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Detection of CO in H_2-rich gases with a Samarium doped Ceria (SDC) sensor for Fuel-cell applications

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Abstract

An original sensor has been studied in order to detected low CO concentration in H_2-rich atmosphere for PEM (Protonic Exchange Membrane) fuel cell applications. The SCD (Samarium doped Ceria) sensor is a potentiometric sensor working with an electrode dissymmetry Au/Pt. The originality of this sensor is mainly working with the two electrodes in the same atmosphere without a reference cell. The Δ (emf) response, defined by the difference between the emf (electromotive force) value under carrier gas and the emf value under CO, is correlated with the CO concentration. Experiments has been carried out on a laboratory testing bench, either with a large measurement cell equipped with a hot plate and two mobile gold points as electrical contacts or in a small cell with self-heated sensors supplied with a platinum heater on the reverse side of the substrate. Responses to low CO concentrations (0 to 4000 ppm v/v) in H_2 –rich gases (5%v/v) varies between 25 mV to 100 mV, but saturation is observed beyond 400 ppm v/v of CO. In wet atmosphere, the sensitivity is partially reduced but the sensor response remains perfectly usable. At the moment no satisfying model can be used to explain the experimental results. Nevertheless, the performances of these SCD sensors appear sufficiently good to satisfy the fuel-cell application.

Keywords:

SCD sensors, CO detection, fuel-cell, hydrogen rich gases

I Introduction:

In the field of fuel cell, it is of prime importance to be able to detect very low concentrations (below 50 ppm) of CO in pure H_2 or H_2-rich gases. The Protonic Exchange Membrane fuel cells (PEM-FC) use platinum as catalyst, especially at anode in order to dissociate hydrogen. The problem of poisoning of platinum by CO is now well known. In order to keep good performances of PEM-FC, it is necessary to avoid the presence of CO in the fuel gas. Nevertheless hydrogen is often produced by reforming process of various fuels with a contamination of CO. In consequence, it is necessary to have CO sensors. The difficulty to find adequate CO sensors for these fuel-cell applications is related to the absence of gaseous oxygen in the reformer gases.

We have a past experience to use SnO_2 sensors to detect H_2 in pure nitrogen [1]. Tin oxide sensors are generally not well adapted to work without oxygen. Moreover, the thermodynamic stability of this oxide in H_2-rich atmosphere depends on not only the temperature, but also partial pressures of oxygen, carbon dioxide and water. With SnO_2 doped with Pd, it seems that low CO concentrations can be detected below 600K (limit of stability) according to the levels of P_{H2O} and/or P_{CO2} [2]. Nevertheless, the process occurring is not clearly understood and the long term stability is not demonstrated.

Sensors using CuCl or CuBr as sensing materials have also be used with success for the detection of CO in reducing gases [3]. Nevertheless, the difficulties seem to be connected to the high influence of humidity which is a key factor in fuel cell applications.

Another solution proposed for such application is to use mini-fuel cells (PEM-FC or solid oxide fuel cell (SOFC)) and to follow the degradation of the fuel cell performances due to the presence of CO [4-6]. The difficulties are here related to the fuel cell technology because such sensors need an effective gas separation between the anode (H_2 with CO to be measured) and the cathode in a reference gas (air for example).

In regards of the state of the art, we propose to use a very simple sensor based on single chamber SOFC. We develop such types of devices from several years [7, 8]. The interest is mainly to work without a reference cell. The principle is close to a potentiometric sensor based on the difference of catalytic activities of the two electrodes. We propose in this paper to use SDC (ceria doped with samarium) as electrolyte material and gold and platinum as electrodes. The main interest is that SDC is now well used as electrolyte for SOFC, and one reason is related to its good stability under pure H_2 in a very large range of temperature. The emf signal observed in the H_2-rich carrier gas, namely the baseline, is modified when CO traces are introduced in the gaseous flow. The Δ (emf) response, defined by the difference between the emf value under carrier gas and the emf value under CO, is then correlated with the CO concentration in the reformed gas.

II Experimental:

II 1 Sensors preparation

New potentiometric sensors have been elaborated using SDC (15 mol % samarium doped ceria, $Ce_{0.85}Sm_{0.15}O_{1.925}$) as ionic conductor. It has been chosen because of its high conductivity at low temperature in regards of many ionic materials. This material is also well known in the fuel cell domain for electrolyte of SOFC. In consequence, its range of stability under hydrogen is very good. We can consider that SDC will be reduced only above 600°C in absence of oxygen.

The SDC powder is provided by Nextech Materials Corp. Its purity is controlled by XRD and the mean size of the grains is about 100 nm. Thick films of SDC are deposited on an alumina substrate by screen-printing with a semi-automatic Aurel C890 machine. The ink is made of the SDC powder mixed with an organic binder and a solvent which allow to adjust the rheological parameters of the inks. After annealing at 1200°C, the thickness of the films is about 20 – 30 µm. Finally, two electrodes, gold and platinum, with similar area (ratio Pt/Au = 1) are deposited on SDC by reactive sputtering (thickness of 0.5 µm) with a Balzers MED 010 machine. An example of this type of sensor is shown in Fig. 1. In the case of self heated sensor, the same type of substrate is used. One side of this substrate (sensing material and electrodes) is prepared as previously described; in order to heat the sensible element, the back side is then provided with a platinum heater. This platinum heating element and its electrical connections are also deposited by screen printing.

II 2 Tests conditions

The first part of the experiments has been carried out on a laboratory testing-bench by using a measurement cell equipped with a hot plate. Inside the cell, samples are placed on the surface of this plate which is heated with electrical resistances; mobile gold points are used to establish electrical contact by pressure on the electrodes. The gaseous flow and hydrogen concentration are fixed at 6 lh^{-1} and 5% v/v in nitrogen relatively. In order to simulate reformer gases, water (1.8% v/v) and carbon dioxide (1.9% v/v) could be added. The temperature of the sensor is varying between 200°C and 350°C. The electromotive force (emf) is defined by the difference between gold electrode potential and platinum electrode potential ($V_{Au} – V_{Pt}$). The variation of this emf with low level of carbon monoxide (0 to 4000 ppm v/v) has been evaluated in absence of oxygen. The oxygen concentration in the reference gas has been measured with different oxygen sensors. It is always lower than 50 ppm v/v.

The second part of the experiments has been carried out with self - heated sensors. In this case, the temperature and the emf response of the sensor are monitored with an electronic board that can be connected to a PC computer. The sensors are placed in a gas cell which is connected to the laboratory bench previously used.

III RESULTS

III 1 Preliminary results with SnO_2 and YSZ sensors

Before to realize and test news SDC sensors, we have verified that it is very difficult to use “conventional” sensors for the fuel cell application. We have prepared two types of sensors, on one hand SnO_2 sensors which are semi-conductor type, and on the other hand YSZ (yttria doped zirconia, ZrO_2 with 8 mol % Y_2O_3) sensors which are electrochemical sensors. As in the case of SDC sensors, SnO_2 and YSZ materials have been deposited by screen-printing method; two electrodes (0.5 µm thicknesses) have then be deposited by sputtering (two gold electrodes in the case of SnO_2 sensors, platinum and gold electrodes in the case of YSZ sensors).

SnO_2 sensors are usually used in oxygenated atmosphere; conductance variations could be correlated to electronic interactions and/or reactions with oxygen species of pollutant gases on the SnO_2 surface. In [2], the authors have calculated the thermal stability domains of tin oxide according to the nature of the carrier gas. Under reducing atmospheres like reformer gases, the stability of SnO_2 depends on experimental conditions and is not always warranted. SnO_2 sensors prepared in our laboratory have been tested under the fuel cell conditions defined previously. Unfortunately, and contrarily to the results in [2], no response has been recorded for CO injections in a large range of sensor temperature.

Concerning the potentiometric sensors, we have also in our group, from several years, different developments of such sensors [7, 8]. The common point with all these sensors concerns their design. All of them are screen printed sensors on alumina substrates with a dissymmetry of electrodes and without gas separation between these two electrodes. In a general manner, in the case of an oxygenated carrier gas, these sensors are considered as “mixed potential” sensors when they work at high temperature, and they can be considered more like “catalytic” sensors at lower temperature (under 400 or 500°C). Similar experiments as the one previously realized with SnO_2 sensors have been conducted in wet or dry conditions with an YSZ sensor in a large range of temperature. The best results have been obtained at 300°C in dry conditions, for injections of CO concentrations between 50 and 200 ppm v/v. As it can be observed on Fig. 2, only very small (and practically unusable) signal is obtained: Δ (emf) is of the order of 3 mV for 100 ppm v/v CO. These low responses confirm the difficulty of the objective of the present publication.

III 2 Tests on SDC sensors

III 2 1 Tests on sensors on hot plate

The influence of the temperature on the emf response has been firstly estimated in dry conditions (background constituted with H_2/N_2). When CO is introduced in the carrier gas, the emf value decreases and a so-called ″negative″ response is observed. The observed response (defined by Δ(emf)) at low temperature (200-250°C) is of the order of 25 – 30 mV for CO concentration in the range 50-200 ppm v/v. The intensity of the signal decreases when the temperature raise to 300°C and practically no response is observed at 350°C. An example of the sensor response at 200°C is shown in the Fig. 3.

In order to simulate a reformer gas, H_2O (1.8 % v/v) and CO_2 (1.9% v/v) have been added. For a given concentration, the response (defined by Δ(emf)) at 200°C under this wet atmosphere is slightly reduced if we compare with the results obtained at 200°C in dry gas. Nevertheless, this response remains perfectly usable: Δ(emf) is of the order of 20 mV for 50 ppm v/v CO. Fig. 4 shows the reproducibility of the sensor for several injections of 50 ppm v/v CO during a period of two days. The duration of the injections is 1hour and the time between two consecutives injections is 5hours. These results demonstrate the good stability of the sensors in the reference atmosphere (absence of O_2) and under 50 ppm v/v CO. Moreover, we observe the good reversibility of the sensor response.

Nevertheless, when the CO concentration is increased, the sensor response is rapidly saturated. Results for CO concentrations up to 4000 ppm v/v are shown in Fig. 5. On this figure very small variations of the Δ (emf) response are observed above 400 – 500 ppm v/v.

In order to improve the sensor response and/or the usable concentration range, the geometry of the sample and particularly the ratio of the area of electrodes Pt/Au have been modified, keeping constant the area of the Au electrode. The results obtained when the ratio Pt/Au is two are shown in Fig. 6. In comparison with the standard case (ratio Pt/au = 1), we observe an increase of the sensor response. For example, if we consider the response for 50 ppm v/v CO, the value of Δ (emf) is almost 30 mV on Fig. 3 (ratio Pt/Au = 1), and this value is close to 75 mV on Fig. 6 (ratio Pt/Au = 2). Nevertheless, the saturation effect is always observed and the next results presented in this paper will be obtained with 1 as Pt/Au ratio.

Although these tests have been conducted in a gas cell with a large volume (one litre) and a gas flow of 6 lh^{-1}, a relatively short response time is obtained as we can observe on Fig. 7 for 50 ppm CO v/v in wet conditions at 200°C. This result expresses the rapid interaction of CO with H_2 on the surface of the Pt electrode, even if the temperature is not above 200°C.

In the PEM fuel cell technology, the Pt electrode is also poisoned by H_2S which is an undesirable product of reforming processes. In the case of H_2S, the minimal acceptable concentration is less than 1 ppm v/v because the effect of H_2S on the electrical performances of the PEM is practically irreversible. The responses of a SDC sensor, at 200°C, for H_2S concentrations less than 2 ppm v/v have been studied and they are reported in Fig. 8. A small response of the sensor could be observed (Δ (emf) ≅ 5 mV for 0.5 ppm v/v H_2S), but it is so-called "positive" response, because it is on the reverse side of the CO response, i.e. the emf value increase when H_2S is introduced in the carrier gas. Moreover, with these very low H_2S concentrations, this effect appears reversible and it would seem that sensor performances are not cancelled.

III 2 2 Tests on self - heated sensors

The interesting results presented previously have been obtained in a gas cell equipped with a hot plate and metallic points as electrical contacts. This affects certainly the response time and the drifts observed on Fig 4 and 6 as for example. The noise of the signal is also due to our current electronic unit (measure and temperature regulation). Experiments with self-heated sensors are now realized. The sensors are similar to the previous one, but all electrical connexions are now fixed on a sensors socket. The large gas cell with the hot plate is now replaced by a small gas cell. The temperature and the emf signal of the sensor are controlled with an electronic board that can be connected to a PC computer. This device could be used for in situ experiments in the future.

The electrical performances of the sensors have been evaluated in a similar manner as previously. The operating conditions of the tests are the same as in the first experiments except the volume cell that is less than 0.1 litre. At first time, the influence of temperature confirms that the best responses are observed around 200-250°C. The results obtained at 250°C with one of these sensors in absence or presence of H_2O and CO_2 are shown in Fig. 9-a and 9-b respectively. We note a good response of the sensor in both cases, even if the sensitivity slightly decreases with reforming conditions (presence of H_2O and CO_2). Moreover, the measured response time is short (less than 1 minute for 90% response) if we consider the experimental conditions (dead volume, gaseous flow...). Nevertheless, as already mentioned in the first experiments, the response seems to be saturated for CO concentrations higher than 400 ppm v/v. However, for low CO levels (less than 20 ppm v/v), the response is quasi linear with the CO concentration as it is shown in Fig. 10. On this figure, the results obtained with two sensors (named A and B) of the same lot are very similar. Nevertheless, more than two sensors ought to be tested to account for the good reproducibility of the sample preparation.

IV Discussion:

The SDC sensors presented in this study are close to several new type of electrochemical sensors which are currently develop by several groups, especially for automotive exhaust applications [9, 10]. In regard of conventional electrochemical sensors with a physical separation between the two electrodes, these sensors have their two electrodes in the same atmosphere. These sensors are usually called “Non Nernstian” potentiometric sensors [11, 12] or “Mixed Potential” sensors [13, 14]. They present a dissymmetry of electrodes and the response is connected to the differences of the electrochemical reactions occurring at both electrodes. These sensors generally work at relatively high temperature (500 to 800°C). If these types of sensors are used at lower temperatures, under 400°C for example, their properties are essentially dependent on the catalytic activities of the electrodes, more precisely on the difference of the catalytic activities of the electrodes. In this sense, there are sometime mostly called “catalytic” types. According to the best responses obtained at very low temperature, the SDC sensor presented in this paper is relevant of this last group.

As it could be observed on different figures of this article, the initial value of the sensor (under carrier gas) is different from one experience to another one. For a given value of the ratio Pt/Au (one for example), this baseline is greatly dependent on the temperature of the sensor in the range 200-300°C. The variation of this value is of the order of several hundreds millivolts. The use of the hot plate for several tests presented in this paper could induce small variations of the sensor temperature due to its position in the cell. Then, great variations of the baseline level could be observed (Fig. 3, 4, 7, 8). In the case of self-heated sensor, the temperature is more correctly reliable and the baseline less fluctuates. Moreover, the baseline depends on the sample geometry and particularly on the area of the electrodes. On the contrary, for a given sample, the stability of the baseline is correct in the same experimental conditions during several days as seen on the Fig. 4. In fact, the major interesting result for a future application is the practically constant value of Δ (emf) for a given CO concentration in a given carrier gas whatever the level of the baseline which, on the contrary, depends on several experimental parameters.

For the SDC sensor, the origin of the potential in the reference gas (in absence of CO) can be considered as mainly due to the difference of the catalytic activities towards H_2 dissociation between the two electrodes. When CO is present, H_2 adsorption on Pt electrode is hindered whereas the gold electrode potential is globally unchanged: the emf is then modified which induces the CO response. At high temperature, the CO adsorption on Pt electrode is less and the sensor response disappears. For the moment, it is difficult to propose a sensing mechanism only on the basis of the preliminary results presented in this paper. Nevertheless, the mixed potential theory seems difficult to be applied. Indeed, with this mechanism, oxido-reduction reactions generally take place on both electrodes whereas, in our case, the synthetic gas flow is principally constituted of inert (like N_2) or reducing (like H_2 and CO) gases, with only traces of O_2 (less than 50 ppm v/v). By another way, H_2O is a potential source of O_2, but H_2O appears only in slight trace in the case of dry experiments when the sensor response is the best.

Other results presented in this article confirm the main role of the catalytic activity of the electrodes in the signal of the SDC sensors, namely the influence of the size of the electrodes. We have observed a large influence of the Pt electrode area on the baseline (emf measured under carrier gas) but also on the sensor response (Δ (emf)). When Pt area electrode is increased while Au area electrode is kept constant, it could be supposed that Pt electrode potential is more largely modified with CO adsorption and the difference between the two electrode potentials is amplified. Nevertheless, the saturation phenomenon is always observed.

The response to H_2S, in opposite sense of the one to CO, is more difficult to explain. This means that the behaviour of H_2S on the two electrodes is very different than the CO. This could be related to the well known reactivity of H_2S on metals at very low temperatures. Some effects have been observed at room temperature on platinum. Moreover, the reactivity of H_2S with gold is not zero as in the case of CO [15]. These effects on gold can contribute to the positive response observed. Another point is related to the poisoning effects with H_2S: in our case, we don't observe any irreversible effects with H_2S. This appears surprising taking into account the well recognized poisoning action of this gas. In fact, only very low H_2S concentrations have been tested but the durations of experiments are long (several hours). One possible explanation is the relative high temperature (200 to 250°C) which improves the desorption step.

The last point is related to the tests of self-heated sensors. The results obtained are close to those obtained with the hot plate. Nevertheless, using a reduced cell volume, the measured response time and recovery time are particularly short. The response time is less than one minute for 90% response. These short adsorption-desorption steps appear surprising taking into account the very low temperature (250°C). This particular property reinforce the catalytic mode described previously in regards of the contribution of the electrolyte material which seems to play here a simple role of transduction material in order to translate the difference of catalytic activity. Despite our lake of exact model to explain our experimental results, we think that the potential benefits in terms of application are enough important to be presented. The SDC sensors are particularly interesting in order to detect in situ low concentrations of CO in H_2-rich gases with a quasi linear response in the range (0-20 ppm v/v). So, they are very well adapted to monitor the quality of reformed gases in fuel-cell applications, especially "normal" PEM fuel-cell which work at low temperature (below 80°C) and are poisoned by traces of CO. In regards of these "normal" PEM, there are current developments to propose high temperature PEM (HT-PEM) able to work between 150 to 180°C. One interest of such new generation of PEM is the acceptation of high CO concentration in the range 2000 – 4000 ppm. According to the observed saturation effects, the "normal" SDC sensors (ratio Pt/Au = 1) are not well adapted to monitor the quality of reformed gases in the case of HT-PEM. So we will have to continue in the future to work on the design of the sensors, especially with large difference of the electrodes sizes. For such HT-PEM, we have also current studies on the use of miniaturized PEM as the one presented in the introduction. On the contrary of the SDC sensors, it appears that these types of mini-PEM sensors seem well adapted to detect high concentration CO in H_2 rich gas, and inadequate for low CO concentration.

V Conclusions:

In this work, an original potentiometric sensor has been developed in order to detect low CO concentrations in H_2-rich gases for PEM fuel cell application. Conventional sensors are not able to function correctly because the absence of oxygen in the reformer gaseous flow. The potentiometric sensor is elaborated with SDC (samarium doped ceria) as electrolyte which is a well known material for SOFC technology and stable under hydrogen below 600°C. Moreover, this sensor is a very simple mono chamber potentiometric sensor based on the dissymmetry of electrodes (Pt-Au) without reference cell. The hydrogen adsorption (and dissociation) on Pt electrode is hindered when CO is present in the gaseous flow that induces a variation of the emf signal correlated with CO concentration. The SDC sensors present interesting performances to detect very low CO concentrations (1 to 20 ppm) with good reversibility and stability in H_2 rich atmospheres. A way to improve sensor's performances could be to increase the surface ratio Pt/Au beyond 1, the commonly used value. Currents works are concerning the comprehension of sensing mechanism and particularly the reactions involved on platinum and/or gold electrodes.

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Figures:

Fig. 1: SDC sensor with the SDC sensing material (left) and the low temperature connections (right).

Fig. 2: Transient response to 0-200 ppm v/v CO of a YSZ sensor at 300°C. / Carrier gas: H_2 (5% v/v) in N_2.

Fig. 3: Transient response to 0-200 ppm v/v CO of a SDC sensor (with ratio Pt/Au = 1) at 200°C. / Carrier gas: H_2 (5% v/v) in N_2.

Fig. 4: Stability of the response to 50 ppm v/v CO of a SDC sensor (with ratio Pt/Au = 1) at 200°C. / Carrier gas: H_2 (5% v/v) in N_2 with [H_2O] (1.8%) and [CO_2] (1.9%).

Fig. 5: Response (Δ emf (mV)) of a SDC sensor (with ratio Pt/Au = 1) at 200°C as a function of the CO concentration (0 – 4000 ppm v/v). / Carrier gas: H_2 (5% v/v) in N_2.

Fig. 6 Transient response to 0-400 ppm v/v CO of a SDC sensor (with ratio Pt/Au = 2) at 200°C. / Carrier gas: H_2 (5% v/v) in N_2 .

Fig. 7 Details of the response to 50 ppm v/v CO of a SDC sensor (with ratio Pt/Au = 1) at 200°C. / Carrier gas: H_2 (5% v/v) in N_2 with [H_2O] (1.8%) and [CO_2] (1.9%).

Fig. 8: Transient response to 0-1.5 ppm v/v H_2S of a SDC sensor (with ratio Pt/Au = 1) at 200°C. / Carrier gas: H_2 (5% v/v) in N_2 with [H_2O] (1.8%) and [CO_2] (1.9%).

Fig. 9: Transient response to 0-400 ppm v/v CO of a self-heated SDC sensor (with ratio Pt/Au = 1) at 250°C.

a): Carrier gas: H_2 (5% v/v) in N_2 , 0-400 ppm v/v CO ;

b): Carrier gas: H_2 (5% v/v) in N_2 with [H_2O] (1.8%) and [CO_2] (1.9%), 0-200 ppm v/v CO.

Fig. 10: Response (Δ emf (mV)) of two self-heated SDC sensor (with ratio Pt/Au = 1) at 250°C as a function of the CO concentration (0 – 20 ppm v/v). / Carrier gas: H_2 (5% v/v) in N_2 with [H_2O] (1.8%) and [CO_2] (1.9%). / A and B are two sensors of the same lot of preparation.

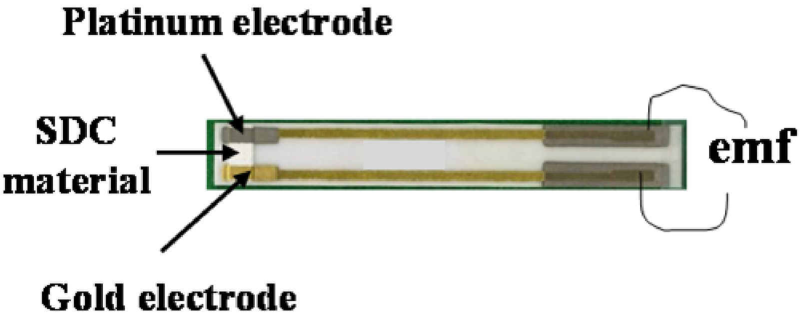

Figure 1

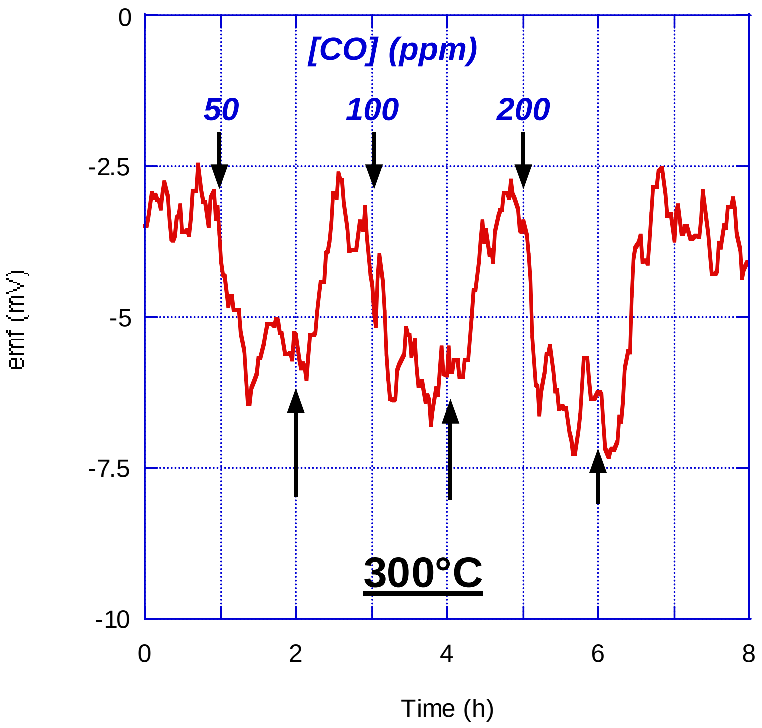

Fig. 2

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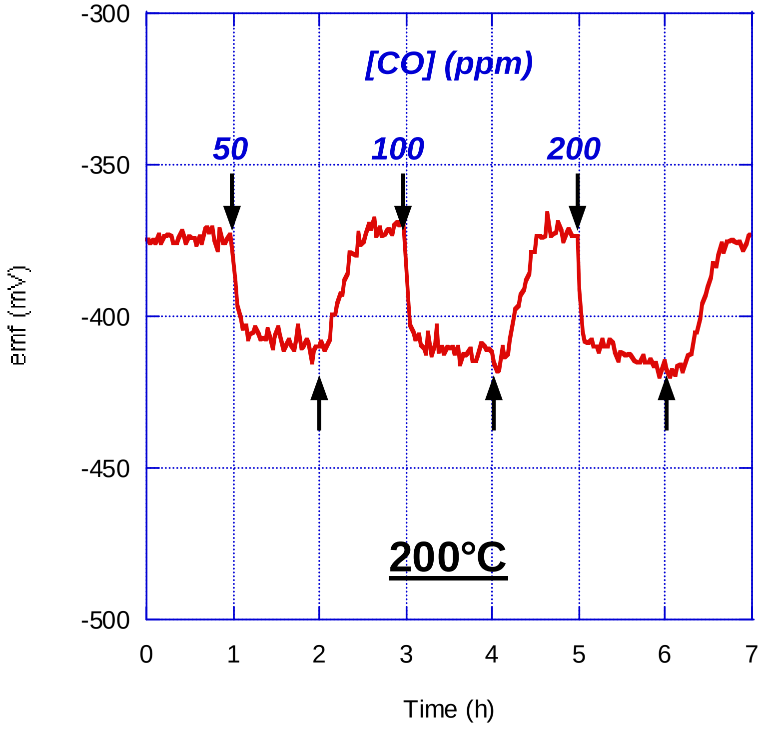

Fig. 3

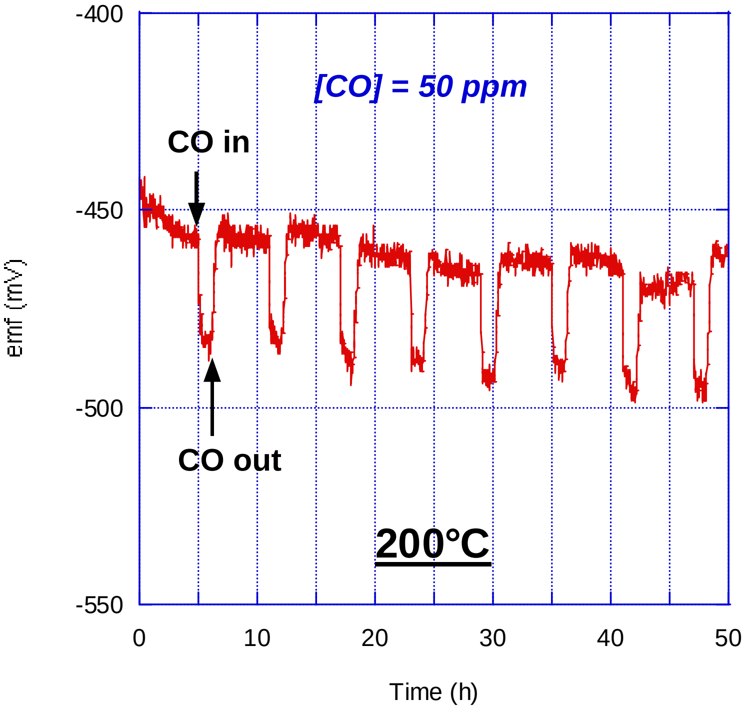
-400
-450
-500
-550
emf (mV)
0
10
20
30
40
50
Time (h)
[CO] = 50 ppm
CO in
CO out
200°C

Fig. 4

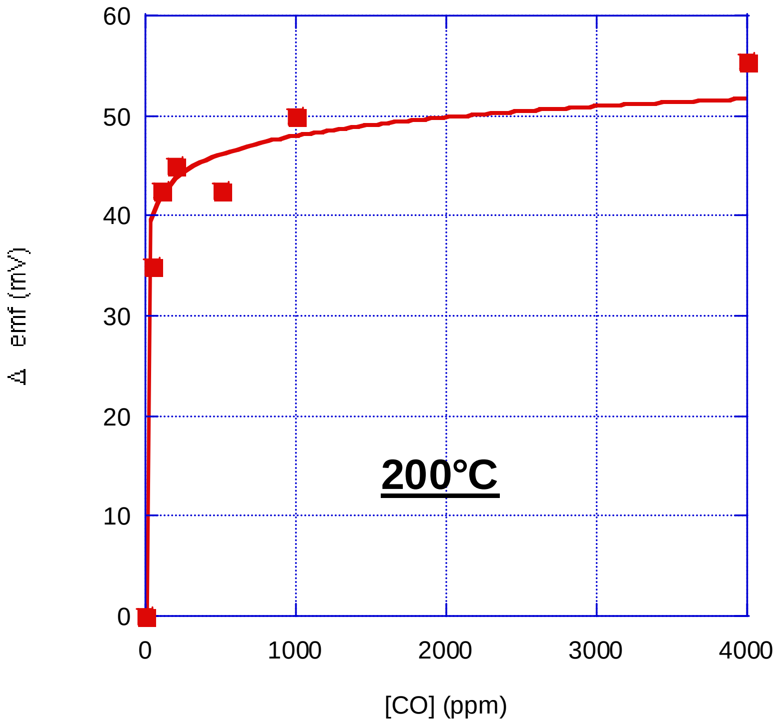

Figure 5

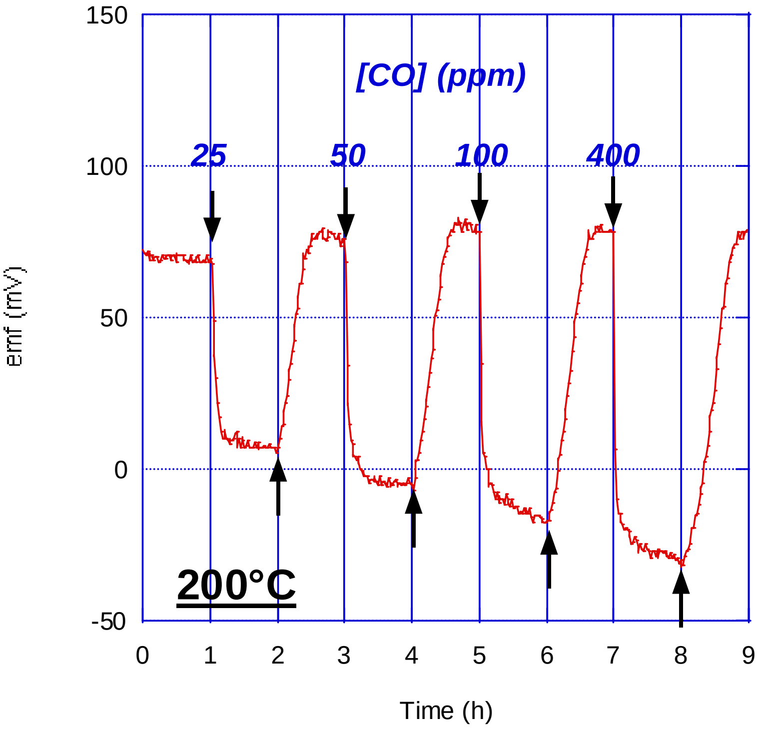
150
100
50
0
-50
emf (mV)
0
1
2
3
4
5
6
7
8
9
Time (h)
[CO] (ppm)
25
50
100
400
200°C

Figure 6

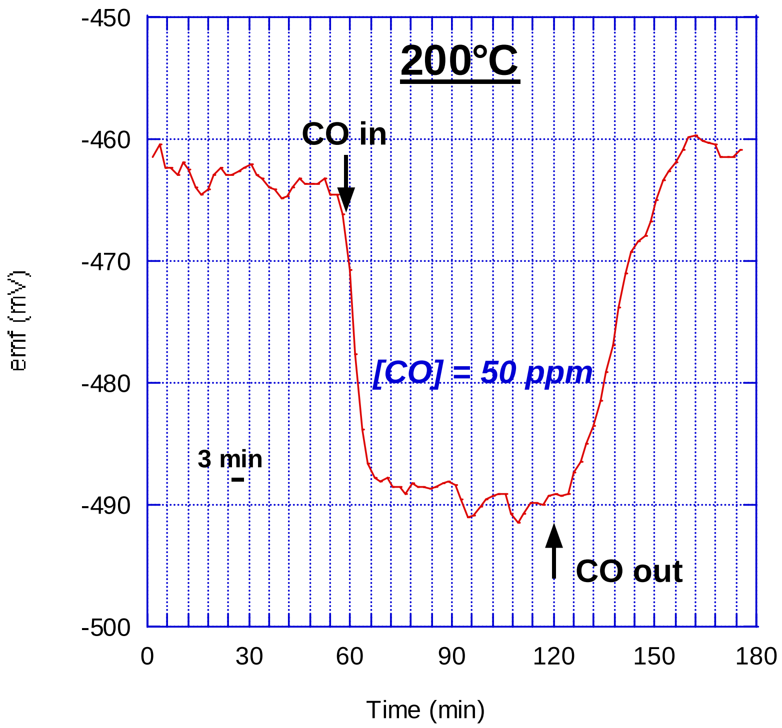
200°C
CO in
[CO] = 50 ppm
3 min
CO out
emf (mV)
-450
-460
-470
-480
-490
-500
0
30
60
90
120
150
180
Time (min)

Figure 7

Sensors and Actuators, 2009, 141(1), 7-12, doi:10.1016/j.snb.2009.06.004

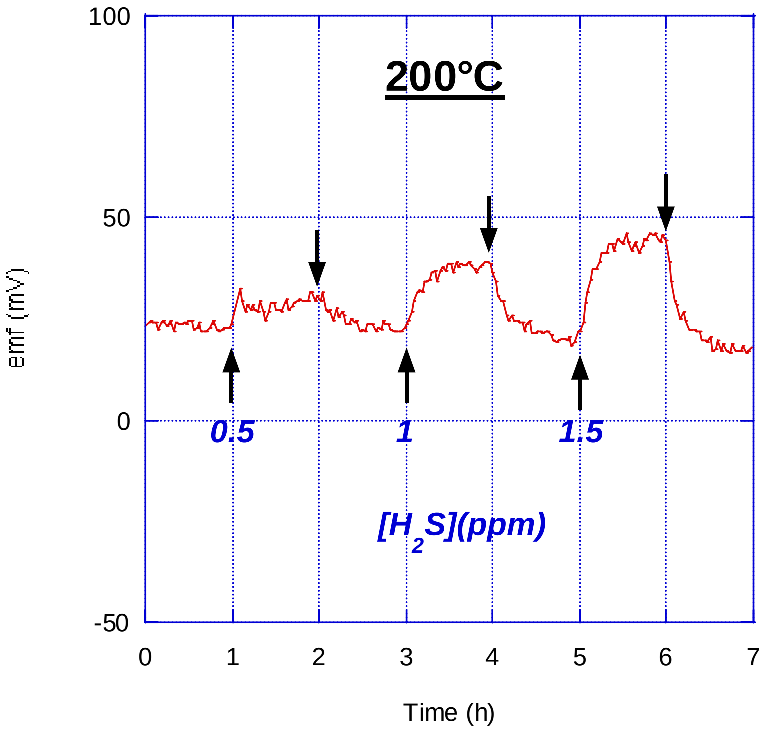

Figure 8

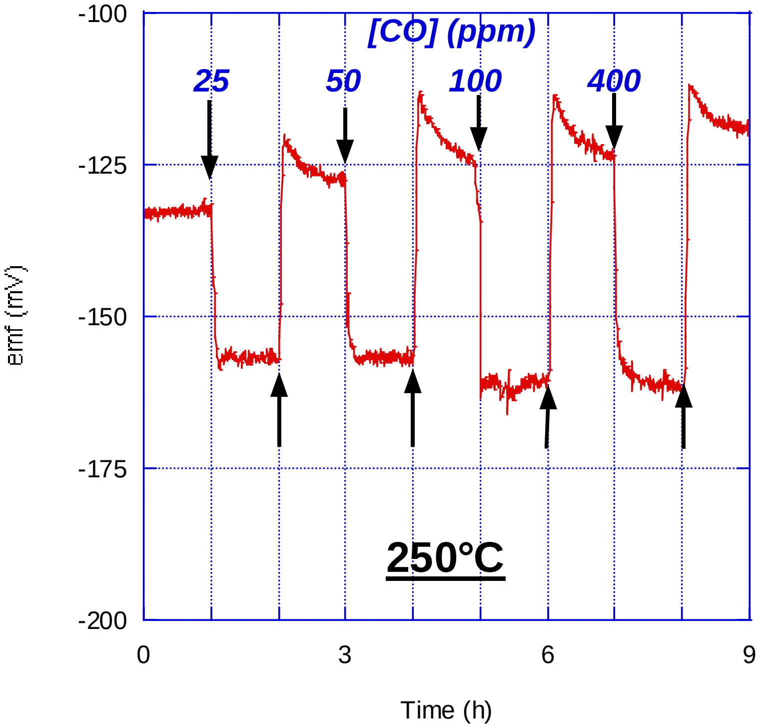

Figure 9a

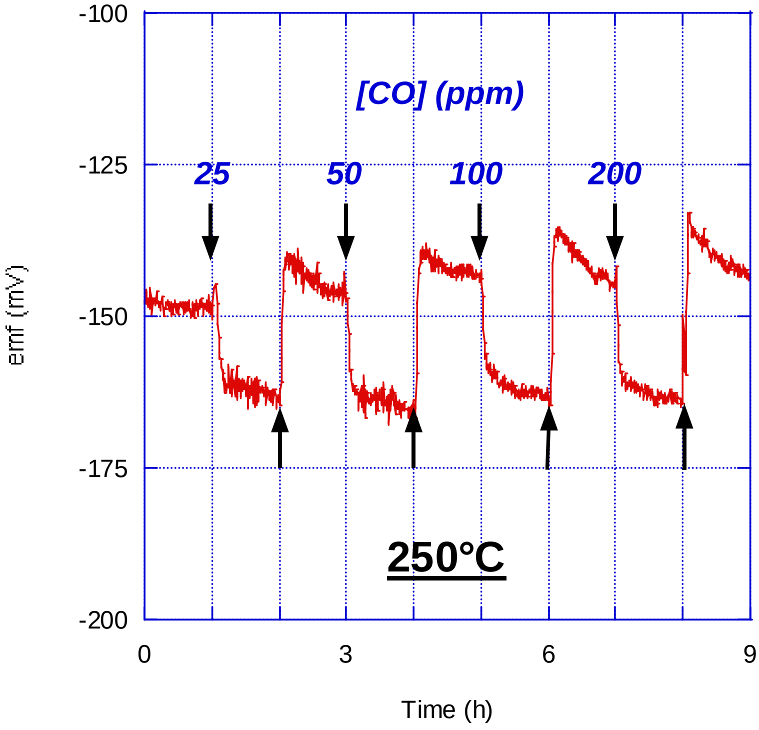

Figure 9b

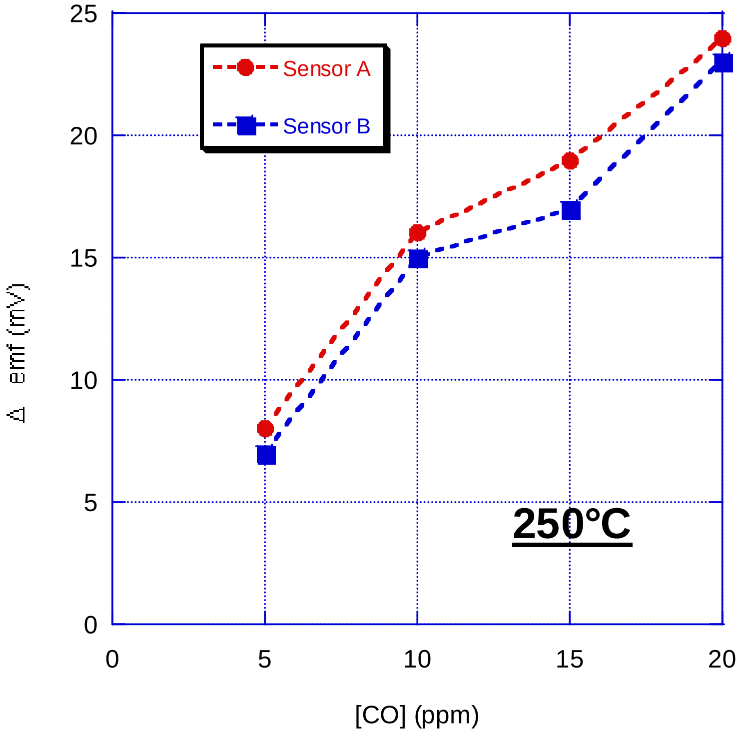

Figure 10