

# Technical report

## Solid-state electrolyte sensors for rebreather applications: a preliminary investigation

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### Key words

Rebreathers/closed circuit, oxygen, carbon dioxide, transducer, capnography, helium, trimix

### Abstract

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**Introduction:** Recently developed prototypes of zirconium dioxide and NASICON-based micro solid-state electrolyte oxygen (O<sub>2</sub>) and carbon dioxide (CO<sub>2</sub>) sensors were tested for their potential suitability in rebreathers. The O<sub>2</sub> sensor has a quasi-indefinite lifetime, whilst that of the CO<sub>2</sub> sensor is approximately 700 h. This is a preliminary report of a new technological application.

**Methods:** The O<sub>2</sub> sensor was tested in a small pressure chamber to a partial pressure of oxygen (PO<sub>2</sub>) of 405 kPa (4 bar). The CO<sub>2</sub> sensor was tested up to 10 kPa CO<sub>2</sub>. The response times to a step change of pressure were measured, and cross-sensitivity for helium tested using trimix. A rebreather mouthpiece was modified so that breath-by-breath gas recordings could be observed. Power consumption to heat the sensors was measured.

**Results:** The O<sub>2</sub> sensor demonstrated non-linearity, particularly above 101.3 kPa (1 bar) PO<sub>2</sub>, whereas the output of the CO<sub>2</sub> sensor showed an inverse logarithmic relationship. Cross-sensitivity to helium was observed. The mean t<sub>90</sub> response times were 90 (SD 10) ms for the O<sub>2</sub> sensor, and 100 (SD 10) ms for the CO<sub>2</sub> sensor. Breath-by-breath recordings showed slight damping of the CO<sub>2</sub> trace due to electronic filtering. Power consumption was 1.5–2 W per sensor.

**Conclusions:** The fast response times would allow accurate breath-by-breath measurement. Even though the O<sub>2</sub> sensor has a non-linear response, measurement is possible using multi-point calibration. Further design is necessary to allow trimix to be used as the diluent. A major disadvantage is the high power consumption needed to heat the sensors to high temperatures.

### Introduction

#### GAS MONITORING IN CLOSED-CIRCUIT REBREATHERS

While in open-circuit scuba diving, exhaled gas is usually vented into the water, in a rebreather it is returned to a counterlung, carbon dioxide (CO<sub>2</sub>) is removed in a 'scrubber' and metabolised oxygen (O<sub>2</sub>) is added from the supply tank. Information about the various types of rebreathers available can be found elsewhere.<sup>1,2</sup> In a closed-circuit rebreather (CCR), the partial pressure of O<sub>2</sub> (PO<sub>2</sub>) is usually kept at a constant level and only metabolised O<sub>2</sub> is replaced with fresh O<sub>2</sub> from the supply tank.<sup>3,4</sup> Oxygen is diluted in the breathing circuit with nitrogen or helium, or a combination of these gases. To maintain the PO<sub>2</sub> at a constant level, a control loop is needed. Therefore, electrochemical oxygen transducers, whose output signals are proportional to the PO<sub>2</sub>, are used as sensing elements. In a manually controlled rebreather, the diver reads the PO<sub>2</sub> from a display, then, if necessary, adds O<sub>2</sub> manually and/or, as in one model, adjusts the O<sub>2</sub> injection needle valve. In an electronically controlled rebreather, this control task is usually performed by a micro-controller and a solenoid valve.

CCRs have many advantages, such as:

- high gas efficiency, close to 100%;
- stealth (silent, bubble-free diving);
- warm, humid breathing gas;
- extended diving time;
- reduced decompression obligations due to optimised gas mixtures and decompression mixtures.

Risks and accidents associated with rebreathers have been discussed in detail elsewhere.<sup>5</sup> The most commonly identified systems failures that cause fatalities are:

- PO<sub>2</sub> outside of life-sustaining limits;
- high CO<sub>2</sub> levels.

In current rebreathers, O<sub>2</sub> is measured using galvanic sensors. The core element of this transducer is an electrochemical cell (fuel cell) consisting of two electrodes of dissimilar metals (cathode – a noble metal behind a diffusion barrier, made usually of Teflon; anode – lead) in contact with a liquid or semi-solid basic electrolyte, usually potassium hydroxide. The transducer output is linear in the range up to 203 kPa (2 bar) PO<sub>2</sub> with a slope of 40–70 mV bar<sup>-1</sup>. The most common and, for the diver, most dangerous failure mode of a PO<sub>2</sub> transducer is an incorrect electrical output for a given PO<sub>2</sub>.

In order to achieve maximum safety in the case of a PO<sub>2</sub> transducer failure, two main strategies are used:

- redundancy, using several transducers together with a voting logic;
- transducer validation.<sup>6,7</sup>

As well as having several failure modes, current PO<sub>2</sub> transducers also face disadvantages such as:

- short lifetime (< 18 months);
- relatively slow and temperature-dependent response (t<sub>90</sub> typically approximately 6 s at room temperature).

High CO<sub>2</sub> levels may be due to scrubber failure from various causes or check valve/direction valve failures that lead to 'pendulum breathing', in which the breathing gas no longer passes through the scrubber. Another important physiological rather than technical cause of hypercapnia is hypoventilation, especially at depth.<sup>8</sup> When CO<sub>2</sub> is chemically absorbed in the scrubber, heat and water are produced as by-products. Temperature at several points in the scrubber can thus be used for monitoring the scrubber and predicting its life-time.<sup>9</sup>

Although these technologies have been successfully implemented already, they provide feedback on the activity of the scrubber only, not on the inspired CO<sub>2</sub>. CO<sub>2</sub> measurement is usually performed using optical sensors. CO<sub>2</sub> shows strong (infrared) light absorption at 4.26 μm and 15 μm wavelengths. Most commercial optical CO<sub>2</sub> transducers are based on Beer-Lambert light absorption at 4.26 μm. They have fast response times, a quasi-indefinite lifetime and are inexpensive, and until recently have not been used in rebreathers. In a rebreather, where the humidity in the loop is approximately 100%, condensation may lead to the failure of optical transducers, as any additional absorption in the optical measurement path leads to unreliable readings.

A proof of concept of a gaseous CO<sub>2</sub> monitor for rebreathers based on an optical CO<sub>2</sub> transducer (Gas Sensing Solutions, UK) was first presented by Amphilogic Ltd (UK) in 2008 at the Birmingham dive show. Recently, a CO<sub>2</sub> transducer for rebreather applications, based on the same transducer module has been launched.<sup>10</sup> In order to overcome the problem with high humidity, hydrophobic membranes and a sponge are mounted in front of the transducer. However, such measures are likely to lead to increased response times. Another approach is based on an optical CO<sub>2</sub> transducer at the end of the exhale hose. A preliminary study of the performance of this CO<sub>2</sub> transducer, particularly in relationship to its positioning in the rebreather circuit was reported recently in this journal.<sup>11</sup>

## SOLID-STATE TRANSDUCERS FOR GAS MONITORING

An alternative to liquid electrolyte transducers is solid-state technology, based mainly on the ionic conductivity

of ceramic materials.<sup>12,13</sup> This technology has been used for many years in cars for combustion control (Lambda probe). At present, only yttrium oxide-doped zirconium dioxide (Zirconia, YDZ) is used in commercial sensors as a conducting solid-state electrolyte. Conductivity in YDZ requires high temperatures. Therefore, the sensor is heated by an electrical resistance to reach an operational temperature of about 650°C. The O<sub>2</sub> transducers typically used in cars are not applicable for a rebreather, mainly because of their power consumption and size; they also require a reference chamber. In automotive applications, ambient air is taken as a reference, which is, of course, impossible for an underwater breathing apparatus. Micro manufacturing allows miniaturisation of such transducers. An overview of micro solid-state gas sensors can be found elsewhere.<sup>14</sup> A suitable ionic conductor for a CO<sub>2</sub> sensor is sodium super-ionic conductor (NASICON).

Recently, micro solid-state sensors have been developed in Germany for O<sub>2</sub> measurement in low earth orbit and tested in the International Space Station (ISS). These are based on the amperometric principle, where the electrical current is proportional to PO<sub>2</sub> flow through the sensor membrane when the O<sub>2</sub> is 'pumped' from one electrode to the other by an applied voltage. The reaction proceeds in several steps. Firstly, molecular O<sub>2</sub> is transformed to oxygen ions at the cathode. Then these ions migrate through the solid electrolyte towards the anode, where they recombine again into O<sub>2</sub> molecules. In addition, a diffusion barrier limits the O<sub>2</sub> flux to the cathode. If the flux limitation is high enough, the PO<sub>2</sub> at the cathode is very low. In this particular case, the measured current is limited by the diffusion of O<sub>2</sub> to the cathode and, therefore, a linear dependence on the ambient PO<sub>2</sub> is achieved as long as 'crowding' of O<sub>2</sub> at the anode is avoided. Unlike the typical Lambda probe in a car, these types of sensors do not require a reference but instead require an efficient O<sub>2</sub> clean up from the anode.

Based on the test results from the ISS, a new project was started to develop micro solid-state O<sub>2</sub> and CO<sub>2</sub> transducers for respiratory analysis using this sensor technology. This is supported by the European Space Agency within the framework of the Microgravity Application Promotion Program (MAP Project No. AO-99-058; ESTEC Contract No. 14350/01/NL/SH). The focus for these investigations is to design and test a new miniaturised transducer system that enables simultaneous in-situ measurement of O<sub>2</sub>, CO<sub>2</sub> and volume/mass gas flow rates. This allows for direct in-situ measurements inside the mask during cardio-respiratory studies of astronauts, athletes and medical patients. Since the in-situ measurements are performed with the transducer inside the mask, one measures these parameters in the main gas stream, avoiding any of the problems of side-stream measurement. The main characteristics of the transducers are listed in Table 1.

The aim of the present investigation was to validate the

**Table 1**  
Specifications of the micro solid-state O<sub>2</sub> and CO<sub>2</sub> sensors

### Oxygen

- Yttria-doped Zirconia (YDZ)
- Amperometric principle
- Operational temperature approx. 650°C (high temperature is required for linearity)
- 1.8 W power supply (1 W supply in principle possible, but more difficult to fabricate)
- Accuracy of < 2% volume at ambient pressure should be possible after 3 s heating
- Accuracy of +/- 0.1% at ambient pressure after 3–4 min
- Quasi-indefinite lifetime

### Carbon dioxide

- NASICON-based
- Potentiometric transducer principle (with a solid reference), thus the electromotive force (EMF) is measured
- Operational temperature approximately 550°C
- 1.8 W power supply
- Operational lifetime approx. 700 h

performance of the micro solid-state sensors in conditions similar to those existing in a rebreather (PO<sub>2</sub> 15–203 kPa, PCO<sub>2</sub> 0–6 kPa). The response times of the transducers were of considerable interest as, due to their small size, they can be integrated into a mouthpiece, which theoretically allows breath-by-breath analysis of the inhaled and exhaled gases. Of particular interest was performance of the present transducer design when used in conjunction with the gas mixtures characteristic for the diving environment, i.e., O<sub>2</sub> in nitrogen (N<sub>2</sub>) and helium (He)-N<sub>2</sub> admixtures.

### Methods

A test bench, which included a small pressure chamber (approximately 10 cm<sup>3</sup> volume) that can be pressurised up to 10 bar (Figure 1), was designed for measuring the performance of the solid-state sensors. Gas was supplied to the chamber via an adjustable needle valve from a 4-L cylinder. The supply pressure was reduced to 8 bar over ambient with a commercially available first stage regulator [DS4, Apeks]. A mechanical gauge and a calibrated absolute pressure transducer [MPX5700AP, Motorola] were used to measure the pressure inside the chamber. A pressure regulator [Porter USA] kept the pressure at a manually selectable level (0–4 bar above ambient). Alternatively, a quarter-turn shut-off valve could be mounted instead of the pressure regulator. In the closed position, the pressure inside the chamber will rise to the output pressure of the first stage regulator; opening the valve leads to a sudden loss of pressure, allowing measurement of the response time of the sensors to a step change in pressure. All components

are either oil/grease free or are assembled using oxygen-compatible grease.

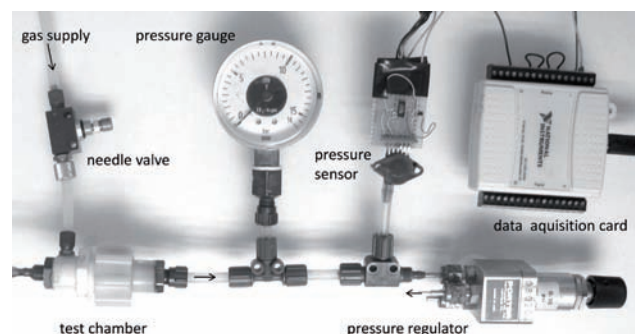
The O<sub>2</sub> sensor output was loaded with a 1 kΩ resistor. The voltage was then amplified with an operational amplifier (AD8630, Analog Devices). The CO<sub>2</sub> sensor signal was buffered with a precision operational amplifier (AD8601, Analog Devices) with a very low input bias current, typically 0.2 pA, making it the ideal choice for signal conditioning of a CO<sub>2</sub> sensor with a high output impedance. The conditioned sensor signals and the output signal of the pressure transducer were sampled with a 12-bit data acquisition card (USB-6008, National Instruments).

The solid-state sensors were heated by a printed platinum resistor (approximately 3 Ω at room temperature and 9 Ω at 700°C). For temperature control and transducer readout, custom electronics were developed, based on an 8-bit microprocessor (ATXmega16A4, Atmel). The temperature control programme essentially measures the heating current with the internal AD converter of the microprocessor, calculates the resistance and then sets the pulse-width modulation based on a proportional integral algorithm (PI control). Another two analog inputs of the microprocessor are used to sample the signal from the O<sub>2</sub> and the CO<sub>2</sub> sensors. These data, together with the heater currents and the calculated resistances, are transmitted via a serial interface to a PC at a sampling rate of 50 Hz. Data acquisition, conditioning and visualisation were performed using the software package Lab View™ 8.5 (National Instruments, Austin, Texas, USA).

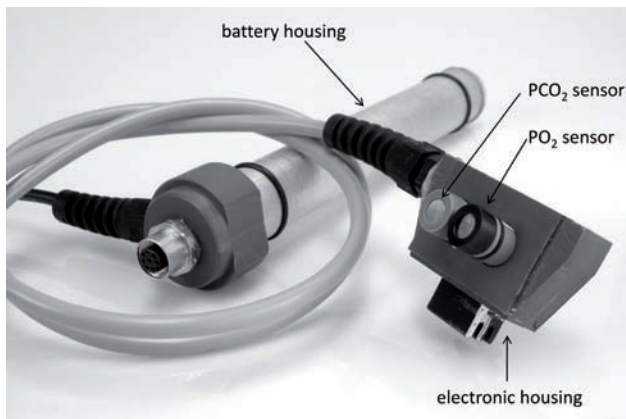
### EXPERIMENTAL PROCEDURES

Four separate tests were conducted. First, the O<sub>2</sub> sensor was installed in the test chamber and the gas flow was set to 0.5 L min<sup>-1</sup> STPD. The chamber was gradually pressurised with O<sub>2</sub> in 0.1 bar steps from 1 to 4 bar absolute pressure. For O<sub>2</sub> values below 1 bar (101.3 kPa) the chamber was pressurised with air. To assess He cross interference, the O<sub>2</sub> sensor was exposed to trimix 21/50 from 1 bar to 5 bar absolute pressure.

**Figure 1**  
Test bench for hyperbaric characterisation of micro solid-state O<sub>2</sub> and CO<sub>2</sub> sensors



**Figure 2**  
PO<sub>2</sub> and PCO<sub>2</sub> solid-state transducer module, electronics and rechargeable battery pack



The heating power was about 2.5–2.6 W.

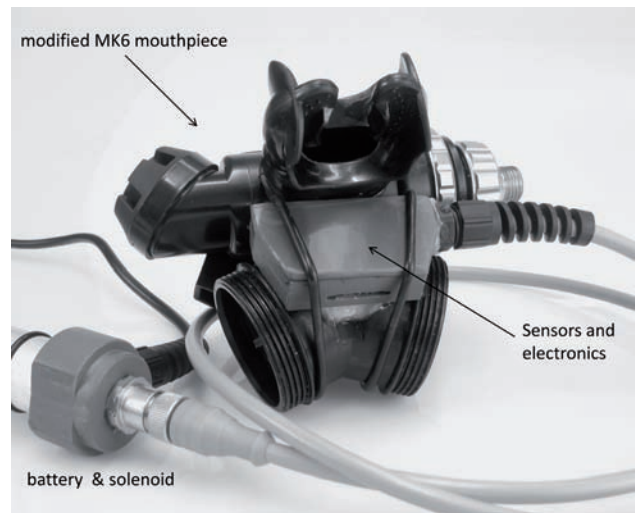
For response-time measurement, the pressure regulator was substituted with the quarter-turn shut-off valve and the chamber was pressurised with air at a gas flow of 0.1 L min<sup>-1</sup> STPD. After reaching 7 bar, the shut-off valve was opened leading to a rapid decrease in pressure. Pressure signals and transducer signals were recorded and t<sub>90</sub> was estimated using Microsoft Excel®.

Assessment of the signal response of the CO<sub>2</sub> sensor to CO<sub>2</sub> partial pressures (PCO<sub>2</sub>) from 0–10 kPa was performed by gradually increasing the pressure inside the chamber using an air/CO<sub>2</sub> mixture with an FCO<sub>2</sub> of 1%. The CO<sub>2</sub> sensor is a potentiometric transducer, thus the output is logarithmic (Nernst equation). The response time of the sensor was measured in the same manner as for the O<sub>2</sub> sensor but using the 1% CO<sub>2</sub> gas mixture.

For the third test, a small module, measuring 22x10x5 mm and containing CO<sub>2</sub> and O<sub>2</sub> sensors and the required electronics, was built into a custom-made PVC housing filled with silicone gel. As well as transducer control and readout, it was also equipped with a solenoid controller, a display interface and a serial output for real-time data transmission to a PC (Figure 2).

A commercially available rebreather mouthpiece (Mk6, Poseidon, Sweden) was modified with a support that allowed fitting of the transducer module onto the mouthpiece such that the sensors sat between the mushroom valves (Figure 3). The module was connected via a water- and pressure-proof cable to the battery housing. It was also possible to install into the head of the battery housing a micro-solenoid for O<sub>2</sub> partial pressure (PO<sub>2</sub>) control. As reference, a commercially available main-stream infrared CO<sub>2</sub> analyser (IRMA, Phasein, Sweden) was fitted between the bite and the rebreather mouthpiece.

**Figure 3**  
Transducer module fitted onto a modified rebreather mouthpiece

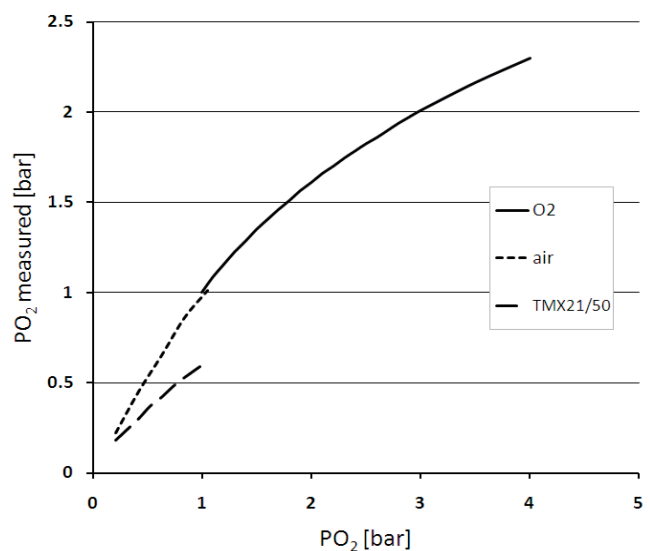


In a final study to measure breath-by-breath PO<sub>2</sub> and PCO<sub>2</sub>, a subject (35-year-old male) breathed through the rebreather mouthpiece. In all studies, repeat observations were made. The CO<sub>2</sub> transducer was two-point calibrated with air (assuming 350 ppm CO<sub>2</sub>) and with air containing 1% CO<sub>2</sub>.

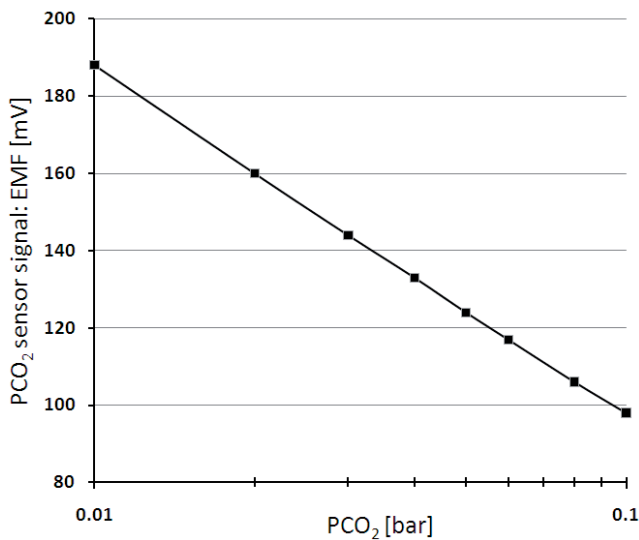
## Results

Figure 4 illustrates the results of the O<sub>2</sub> sensor characterisation with 100% O<sub>2</sub>, air and Trimix 21/50. As the sensor elements studied were prototypes only mean values are shown at this

**Figure 4**  
Illustrative characterisation of the O<sub>2</sub> sensor with O<sub>2</sub>, air and trimix 21/50



**Figure 5**  
Characterisation of the CO<sub>2</sub> sensor from 0.01 to 0.1 bar PCO<sub>2</sub> with a 1% CO<sub>2</sub>/air mixture



stage. The sensor signal is non-linear, particularly at a PO<sub>2</sub> above 1 bar. The heating power was fairly constant with 1.65 W at 1 bar and 1.75 W at 4 bar chamber pressure. Using the same calibration factor as in the previous test, the sensor output using trimix was lower than expected. Instead of 0.21 bar O<sub>2</sub> at a chamber pressure of 1 bar, the observed PO<sub>2</sub> was 0.18 bar. At 5 bar chamber pressure, equal to a PO<sub>2</sub> of 1.05 bar, the observed PO<sub>2</sub> was only 0.65 bar. In comparison to the previous tests with 100% O<sub>2</sub>, the required heating power was increased by approximately 40%.

Because the PCO<sub>2</sub> transducer is based on a potentiometric principle, the EMF produced is inversely related to PCO<sub>2</sub> in a logarithmic fashion, shown as a semi-log plot in Figure 5. The required heating power was 1.7 W and was fairly constant from 1 to 10 bar absolute pressure. The sensitivity was found to be -90 mV per decade (EMF @ 0.1 bar – EMF @ 0.01 bar PCO<sub>2</sub>).

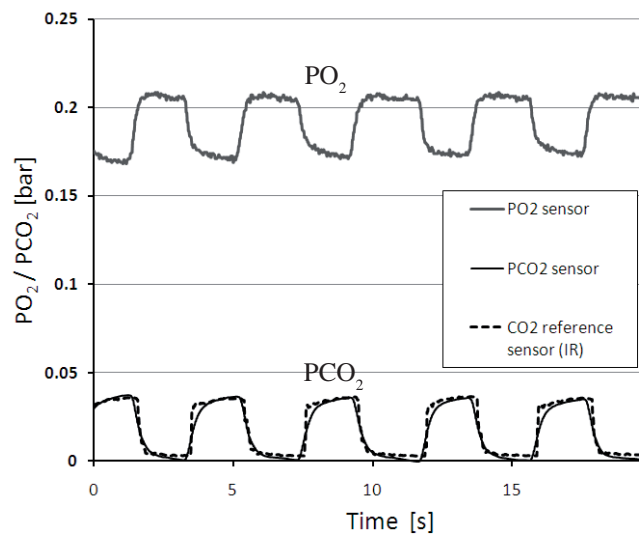
Using the quarter-turn valve, the pressure inside the chamber fell from 7 to 1 bar within approximately 25 ms. The mean t<sub>90</sub> response time of the O<sub>2</sub> transducer was 90 (SD 10) ms, and that of the CO<sub>2</sub> transducer was 100 (SD 10) ms (*n* = 10).

Figure 6 shows an example recording of the breath-by-breath PO<sub>2</sub> and PCO<sub>2</sub> outputs and that of the reference CO<sub>2</sub> monitor. The average end-tidal CO<sub>2</sub> difference between the solid-state PCO<sub>2</sub> transducer and the infrared reference transducer over a 3 min recording period was found to be 0.26 (0.06) kPa.

## Discussion

The recently developed prototypes of solid-state O<sub>2</sub> and CO<sub>2</sub> sensors seem to be suitable for applications in an O<sub>2</sub>/

**Figure 6**  
Sample breath-by-breath O<sub>2</sub> and CO<sub>2</sub> recording from the transducer module in the rebreather mouthpiece; measurements from mainstream CO<sub>2</sub> monitor also shown



N<sub>2</sub> rebreather. The main advantages are:

- very long lifetime;
- high accuracy;
- mechanically robust;
- insensitive to moisture/humidity (heated up to 600°C);
- very fast response time, that allows assessment of PO<sub>2</sub> and PCO<sub>2</sub> values in inhaled and exhaled gas.

The main disadvantages are a high power consumption and non-linearity.

The current sensor prototype was developed for O<sub>2</sub> measurements below 1 bar PO<sub>2</sub>. Above 1 bar PO<sub>2</sub>, the sensor is non-linear, and so requires multi-point calibration to enable accurate measurements. In comparison to traditional wet-electrochemistry-based O<sub>2</sub> transducers containing a consumable lead anode, the micro solid-state technology is non-consuming and theoretically the signal response of the sensor does not change over time. Thus, a one-time factory calibration is conceivable. Even though this would obviate the need for pre-dive calibration, we believe that a pre-dive calibration with normobaric O<sub>2</sub> is a necessary safety measure to validate transducer function.

Further development will include optimisation of the diffusion layer in terms of thickness and density to improve linearity up to a PO<sub>2</sub> of 2 bar. An alternative approach to improving linearity might be three-electrode configuration together with a potentiostat, which is an electronic circuit used to maintain the voltage on one electrode at a defined level by using a third electrode, usually referred to as the reference electrode. Long-term aging effects caused by thermal stress (because of fast heating and cooling

of the transducer elements) were not investigated in this preliminary study.

The typical heating power required for a sensor is 1.7 W. Even with He in the diluent, no higher values than 2.5 W heating power were recorded. A standard 3.3 V, 2000 mA h<sup>-1</sup>, rechargeable battery would allow operation of a single transducer for approximately 3 h with air as the diluent, which is sufficient for a rebreather for recreational purposes. Promising approaches to reduce the required heating power are based on further miniaturisation of the sensor element, the use of different ionic conductors that work at lower temperatures and smart system design with an emphasis on isolation and packaging of several transducers in one device.<sup>15,16</sup>

The PO<sub>2</sub> transducer in its current version does not appear suitable for use in a rebreather with He gas mixtures as the addition of He to the diluent decreased the sensitivity of the sensor. We believe this is due to an increased temperature gradient that is created by the presence of He. He has a much higher heat conductivity than air (or O<sub>2</sub> or N<sub>2</sub>), which causes an increased heat loss at the sensor surface. To compensate for this, more power is necessary. The heating resistor, which is also used for sensing the sensor temperature, is located in the first layer above the sensor substrate. The increased heating power required results in a larger temperature gradient across the sensor. The diffusion layer, which is the current-limiting component, is distant from the resistor layer and, because of the temperature gradient, is most likely colder when He is used compared to air, which then leads to a reduced output signal in the presence of He. Possible solutions could be the integration of a temperature-sensing element in front of the sensor surface or measures for calibration of the transducers for different He fractions.

Figure 6 shows the results of the comparison of the PCO<sub>2</sub> transducer and the reference infrared-based CO<sub>2</sub> transducer. Even though the O<sub>2</sub> and CO<sub>2</sub> sensors have a short t<sub>90</sub> response time and can be used for breath-to-breath gas analysis (Figure 6), the signal response of the solid-state PCO<sub>2</sub> transducer is slower than that of the reference monitor. This is because of an internal low-pass filter in the electronics. The filter frequency will be changed in the next hardware revision to allow full exploitation of the sensor's capabilities. It should be noted, that a CO<sub>2</sub> calibration using a precision gas with a higher CO<sub>2</sub> content might lead to more accurate values. Whether such a calibration can be carried out as a one-time factory calibration, or has to be performed at regular intervals by the user or a service centre, has yet to be assessed by long-term studies.

## Conclusions

This paper presents preliminary data describing the characteristics of micro solid-state sensors for O<sub>2</sub> and CO<sub>2</sub> monitoring being developed for gas monitoring in diving

rebreathers. These data confirm the potential successful application of this technology. Issues of non-linearity and helium cross-sensitivity require further investigation. Future testing will also address vulnerability of the heated transducers to heat shock and immersion in water, aging effects (especially from thermal stress) and how different breathing patterns influence transducer output on a breath-by-breath basis.

## Acknowledgements

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## Continuing professional development

### Important notice: New Continuing Professional Development Coordinator needed

Associate Professor Michael Bennett is now the Chief Examiner for the ANZCA Certificate in Diving and Hyperbaric Medicine. He has been advised by the College that he will not be able to continue to produce the CPD exercises in future issues of *Diving and Hyperbaric Medicine* because of a perceived potential conflict of interest. Therefore, we are seeking a volunteer to continue the coordination of these.

**ANZCA SIG members: time to front up and support your College, this journal and your colleagues!**

**Those interested should contact the SIG Chairperson, Dr Margaret Walker, E-mail: <margaret.walker@dhhs.tas.gov.au>, or Associate Professor Michael Bennett, E-mail: <M.Bennett@unsw.edu.au>.**

This notice has now been in the last three issues of the Journal. Regrettably, nobody from SPUMS, and more particularly from the ANZCA SIG, working in hyperbaric medicine, has put themselves forward. As a result, there is no CPD item in this issue. Fortunately, we have several EUBS members who have volunteered their services for the next several issues. However, this does not resolve the problem of a coordinator for ANZCA SIG members. Without a College moderator, future CPD exercises regrettably will not be available as MOPS points on the ANZCA programme.

*Michael Davis, Editor*