

Compact electrochemical bifunctional NO_x/O₂ sensor with metal/metal oxide internal reference electrode for high temperature applications

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Abstract

Simultaneous measurement of total NO_x and O₂ using two electrochemical methods are demonstrated using metal/metal oxide internal oxygen reference electrode-based sensors at high temperatures. The Pd/PdO-containing reference chamber was sealed within a stabilized zirconia superstructure by a high pressure/temperature plastic deformation bonding method exploiting grain boundary sliding between the ceramic components. Amperometric and potentiometric NO_x sensing devices were assembled on the outside of the sensor. Pt-loaded zeolite Y was used to obtain total NO_x capability. Both the amperometric and potentiometric type sensors showed total NO_x response, with the potentiometric device showing better NO_x/O₂ signal stability and lower NO_x–O₂ cross-interference. Since these sensors do not require plumbing for reference air, there is more flexibility in the placement of such sensors in a combustion stream.

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1. Introduction

Accurately monitoring gases such as O₂, NO_x, CO, CO₂ in combustion processes enables energy output optimization and pollutant minimization in fossil fuel-driven applications. Sensor-enabled feedback control of oxygen levels in coal boilers has been estimated to enable yearly savings of \$409 million from the coal-fired power industry alone [1]. NO_x (NO + NO₂) sensing has also been considered a key element in optimization of the next generation internal combustion engines [2]. A reliable and accurate NO_x sensor is needed to monitor NO_x breakthrough and trigger the regeneration of NO_x adsorption catalysts, and in controlling the injection of reductants for continuous NO_x reduction [3].

For transport applications, monitoring NO_x and O₂ simultaneously is necessary for emission control and air/fuel ratio measurement. Most high-temperature sensors capable of detecting O₂ and NO_x are based on stabilized zirconia, which has

high ionic conductivity, mechanical and chemical stability at high temperatures [4,5]. Zirconia oxygen sensors have long been used to increase fuel efficiency and minimize emissions [6]. A multi-stage configuration device with external oxygen reference is the most common approach to detect total NO_x and oxygen [7]. In this design, air is provided from outside the combustion environment to a reference electrode that is separated from the sensing environment by a zirconia channel [6]. The oxygen concentration differential between the reference air and the measuring environment generates an open circuit potential that obeys Nernst law, allowing for direct calculation of the concentration of oxygen. Meanwhile, gases in the combustion environment diffuse through a narrow channel into one or two chambers constructed of laminated yttria-stabilized zirconia (YSZ) sheets. The first chamber is normally equipped with oxygen pumping electrodes, which can selectively remove oxygen from the gas mixture to minimize the oxygen interference. A pair of noble metal electrodes then electrochemically converts the NO_x mixture into NO or NO₂ exclusively, which is finally detected by either potentiometric or amperometric methods.

Although great effort has been devoted to develop NO_x/O₂ dual sensors with the multi-stage configuration, there are prob-

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lems with this design. In order to construct gas chambers and introduce reference air from the external environment, a number of YSZ sheets and insulation layers (up to 13 separate layers) need to be well aligned and laminated [8,9]. This process drastically increases the complexity of sensor fabrication and compromises the sensor durability under thermal shock and thermal cycles. Also, requiring external air constrains the location of sensors inside the combustion environment and impedes developments toward sensor miniaturization.

We have previously demonstrated that the external air reference for an oxygen sensor can be replaced by a Pd/PdO internal reference, which is sealed inside a YSZ chamber and provides an effective reference oxygen pressure (pO_2) [10]. Physical gas leakage was eliminated by fusing a stabilized zirconia package into a discrete body through a plastic deformation bonding technique. The joining process using grain boundary sliding enabled the creation of complex, jointless structures [11]. Seals created by directly fusing zirconia components are inherently free of bonding agents and do not interfere with sensor operation. Leak faults induced by thermal expansion mismatch over temperature cycling and long periods of time are similarly eliminated. These sensors exhibit stability on the order of months, with no indication of failure upon test completion.

We have also reported on an amperometric total NO_x sensor based on measuring current upon application of a low anodic potential (~ 80 mV) to Pt electrodes on a YSZ electrolyte [12]. Pt-loaded zeolite Y (PtY), an active NO_x equilibration catalyst with high thermal stability and surface area, was used as a coating on the YSZ to equilibrate the NO_x . In our design of the potentiometric sensor, PtY was used as a separate filter at a different temperature from the sensor, the difference in thermodynamic equilibrium of NO_x and O_2 at the two temperatures resulted in a potentiometric signal for total NO_x [13,14]. The optimal signal was obtained with the sensor at 600°C and the filter at 400°C . Additionally, sub-ppm NO_x detection was obtained based on the dissimilar catalytic activity of reference electrode Pt/PtY and sensing electrode WO_3 [14]. Use of the PtY filter also effectively minimized interferences from 2000 ppm CO, 1000 ppm propane, and 10 ppm NH_3 . Other gases, including 30% CO_2 , 5–10% H_2O , 1–13% O_2 did not cause significant interference.

In this paper, the two different electrochemical NO_x measuring techniques, amperometric and potentiometric, were combined with the internal reference oxygen sensor to design a single sensor that can measure O_2 and NO_x simultaneously at high temperatures. Detailed characterizations of the individual sensors are available in Refs. [10,12–14].

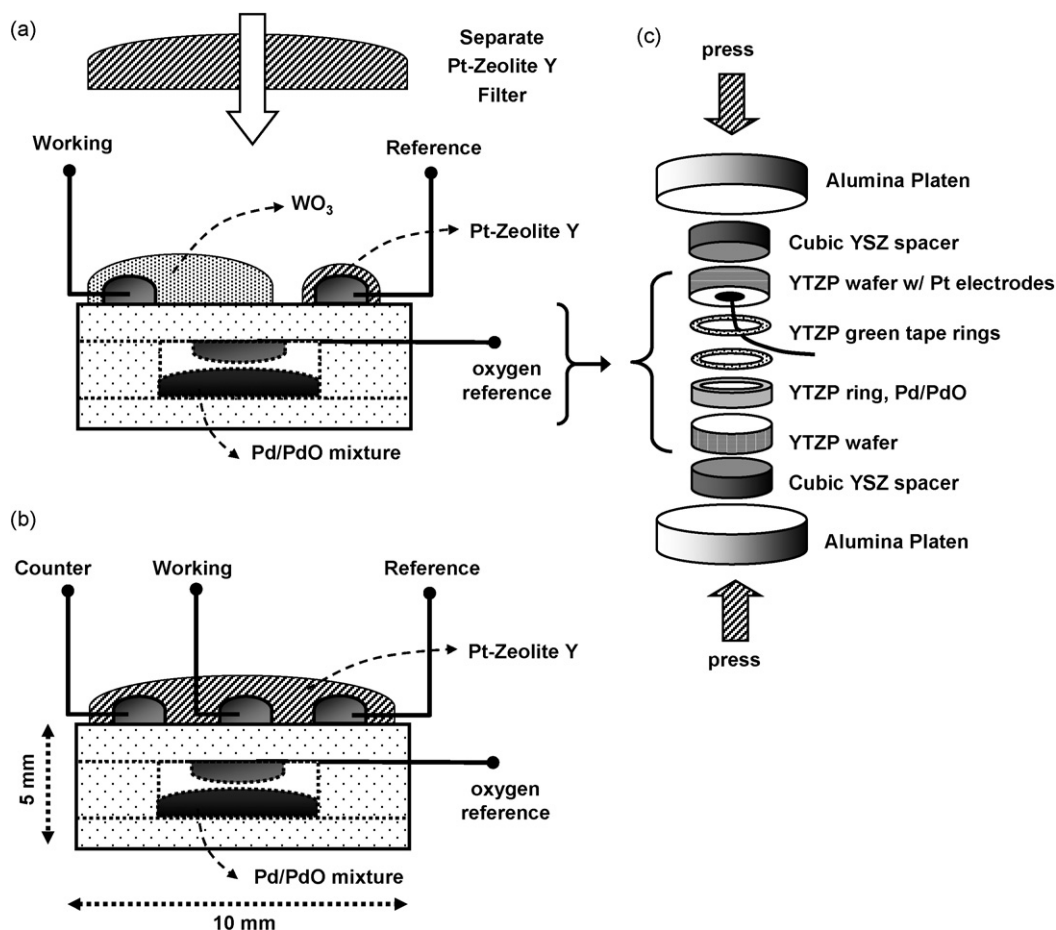


Fig. 1. Cross-sectional view of the sensor assembly: (a) potentiometric configuration for NO_x/O_2 sensor; (b) amperometric configuration for NO_x/O_2 sensor; (c) components of the oxygen sensor package. Cubic YSZ spacers and alumina platens were removed following joining and not part of the package.

2. Experimental

2.1. Preparation and characterization of sensor materials

Fig. 1 shows the schematic of the sensor assembly. The electrolyte, ring and bottom wafer for the O₂ sensor (Fig. 1c) were cut from densified rods/tubes of 3 mol% yttria-stabilized tetragonal zirconia polycrystals (YTZP, Custom Technical Ceramics). The 8 mol% cubic yttria-stabilized zirconia spacers were cut from a rod (Custom Technical Ceramics) and used to prevent reaction between YTZP and the alumina platen. The YTZP green tapes (Nextech Materials) were laser cut from sheets into rings that matched the YTZP rings cut from tubes. The Pt wire (99.95%, 0.13 mm in diameter, Fischer Scientific) to the inner reference electrode was sandwiched between two pieces of YTZP green tape. The devices were constructed by sealing PdO powder (Alfa Aesar) into the reference chamber [10]. The samples of PdO typically contain about 15 wt% Pd metal and during sensor preparation, the equilibrium between Pd, PdO and O₂ is established. A small amount of 8 mol% YSZ powder (TZ-8Y, Tosoh) was placed on top of PdO to prevent alloying with the Pt wire of the reference electrode.

The Pt-loaded zeolite Y powder was prepared from Na-exchanged zeolite Y (Si/Al = 2.5, Union Carbide, LZ-Y-52) by ion-exchange. 1.0 g of NaY powder was dried at 100 °C for 4 h followed by mixing with 2.5 mM [Pt(NH₃)₄]Cl₂ (Alfa Aesar) solution. The mixture was stirred overnight at room temperature for ion-exchange. After washing and centrifuging with distilled water several times, the Pt-exchanged powder was dried at 70 °C for 3 h and then calcined at 300 °C for 2 h. The heating rate of calcination was set to 0.2 °C/min to increase the Pt dispersion by preventing autoreduction of the ammonia ligand. The calcined zeolite was exposed to 5% H₂ to reduce Pt²⁺ in the zeolite framework to metallic Pt. The Pt content determined by ICP-OES in Galbraith Lab Inc., is 4.36%. WO₃ was used from a commercial powder (99.8%, Alfa Aesar) without any further treatment.

2.2. Sensor fabrication

2.2.1. Basic platform

The “sandwich” arrangement shown in Fig. 1c was compressed in an argon atmosphere at temperatures ranging from 1250 °C to 1290 °C in a universal testing machine (Instron, Model 1125) at crosshead speeds ranging from 0.01 mm/min to 0.02 mm/min. The strain rate used for all experiments was $4 \times 10^{-5} \text{ s}^{-1}$ in order to avoid overstressing the sample and damaging the Pt wire. In the >1250 °C temperature domain, this strain rate is expected to yield total stresses on a 1 cm² sample of less than 40 MPa [11]. During the heating cycle, the load on the sample was balanced as not to exceed 5 N. Upon reaching the target temperature, the system was left under a 5 N load for 30 min to attain thermal equilibrium. Following joining, a glass plug (C153M glass, Asahi) was applied to the region of the sensor package where the Pt wire breached the inner-to-outer environment. C153M is a frit sealing glass composed of SiO₂, ZnO, and organic compounds (softens at 800 °C, crystallizes at 910 °C). The glass powder was mixed with α -terpineol to make

a slurry and a small amount was applied to the base of the Pt wire. The sensor was heated to 1200 °C for 3 h to densify the glass plug.

2.2.2. Potentiometric configuration

The potentiometric NO_x sensor was constructed as shown in Fig. 1a. Two Pt lead wires were attached to the top of the oxygen sensor with a small amount of commercial Pt ink (Englehard, A4731). The end attaching to YSZ was shaped into a disc of 2 mm diameter in order to increase the mechanical stability. The Pt ink was cured at 1200 °C for 2 h to secure bonding between the Pt wire and YSZ. WO₃ powder was mixed with α -terpineol to form a paste, which was then painted on top of the Pt lead wire and YSZ. The WO₃ layer was spread over as much YSZ as possible. After sintering at 700 °C in air for 2 h, the WO₃ layer was typically about 200 μm thick. PtY was also mixed with α -terpineol and painted on the top of another Pt lead wire to form the reference electrode. The PtY layer is around 100 μm thick after calcination in air at 600 °C for 2 h.

2.2.3. Amperometric configuration

Three Pt wires (working, counter, and reference) were mounted on top of the oxygen sensor to design the amperometric sensors (Fig. 1b). The reference electrode was kept well separated from the working and counter electrode to reduce electrical interference. PtY was mixed with α -terpineol to form a paste and then painted on top of all three electrodes. The sensor was heated in 650 °C in air for 2 h and then cooled back to 500 °C for sensing study.

2.3. Sensor testing

The gas sensing experiments were performed within a quartz tube placed inside a tube furnace (Lindberg Blue, TF55035A). A computer-controlled gas delivery system with calibrated mass flow controllers (MFC) was used to introduce the test gases. Four certified N₂-balanced NO_x cylinders (30 ppm NO, 30 ppm NO₂, 2000 ppm NO, and 2000 ppm NO₂, Praxair) were used as NO_x sources. Sensor tests were carried out with mixtures of dry air, NO₂, and nitrogen with total gas flow rates of 200 cc/min at 500 or 600 °C. As shown in Fig. 2a, the gas mixture from MFCs could be introduced into the tube furnace either through or bypassing the PtY filter. The filter is a U-shape quartz tube with 100 mg PtY placed on quartz wool. The function of the PtY filter was described in detail in our previous work [13,14].

Pt wires were used to connect the sensor to external leads. For both configurations (Fig. 2b and c), the open circuit potential between the oxygen reference and the Pt/PtY reference (the reference electrode on the top of sensors, covered by PtY) was recorded by a multiplex data acquisition system (HP 34970A) with 10 G Ω internal impedance. The oxygen reference electrode was connected to the negative terminal of the HP multimeter.

For the amperometric configuration (Fig. 2b), an in-house designed portable potentiostat was used to apply a constant potential on the working electrode. The potentiostat is a standard three wire design using two CA3140 operational amplifiers optimized for low frequency operation and could be powered by

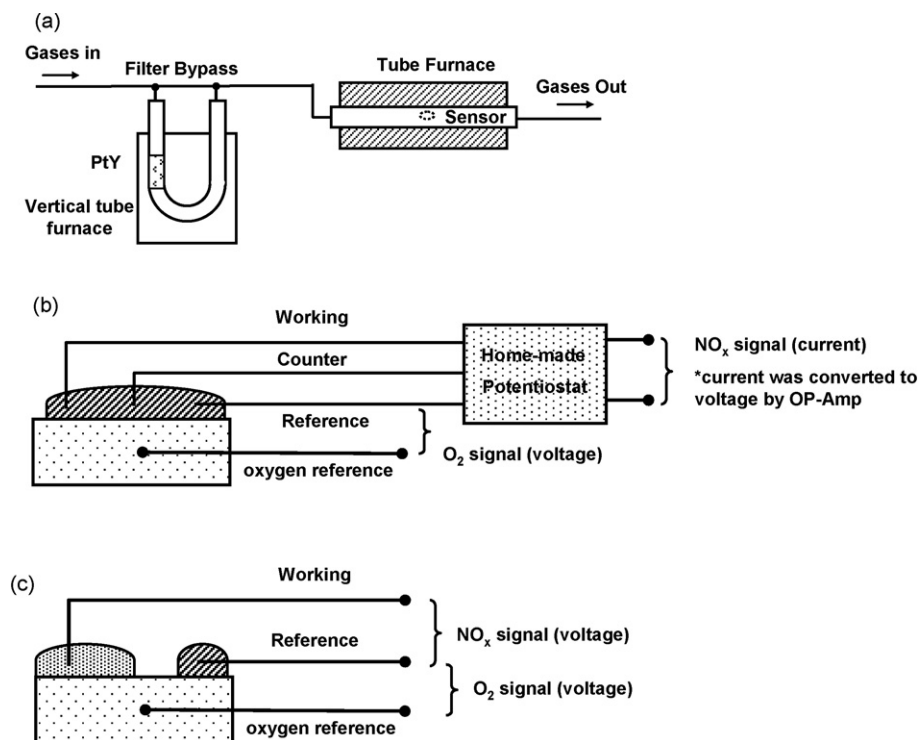


Fig. 2. (a) Sensor testing setup (PtY = Pt-zeolite Y); (b) measurement setup for the amperometric NO_x/O₂ configuration; (c) Measurement setup for the potentiometric NO_x/O₂ configuration.

two nine volt batteries or 110 V connection. The first amplifier is configured as a voltage follower with the auxiliary electrode as output and reference electrode as feedback. A dial selectable, 200 mV source is connected to the non-inverting input of the amplifier. Compliance voltage is 6 volts. The working electrode is connected to second amplifier which is configured as a current to voltage converter with switch selectable gains to provide 1 V/μA and 10 V/μA sensitivities. A 1.0 μf capacitor is wired in series with the gain resistors for stability and noise rejection. In the potentiometric configuration (Fig. 2c), the open circuit potential between the WO₃ and Pt/PtY reference electrode was measured by the HP multimeter with the Pt/PtY electrode connected to the negative terminal.

3. Results and discussion

3.1. Oxygen sensing behavior

The oxygen sensing behavior for the amperometric and potentiometric sensor was examined at their optimal working temperatures of 500 °C and 600 °C (based on Ref. [10]), using the electrodes shown in Fig. 2b and c. As shown in Fig. 3, the open circuit potential between the Pd/PdO internal reference and the sensing electrode exhibits a linear dependence on the logarithm of the partial pressure of oxygen, as expected from the Nernst equation. In our original paper reporting on sealed oxygen sensors [10], we demonstrated that measured potential correlated well with the theoretical potential. Using the same methodology, the data from Fig. 3 at 600 °C for the six external oxygen concentrations was used to calculate the internal pressure of oxygen and was found to be $4.08 \times 10^{-4} \pm 3.31 \times 10^{-5}$.

The small standard deviation suggests that the internal pressure remains constant. The theoretical value expected for internal pressure of oxygen at 600 °C is 7.1×10^{-4} [10], and we suggest that the difference with the experimental value arises from positioning the sensor within the oven where it stabilized around a temperature of 585 °C.

3.2. Total NO_x sensing

The total NO_x sensing performance for the amperometric and potentiometric sensors was monitored as outlined in Fig. 2b and c. Optimal temperatures of operation of these sensors was based

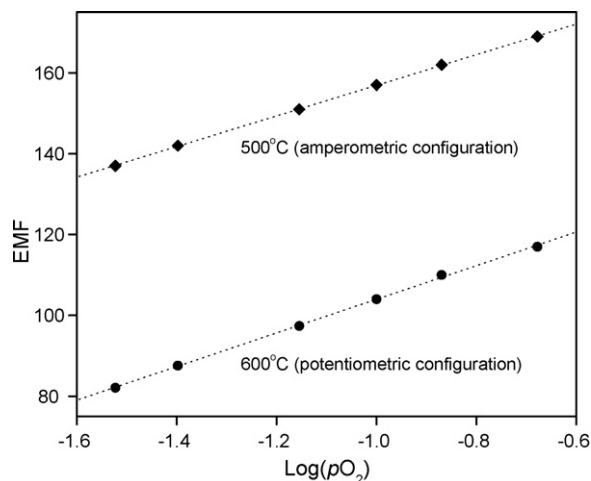


Fig. 3. EMF vs. log [pO₂] plots at 500 °C (amperometric design) and 600 °C (potentiometric design).

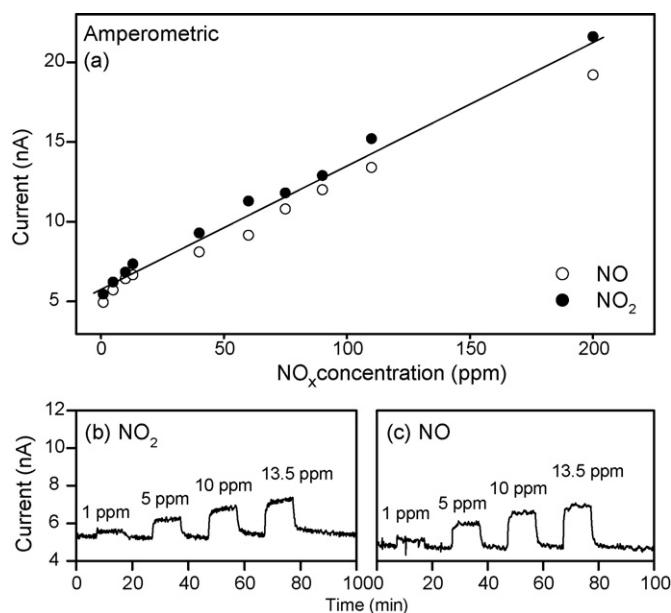


Fig. 4. (a) Current vs. $[\text{NO}_x]$ plots for the amperometric configuration at 500°C in 3% O_2 ; (b) and (c): response transients to 1–13.5 ppm NO_2 and NO .

on previous published data [10,12–14]. The amperometric type sensor at 500°C was anodically polarized to 80 mV (Fig. 1b, vs. the PtY reference electrode) and the current in the presence of 1–200 ppm NO_x in 3% O_2 was recorded. The bias potential of 80 mV was chosen based on the I - V behavior since a measurable current in the presence of NO_x was expected [12]. The calibration plot in Fig. 4a exhibits a linear relationship between current and concentration of NO_x . The similarity of response transients in Fig. 4b and c for 1–13.5 ppm NO or NO_2 demonstrates that the PtY thick film coated on the sensor surface can equilibrate the NO_x mixture and that the sensor produces a total

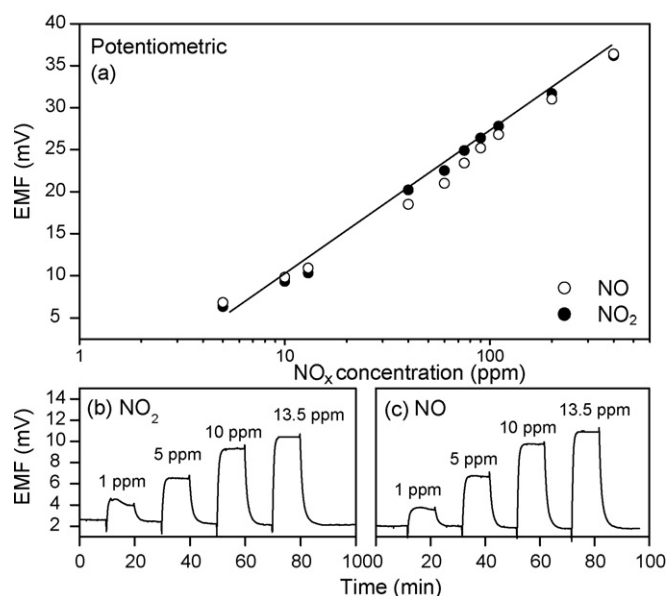


Fig. 5. (a) EMF vs. $\log [\text{NO}_x]$ plots for the potentiometric configuration at 600°C with gases passing through a PtY filter (400°C). O_2 level is 3%; (b) and (c): response transients to 1–13.5 ppm NO_2 and NO in the presence of 3% O_2 .

NO_x response (note that the response to NO_2 is slightly longer than NO).

The potentiometric sensor was tested with the gas mixture passing through the PtY filter heated at 400°C . As shown in Fig. 5, NO_2 and NO generate similar potential with a logarithmic relation to NO_x concentration. The use of the PtY filter makes it possible to measure total NO_x [14]. The strong NO_x signal is due to dissimilar catalytic activity of PtY and WO_3 [14]. Fig. 5b and c show the sensor transients as a function of concentration varying from 1 ppm to 13.5 ppm.

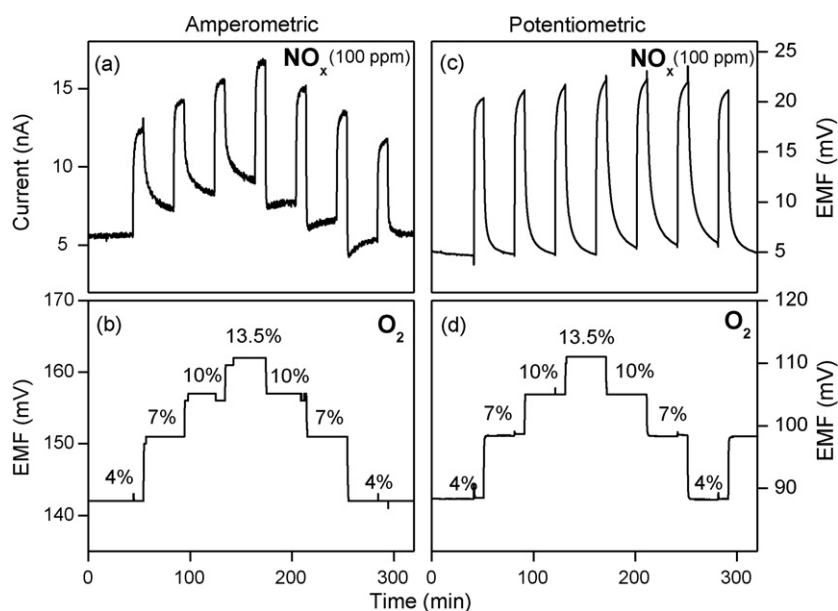


Fig. 6. Simultaneous NO_x and O_2 signal measurements for 100 ppm NO_2 in different oxygen levels: (a) and (b) were acquired after an electrochemical pretreatment. Amperometric sensor at 500°C and potentiometric sensor at 600°C , with filter at 400°C .

3.3. Simultaneous NO_x and O₂ measurements

Both types of sensors were tested with 100 ppm NO₂ in oxygen levels varying from 4% to 13.5% to examine the cross-interference between NO_x and oxygen. For the potentiometric sensor (Fig. 6d), the oxygen signal is not influenced by 100 ppm NO_x. Fig. 6c shows that the signal from 100 ppm NO₂ is influenced by the oxygen concentration, varying by about 10% relative error with oxygen levels changing from 4% to 13.5%.

For the amperometric sensor, the low ionic conductivity of YSZ and high electrode interfacial impedance at 500 °C led to sluggish O₂ response, which was improved by electrochemical treatment involving polarization of the PtY reference electrode to 500 mV for 2 h, followed by overnight treatment at 500 °C. This polarization treatment decreases the electrode interfacial impedance and the response time [15]. Electrochemical pre-treatment has been applied to increase the stability and alter the impedance of electrodes in various electrochemical devices including glassy fiber detectors [16], sol–gel carbon composite electrodes [17], and zirconia sensors [18]. As shown in Fig. 6b, the O₂ response at 500 °C (Fig. 6b) is comparable to the potentiometric sensor 600 °C (Fig. 6c). For the NO₂ signal (Fig. 6a) the amperometric configuration has more pronounced oxygen interference with 39% relative error.

3.4. Long-term stability testing

To examine the long-term stability of the sensor, an amperometric type sensor was tested at 500 °C for 2 months with O₂ (3%, 21%) and NO₂ (40–110 ppm), and the results are presented in Fig. 7. There is a drift in the NO_x background signal with time, the drift decreasing with time, though still noticeable after 2 months. The signal current versus [NO_x] curves obtained at various times have similar slopes (Fig. 7b insert), as also observed in our pervious work [12], which implies that the signal drift could be corrected periodically from the baseline signal (0 ppm NO_x). For the oxygen signal, extremely stable signal was measured during the 2-month period with only 1% error.

A 2-week stability test was carried out with the potentiometric sensor and the data is shown in Fig. 8. The sensor was tested at 45–600 ppm NO₂ and 3–21% O₂. Less than 0.5% error is observed from the oxygen signal, as shown in Fig. 8a. In the case of NO_x signal, maximum error observed in the 2-week period is 8%.

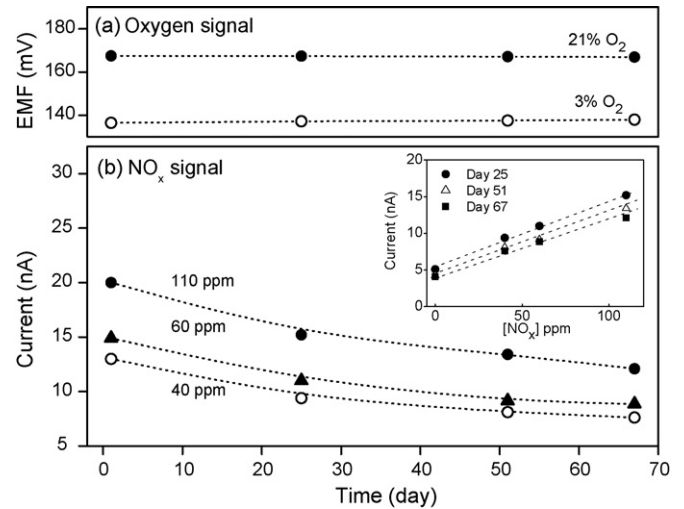


Fig. 7. Long-term stability for the amperometric configuration at 500 °C over a 67-day testing period. With a background of 3% oxygen, the NO₂ was changed from 0 ppm to 110 ppm everyday and the experiment repeated. Oxygen was also changed from 3% to 21% in a daily basis: (a) oxygen signal; (b) NO_x signal, insert: current vs. [NO_x] plots.

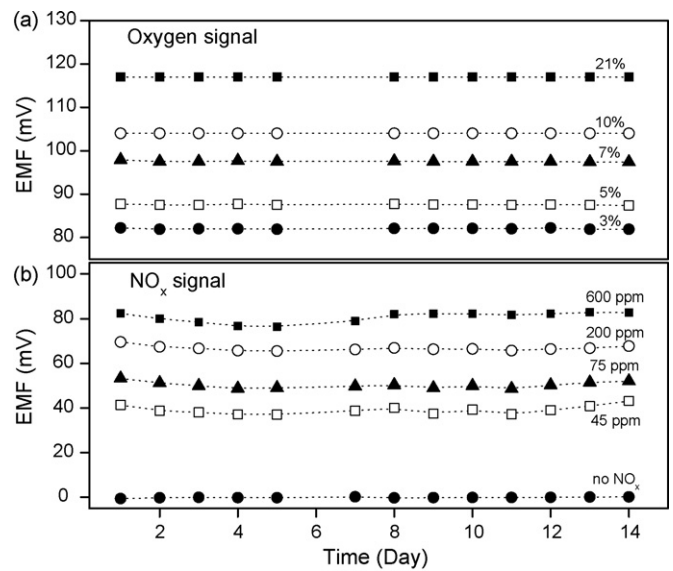


Fig. 8. Signal stability of the potentiometric configuration in 1–13 ppm NO₂ (in 3% O₂) and 3–21% O₂ over a 2-week test period: (a) oxygen signal; (b) NO_x signal (sensor at 600 °C, filter at 400 °C).

Table 1
Comparison of amperometric and potentiometric configurations

Configuration	Oxygen response	NO _x response	NO _x interference to O ₂	O ₂ interference to NO _x	Signal vs. pO ₂	Signal vs. [NO _x]
Amperometric	Slow ^a	Fast	None	~39% ^b	Logarithmic	Linear
Potentiometric	Fast	Fast	None	~10% ^b	Logarithmic	Logarithmic
Configuration	Working temperature	Size	Measurement	Temperature control		
Amperometric	500 °C	Very small (Fig. 1b)	Voltmeter + potential control electronic (<\$150)	One controller		
Potentiometric	Sensor at 600 °C Filter at 400 °C	Need filter housing	Voltmeter	Two controllers		

^a Can be improved by periodic electrochemical treatment.

^b Can be corrected by using Pd/PdO reference.

3.5. Comparison of two configurations

The differences between the potentiometric and amperometric configurations are summarized in Table 1. Generally, the potentiometric sensor provides more reliable signal for NO_x and less oxygen cross-interference. However, it requires a separate filter housing and two sets of temperature controllers. Both sensors were excellent for O₂ measurement. The smaller size of the amperometric sensor may make it attractive in certain applications.

4. Conclusions

An oxygen sensor with Pd/PdO internal oxygen reference was coupled with two electrochemical total NO_x measuring techniques to detect O₂ and NO_x simultaneously at high temperatures. A high-pressure and high-temperature bonding method was used to create an effective physical seal of the Pd/PdO powder contained in a YTZP chamber, followed by assembly of the amperometric and potentiometric NO_x sensing devices outside the chamber. PtY was used to obtain total NO_x measurements. The dissimilarity of catalytic activity between the sensing and reference electrodes was used to generate strong potentiometric signals. Both amperometric and potentiometric type sensors shows excellent O₂ signal stability and total NO_x response, though the potentiometric design provided more stable NO_x detection.

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Jules Routbort received a PhD in Engineering Physics from Cornell University in 1964. He was a postdoctoral fellow at the Cavendish Laboratory, Cambridge and at the Physics Department of Rensselaer Polytechnic Institute. He has been at Argonne National Laboratory since 1968 where his expertise is in the mechanical, diffusional, and fracture properties of materials. He is a Fellow of the American Ceramic Society, Iberdola Visiting Professor in the Condensed Matter Department of the University of Seville, adjunct Professor at Illinois Institute of Technology and North Carolina State University.

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Prabir K. Dutta received his PhD degree in Chemistry in 1978 from Princeton University. After four years of industrial research at Exxon Research and Engineering Company, he joined the Ohio State University, where currently he is professor of Chemistry. His research interests are in the area of microporous materials, including their synthesis, structural analysis and as hosts for chemical and photochemical reactions.