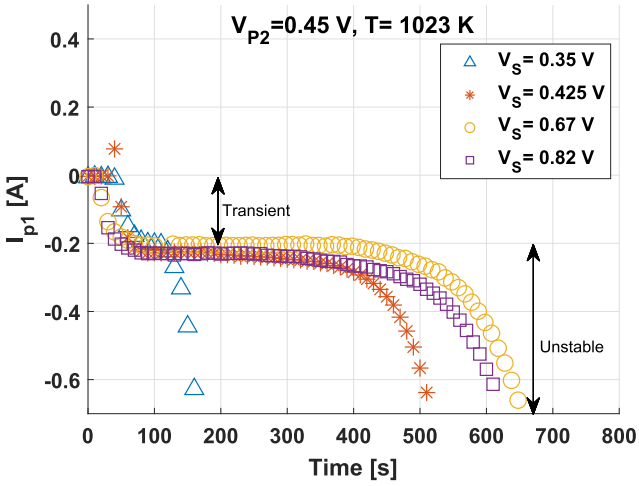


Fig. 4. The effect of reference cell potential on the transient behavior of the sensor output current at HC (propane) concentration = 5000 ppm.

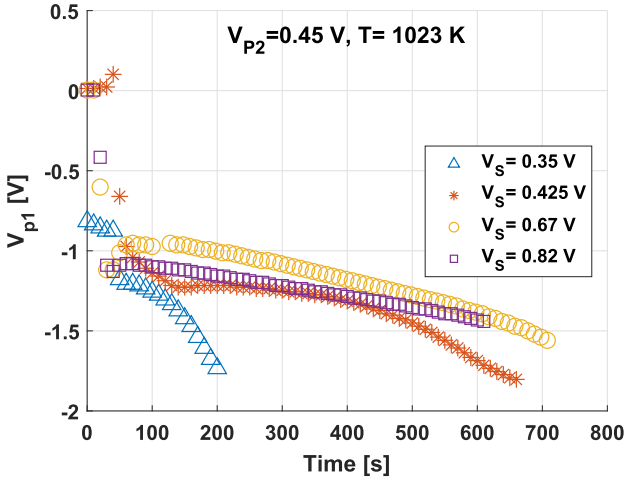


Fig. 5. The effect of reference cell potential on the transient behavior of the sensing cell potential at HC (propane) concentration = 5000 ppm.

rate becomes higher than the reaction rate and the sensor does not operate at DRD. Then, not all the HC can be oxidized inside the sensing cell since the HC oxidation rate is not as high as the diffusion rate of HC into the sensing chamber. The sensor behavior becomes unstable above this value due to the unbalanced diffusion-reaction rate and accumulation of the HC in the sensing cell.

The sensor is exposed to a high value (here, 5000 ppm) propane mixture balanced with nitrogen. This concentration is too high for the sensor to operate at diffusion limiting condition. Instead, the sensor has rate-determining operation. Then, the sensor inputs are changed to maximize the time duration of stable operation of the sensor. These specified sensor inputs are then used to study the sensor behavior at a DRD operating condition.

A. The Effect of Reference Cell Potential

The reference cell potential represents a set point for species concentration in the sensing chamber. For instance, in a wide-band oxygen sensor, the reference cell potential represents the oxygen concentration for stoichiometric combustion (zero oxygen concentration and zero unburned hydrocarbon concentration). For the amperometric HC sensor studied in this work, the reference cell (V_S) potential remains constant during sensor operation using a feedback controller by adjusting the HC sensing cell potential (V_{P1}). Once the reference potential is set, a closed loop controller adjusts the sensing cell potential and current to maintain the partial pressure of the oxidized species at the reference value.

The effect of reference cell potential on the sensor transient behavior is experimentally tested and the results are shown in Fig. 4 and Fig. 5. The sensor is first exposed to pure nitrogen (initial condition) and then to 5000 ppm propane diluted with nitrogen. The 5000 ppm propane is high enough to put the sensor in reaction rate determining operation. Then first objective is to maximize the duration of stable sensor current output before it starts dropping due to accumulation of non-oxidized HCs inside the first chamber and consequently dropping HC sensing cell voltage. Maximizing the stable-output duration at such a high concentration, ensures stable behavior at lower concentrations and therefore, increases the sensor operating range.

Stable-output duration is experimentally found to increase with the reference cell potential. The corresponding sensing cell potential generally increases with the reference potential as shown in Fig. 5 and therefore postpones a V_{P1} drop and postpones sensor unstable behavior. However, increasing the reference cell potential to 0.82 V has an opposite effect on the output stability. This is attributed to electrode decomposition that takes place at high cell potentials [15].

B. The Effect of Temperature

The sensor temperature has a more complicated effect on the sensor behavior as it affects both the diffusion of species through the sensor diffusion barriers (explained in section V-B) and the reaction rate over the electrode as follows [26]:

$$V_{P1} = E^o + \frac{\Delta S_{ox-red}}{nF}(T_s - T_o) - \frac{\bar{R}T}{nF} ln\left(\frac{\prod_{i=1}^{k_1}(x_{i,anode})^{y_{1i}}}{\prod_{j=1}^{k_2}(x_{j,cathode})^{y_{2j}}}\right) - \eta_a \quad (1)$$

where, $\bar{R}$ is the universal gas constant, F is the Faraday constant, T_s is the sensor temperature (in Kelvin), T_o is the reference temperature, ΔS_{ox-red} is the change in entropy of products-reactants at the operating temperature and E^o is the open-circuit potential at the standard state (1 atm; 298.15 K) while $x_{i,anode}$ and $x_{j,cathode}$ are the molar fraction of species around the anode and the cathode respectively. The parameters y_{1i} and y_{2j} and n depend on the species involved in the electrochemical reaction [26].

Therefore, the effect of temperature on the sensor output stability may vary for different operating conditions. The effect of sensor temperature on sensor transient behavior at three different reference potentials is shown in Fig. 6 and 7. The reaction rate and the diffusion rate both increase with the

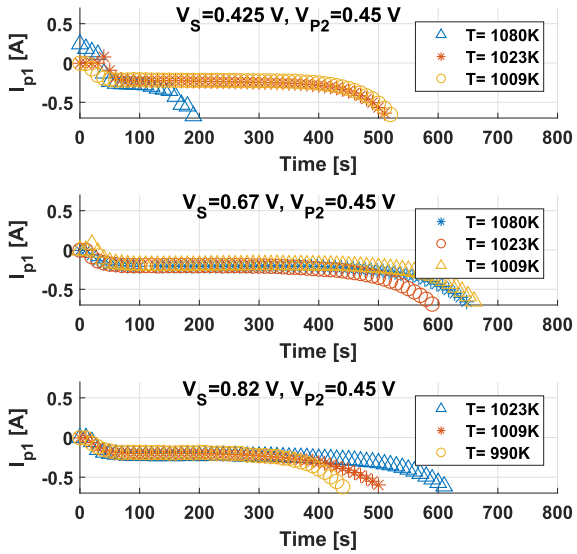


Fig. 6. The effect of temperature on the transient behavior of the sensor output current at HC (propane) concentration = 5000 ppm.

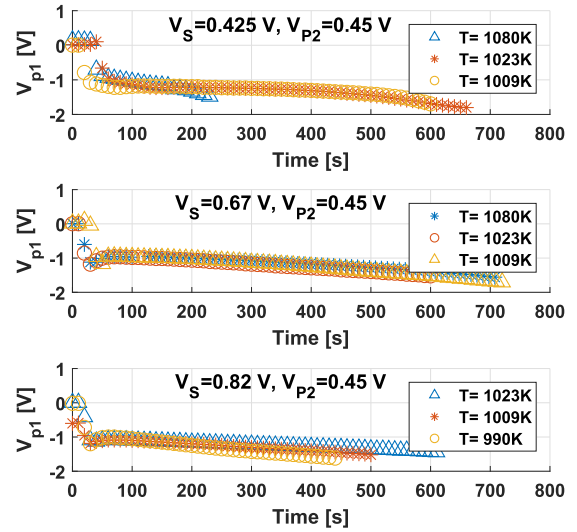


Fig. 7. The effect of temperature on the transient behavior of the sensing cell potential at HC (propane) concentration = 5000 ppm.

sensor temperature [1], [15]. This two-sided effect can either reduce or increase the stable-output duration, depending on the operating condition.

According to the experimental results, at $V_S = 0.45$ V, the stable-output duration decreases dramatically when the sensor temperature increases from $T_S = 1023$ K to 1080 K while at $V_S = 0.82$ V, the stable-output duration increases as the temperature increases. For 1009 K $\leq T_S \leq$ 1080 K and 0.35 V $\leq V_S \leq$ 0.82 V, the most stable sensor performance is experimentally found at $T_S = 1009$ K and $V_S = 0.672$ V. Stabilizing is defined as the time it takes for I_{P2} to drop to 10% lower than its stable response value for a step input of propane.

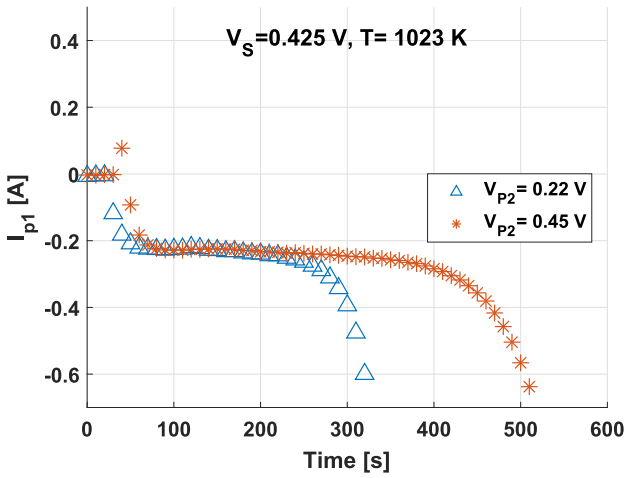


Fig. 8. The effect of second sensing cell potential on the transient behavior of the sensor output current at HC (propane) concentration = 5000 ppm.

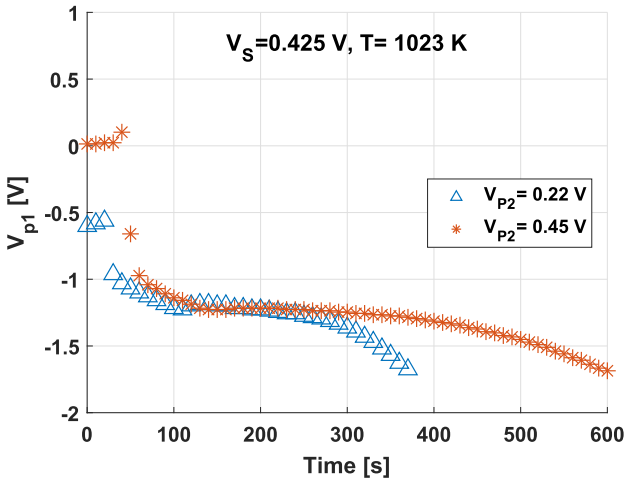


Fig. 9. The effect of second sensing cell potential on the transient behavior of the sensing cell potential at HC (propane) concentration = 5000 ppm.

C. The Effect of Stabilizing Cell Potential

Compared to the HC sensing cell that has a Platinum-Gold (Pt-Au) electrode, the stabilizing cell has a Platinum (Pt) electrode which has a lower activation energy for reducing species inside the stabilizing cell chamber. This significantly affects the gradient of species concentration through all of the sensor chambers and barriers [1] and changes the sensor output. To find the best value of the stabilizing cell potential (V_{P2}), the transient behavior is examined at two different cell potentials, $V_{P2} = 0.22$ V and $V_{P2} = 0.45$ V. The sensor is tested at two stabilizing cell potentials of 0.22 V and 0.45 V and the results are shown in Fig. 8 and 9. The potential $V_{P2} = 0.45$ V is used as it is typically high enough to reduce any combustion engine productions, such as NO_x, that get into the stabilizing chamber [1].

At HC = 5000 ppm in the surrounding gas, the stable-output duration is experimentally found to increase as the stabilizing cell potential increases. The stabilizing cell potential is set to $V_{P2} \leq 0.45$ V to avoid electrolyte decomposition.

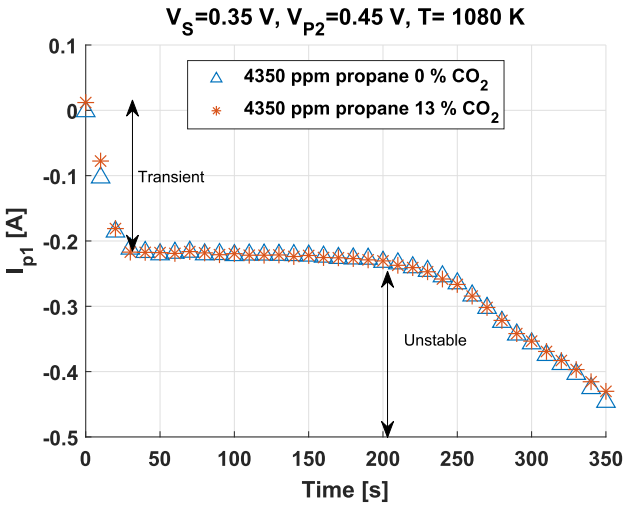


Fig. 10. The effect of presence of CO_2 on the transient behavior of the sensing cell current.

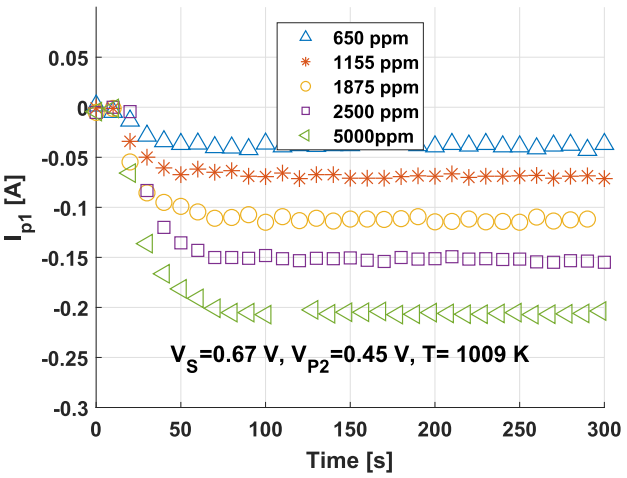


Fig. 11. The effect of propane concentration on the transient behavior of sensor output current at propane concentration = 5000 ppm.

D. The Effect of CO_2 Presence

The focus of this work is mainly on measuring hydrocarbon concentration in rich condition which is applicable to combustion emission study in the absence of oxygen. All the above tests were conducted in the absence of any oxygen containing species as the propane mixture was diluted with pure nitrogen. To make sure other typical combustion products don't interfere with the sensor response, the effect of presence of CO_2 on the sensor response is examined and the results are shown in Fig. 10. To study the sensor transient behavior in the presence of an oxygen containing specie, CO_2 is used. The choice of CO_2 is based on that it is a hydrocarbon combustion product. Testing the sensor with other species is out of the scope of this work and was not conducted. Now, the sensor is exposed to the same propane concentration (4350 ppm) without and with CO_2, with CO_2 concentration = 13 % setting V_S = 0.35 V, V_{P2} = 0.45 V and the sensor temperature equal to 1080 K. For both of the tests, the sensor is initially exposed to the N_2-CO_2 mixture with no propane and then to 4350 ppm propane concentration. The transient results are shown in Fig. 10. The presence of CO_2 does not have a significant effect on the sensor transient behavior and does not have an effect on the steady state sensor behavior.

V. Steady State Sensor Behavior

A. The Effect of Propane Concentration

After maximizing the stable-output duration, the sensor sensitivity to propane (diluted with N_2) is evaluated at the HCMPs (V_S = 0.67 V, V_{p2} = 0.45 V, T = 1009 K). The sensor stable response at different propane concentrations has been studied to find the sensor sensitivity to propane and to find the DRD operating region. The test results at five different propane concentrations are shown in Fig. 11. The steady state sensor output magnitude, decreases with the propane concentration. This is due to the lower diffusion rate of propane through the sensor diffusion barrier and therefore the lower oxidation rate of propane over the sensing electrode. This sensitivity of the sensor output to propane can be used to measure propane concentration. However, to develop an accurate and reliable sensor, the DRD operating region should first be determined. This procedure is explained next.

B. Diffusion Rate-Determining (DRD) Operating Region

The HC sensing cell pumping current is proportional to the molar flux of propane through the sensor diffusion barrier. According to Faraday's law [16]:

$$I_p \propto N_{C_3H_8} \tag{2}$$

where, $N_{C_3H_8}$ is the molar flux of propane through the first diffusion barrier to the HC sensing chamber.

Assuming negligible convection inside the cell and perfect dilution of propane in N_2, the molar flux of propane ($N_{C_3H_8}$) can be estimated using Fick's law [27]:

$$N_{C_3H_8} = -C_t D_{C_3H_8} A_{D1} \frac{x_{C_3H_8,sur} - x_{C_3H_8,senc}}{L_{D1}} \tag{3}$$

where, $N_{C_3H_8}$, C_t, $x_{C_3H_8,sur}$ and $x_{C_3H_8,sur}$ are the molar flux, total concentration and the mole fraction of propane in the surrounding gas and inside the sensing chamber respectively while A_{D1} is the diffusion barrier cross sectional area, L_{D1} is the diffusion barrier length and $D_{C_3H_8}$ is the diffusion coefficient of propane through the porous diffusion.

For DRD operating condition, propane oxidizes with a higher rate than it diffuses into the sensing chamber. Thus, the molar fraction of propane decreases to a very low value ($x_{C_3H_8,senc} \approx 0$). Therefore, based on Eqn. (2) and (3), the sensor current output becomes linearly dependant on propane concentration in the surrounding gas. Based on Eqn. (2) and Eqn. (3), the sensor response at zero propane concentration is equal to zero. The linear behavior of the sensor as well as the zero response at zero propane concentration, reveals that within the DRD operating region, sensor calibration is quite simple and only needs one calibration point to obtain the slope. In addition, the slope of the sensor response vs propane concentration only varies if the dimensions of sensor diffusion

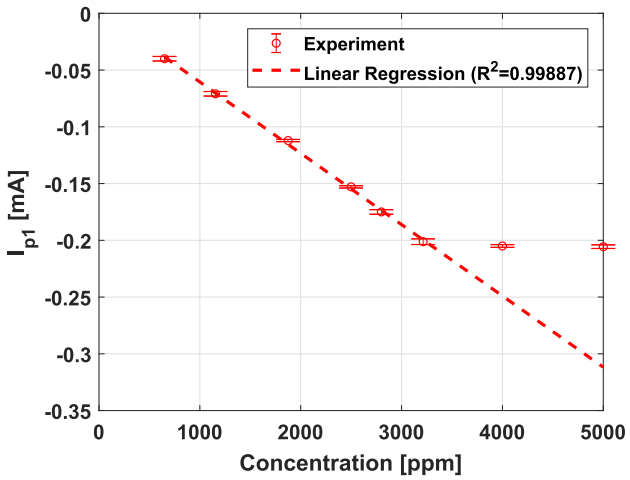


Fig. 12. The sensor output current vs propane concentration. $V_S = 0.67$ V, T = 1009 K, $V_{P2} = 0.45$ V.

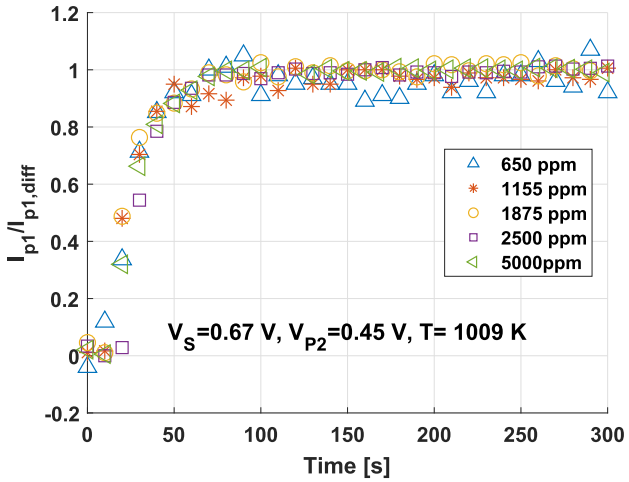


Fig. 13. Transient behavior of the normalized sensor current at different propane concentrations.

barrier change. Therefore, the sensor calibration should not change significantly between different production sensors with the same design. This is one of the main advantages of the developed HC sensor.

In order to define the DRD operating region and the corresponding sensor linear operation band, the sensor output current vs propane concentration is shown in Fig. 12. The sensor output current magnitude increases linearly with propane concentration up to approximately 3200 ppm propane concentration as shown in Fig. 12. The sensor output current then plateaus at propane concentrations higher than 3200 ppm. The sensor is working as DRD for propane concentration below 3200 ppm and for higher propane concentration, the sensor operates at reaction rate determining condition. The test was run three times to understand the experimental uncertainty and the error bars represent this variation.

VI. Sensor Response Time

Sensor response time is used to capture the transient behavior of a system. A better understanding of the sensor transient behavior, is useful to determine appropriate applications for the sensor.

A normalized variable is defined by dividing the transient sensing cell current by the steady state cell current at the same propane concentration ($I_{P1,diff}$). The transient behavior of the normalized sensing cell current is shown in Fig. 13. The results show that the size of the propane step concentration does not have a significant effect on the transient behavior with the response time as shown in Fig. 13.

The step-response characteristics of the sensor is studied considering all of the normalized sensor outputs and the sensor response time (10% to 90%) was found to be 38 seconds. Although this response time is shorter than many other amperometric HC sensors [7], [8], it can be significantly reduced by modifying the sensing cell potential (V_{P1}) controller since the long sensing cell potential settling time, shown in Figures 5, 7 and 9, increases the sensor response time.

VII. Conclusions

The operating parameters of a production amperometric NO_x sensor are changed to enable propane measurement by increasing the sensor sensitivity to propane and by extending the diffusion rate determining (DRD) operating range of the sensor. As a result, a diffusion rate determining amperometric hydrocarbon has been developed with a response that is linearly dependant on hydrocarbon concentration. First, the sensor stability is maximized for purpose of measurement by changing the sensor inputs including the sensor temperature, the reference cell potential, and the stabilizing cell potential. Stability is defined as the time it takes for I_{P2} to drop to 10% lower than its stable response value. Then stability tests are carried out on a controlled sensor test rig at a reaction rate determining operating condition with propane concentration of 5000 ppm (balanced with nitrogen). Based on the experiments, the sensor has a stability of 530 seconds at sensor temperature of 1009 K, a reference cell potential of 0.67 V and a stabilizing cell potential of 0.45 V. This condition is then fixed for subsequent sensor operating condition. Further experimental testing shows that the presence of CO_2 does not have a significant effect on the sensor sensitivity to propane or the transient behavior.

To find the diffusion-rate-determined operating region, the linearity of sensor steady state response vs propane concentration is experimentally studied at the stable operating condition. A linear sensitivity to propane concentration from 0 to 3200 ppm reveals the DRD operating region of the sensor.

To study the sensor transient behavior in DRD operating region, the sensor response time is examined for step changes from zero propane concentration to several propane concentrations using the sensor test rig. It is shown that the sensor response time is almost the same for different size step changes of propane concentration and is approximately 32 seconds. This response speed could be improved by modifying the sensing cell controller and will be a subject of future work.

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