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Filterless optical oxygen sensor based on a CMOS buried double junction photodiode

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Abstract

We present a custom CMOS IC with a buried double junction (BDJ) photodiode to detect and process the optical signal, eliminating the need for any off-chip optical filters. The on-chip signal processing circuitry improves the desired signal extraction from the optical background noise. Since the IC is manufactured using standard commercial fabrication processes with no post-processing necessary, the system can ultimately be low cost to fabricate. Additionally, because of the CMOS integration, it will consume little power when operating, and even less during stand-by.

Keywords

Oxygen sensor; CMOS photodetectors; Filterless color detection; DNA–CTMA sensor

1. Introduction

Key trends in biosensing focus on miniaturization, high sensitivity, ease of integration, and low cost designs. Optical sensing is attractive because it can be minimally invasive and allows standoff detection. In the clinical arena, optical sensors can be used to monitor, for example, oxygen [1,2] and glucose [3], amongst other analytes. Specifically, luminescence-based sensing methods are often favored because they can provide excellent response to specific analytes and require minimal components [4]. A subset of these methods are quenchometric-based and are used to determine the concentration of quencher molecules; the measured luminescence intensity is a function of the quencher (analyte) concentration [5–7]. One example of this is oxygen sensing in which the intensity change from a single luminescent sensor is measured to determine the oxygen concentration [8]. Another approach achieves improved performance by using an array of luminescent sensors each

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with a different sensitivity [9]. Yet another approach is to fine tune the sensor system so that it is intended to operate over a small range, such as the luminescence lifetime-based system described in [10]; this system provides good performance over a 0–20% range of oxygen concentration.

In luminescence-based sensing systems, relevant optical signal detection is a critical issue. Photomultiplier tubes (PMT) provide excellent results because they offer high gain which is useful for measuring small optical signals. However, PMTs use high voltages, are large and fragile, heavy, and expensive. Replacing the PMT in optical sensor applications is essential for realizing handheld, portable devices. A suitable alternative detection method must achieve sufficiently high signal to noise ratios (SNR) through electronic and/or optical means. CCD and CMOS cameras are being used in place of PMTs in many systems [8]. Unfortunately, these imagers alone are not sufficient to achieve the SNRs needed. Normally, the excitation source intensity is much greater in comparison to the sensor element emission, so there is a need to isolate (i.e., filter) the emission from the excitation to enable unbiased signal detection. For example, one system uses a CMOS imager (with a Bayer filter) and two polarizers to isolate the emission and excitation signals [11]; the green excitation is polarized such that only the luminescence passes through the second polarizer in front of the detector. Another method embeds a dye into a polymeric microfluidic device that acts as a long-pass optical filter [12].

In this paper, we present a CMOS-based system for oxygen detection, which is shown in its simplest form in Fig. 1. The sensor is based on an oxygen-responsive luminophore, tri-(4,7'-diphenyl-1,10'-phenanthroline) ruthenium (II) ($[\text{Ru}(\text{dpp})_3]^{2+}$), sequestered within a hybrid xerogel [13–15] with the addition of an organic-soluble DNA material combined with a cationic surfactant such as cetyltrimethylammonium (CTMA), and a custom, optical filter-free CMOS IC for optical detection. The IC has a buried double junction photodiode (BDJ) combined with on-chip signal processing circuitry which eliminate the need for external optical filters and circuitry. The addition of the DNA + CTMA to the $[\text{Ru}(\text{dpp})_3]^{2+}$ results in an increased excited-state lifetime because of the DNA intercalation which attenuates one or more non-radiative processes. DNA–CTMA is chosen because it is an organic-soluble material that is easily incorporated in the xerogel. The result is a more efficient luminescence-based sensor.

2. Material and methods

2.1. Reagents and supplies

The following reagents were used: $[\text{Ru}(\text{dpp})_3]\text{Cl}_2 \cdot 5\text{H}_2\text{O}$ (GFS Chemicals); tetraethylorthosilane (TEOS) (99.9%) and *n*-octyltriethoxysilane (C8-TriEOS) (>95%) (Gelest); HCl (ACS grade) (J.T. Baker); and EtOH (200 proof) (Decon Laboratories). All reagents were used as received unless mentioned otherwise. Deionized water was prepared by using an AmeriWater purification system (Metro Group) to a specific resistivity of at least 18 M Ω cm.

2.2. DNA material preparation

The DNA material incorporated in the xerogel sensor was processed from marine salmon sperm; a typically salmon fishing industry waste product. To obtain high optical quality thin films, a cationic surfactant such as cetyltrimethylammonium (CTMA) is mixed with the DNA, resulting in a complex that is soluble in butanol [13]. The molecular weight of the biomaterial can be adjusted to suit the specific application by using sonication; in this work, the DNA–CTMA is formed with a molecular weight of approximately 300 kDa.

2.3. Xerogel and fiber sensor preparation

A sol was prepared by mixing, in order, TEOS (1.45 mL, 6.5 mmol), C8-TriEOS (2.05 mL, 6.5 mmol), EtOH (2.52 mL, 44 mmol) and HCl (0.800 mL of 0.1 M HCl, 0.08 mmol). This sol was capped and magnetically stirred under ambient conditions for 1 h. A luminophore-doped sol was prepared by mixing 0.273 mL of 25 mM $[\text{Ru}(\text{dpp})_3]^{2+}$ (in EtOH) with 6.82 mL of the aforementioned sol. Sol blanks were prepared by omitting $[\text{Ru}(\text{dpp})_3]^{2+}$. These sols were capped and mixed for 2 min by a touch mixer and stored in the dark under ambient condition for 24 h before use [16].

The oxygen responsive xerogel sensors described in Section 4 are formed at the distal end of an optical fiber (Thorlabs, BFH37-200, 200 μm core, 0.37 NA) by removing a 2 mm segment of cladding and dip coating the optical fiber into the aforementioned sols. The optical fiber serves to guide the excitation to the xerogel layer and deliver the emission signal to the detector. The proximal optical fiber end is connected to the optical fiber port using a SMA connector.

2.4. Xerogel sensor characterization

Electronic absorbance spectra were recorded by using a Shimadzu UV-3101PC UV–VIS–NIR spectrophotometer. The sensor photoluminescence (PL) response was measured by using a Shimadzu RF-5301 PC spectrofluorimeter ($\lambda_{\text{ex}} = 450 \text{ nm}$). Fig. 2 presents the typical absorbance and emission spectra for $[\text{Ru}(\text{dpp})_3]^{2+}$ within a xerogel/DNA–CTMA film.

3. Chip design and fabrication

3.1. BDJ operation

CMOS technology is used here because sensing and signal processing can be integrated within a single chip. Specifically, photodetectors are easily integrated with CMOS circuitry, which has enabled the growth in CMOS-based digital cameras. Traditionally, image sensors that produce a color-dependent response require optical filters, such as Bayer filters. Often, this requires a delicate post-fabrication process to attach the optical filters to the chip, which means that any filter replacement is very difficult. Also, although optical filters can help to separate the excitation from the emission, they also attenuate the desired signal.

Our sensor chips use photodetectors that require no special fabrication steps and they enable us to simultaneously extract color information. The operation of these buried double

junction (BDJ) detectors is based on depth and wavelength dependent absorption through the silicon layer atop the detection element [17] and is given by Eq. (1) and plotted in Fig. 3.

$$I(x) = (1 - R)I_0 \exp(-\alpha x) \quad (1)$$

In this equation, x is depth into the silicon layer, R is the reflection coefficient of air-silicon interface, I_0 is the incident light intensity, and α is the silicon absorption coefficient. The absorption coefficient across the visible to near infrared region decreases with increasing wavelength, so longer wavelength light penetrates deeper into the device before being absorbed by silicon. This property can be exploited to extract different wavelength signals from different depths within the device [18]. In our chips, we create three different silicon layers (P+, N-well and p-substrate) at different depths along the same vertical path. This implies there are two distinct depletion regions (one at each pn junction), hence two locations for optoelectronic current generation. A BDJ has previously been used to realize color sensitive detection [8], but this strategy has not been used for chemical sensing applications.

3.2. BDJ circuitry

Fig. 4 shows the BDJ structure and the associated output currents for two diodes within the BDJ structure. The two vertically stacked diodes (Fig. 4a), enable us to simultaneously measure intensity and wavelength information. Proper examination of the output currents from the diodes allows us to categorize/assess the input optical signal. As seen in Fig. 4b, the current *ratio* from each junction produces a monotonic output with respect to wavelength. In addition, by controlling the voltage on each pn junction, which changes the depletion region width, it is possible to tune the output current for different wavelengths, which provides additional control over the detector response [20]. This control can be used to finely select and/or tune wavelengths of interest without switching optical filters.

Although the BDJ can be fabricated using standard commercial CMOS processes [21], the layer depth is fixed [19]. Therefore, to generate wavelength dependent photocurrents, we use the layers which are available in standard CMOS processing to form the two pn junctions. The *shallower* junction within the BDJ is formed by p-active and N-well layers. The *deeper* junction within the BDJ is formed by the N-well and p-substrate layers [20]. In the current design, the BDJ photodiode active area is $78 \mu\text{m} \times 78 \mu\text{m}$.

The color detector topology is shown in Fig. 5. The two input stages are used for clamping the two pn junction bias voltages for a pixel. Thus, by controlling the bias voltage through these stages, one can control the depletion region width for each diode, which can be used to tune the wavelength selectivity as described previously. The output currents are copied (mirrored) to use for calculations in later stages. As the current from the common BDJ center node is the total current through both photodiodes, a subtraction circuit removes I_1 from $I_1 + I_2$ to determine the current through the lower junction, I_2 . To extend the ability to operate over a larger intensity range impinging upon the pixel, a logarithmic current to voltage converter circuit is used. While mismatch between these two circuits can be a

problem, proper design and symmetric layout can minimize this. The final stage is a differential amplifier that produces an output that is the difference of V_2 and V_1 , which is the logarithmic value of I_2/I_1 . The circuit schematic for this is shown in Fig. 6. To summarize:

$$\text{Wavelength } (\lambda) \propto \log \left(\frac{I_2}{I_1} \right) \quad (2)$$

$$\log \left(\frac{I_2}{I_1} \right) = \log (I_2) - \log (I_1) \quad (3)$$

The generation of photocarriers at each junction depends on all wavelengths of light incident on the BDJ, so the upper photocurrent is mostly generated by shorter wavelengths, but longer wavelength light also contributes some, and vice versa for the lower junction. Because of this, when the input light is not monochromatic, the resulting output ratio depends on the mix of the wavelengths present, and as this mix changes (say, goes from mostly blue to a mix of orange and blue, as is the case in the system discussed here), the current ratio changes. In the case of purely monochromatic light, an ideal system produces no change in output voltage because the ratio of the currents remains constant.

3.3. Calibration

The chip was fabricated in the ON Semi 0.5 micron process available through MOSIS (<http://www.mosis.com/>). Calibration is performed on the sensor chip alone using a light source coupled to a wavelength calibrated monochromator (full-width at half-maximum (FWHM) of approximately 0.2 nm). This strategy is used to characterize the BDJ and processing circuitry response for known wavelengths. The incident intensity on the detectors is wavelength dependent, and the average intensity is approximately $1 \mu\text{W}/\text{mm}^2$. The wavelength step size is 10 nm for these initial experiments.

Fig. 7 shows the BDJ sensor response between 350 and 880 nm. Over this range, the output voltage changes from 0.814 V (at 350 nm) to 3.637 V (at 880 nm). Because the intended use of this chip is to detect the luminescence from a $[\text{Ru}(\text{dpp})_3]^{2+}$ sensor element, it is useful to examine the response for a narrower wavelength range centered around the $[\text{Ru}(\text{dpp})_3]^{2+}$ emission maxima (ca. 610 nm) (Fig. 8). These data are obtained by scanning in 0.5 nm increments between 600 and 620 nm. The BDJ output is measured for repeated experiments, with the variability shown by the error bars in Fig. 8. These error bars show the maximum and minimum values measured for each wavelength, so this chip can easily produce a distinguishable output change for 0.5 nm changes in input wavelength over this range. The voltage response is consistent and is repeatable.

4. CMOS-based oxygen sensor characterization setup

The BDJ-based oxygen sensor system is shown in Fig. 9. O_2 and N_2 are mixed within a custom gas handling manifold using separate inlets and outlets controlled by individual flow

meters (Gilmont Instruments, GF 5542-1500), and the O₂ concentration is adjusted by changing its relative percentage with respect to the N₂ concentration. Measurements are performed in a small, light tight chamber with an inlet connected to the gas mixing manifold. The optical fiber tip used for sensing is inserted into the chamber; the blue excitation light is coupled into the proximal end of the optical fiber. A blue LED ($\lambda_{\text{peak}} = 468$ nm, Radio Shack, Model: 276-316) is used as the excitation source and driven at 4.8 VDC. The optical fiber output is focused onto the BDJ sensor chip. A long-pass filter is *not* used in the optical path.

The CMOS BDJ sensor chip is powered with a single 5 VDC supply, and the total average chip power consumption during operation is <200 μ W. Data are obtained using the BDJ sensor chip with no filters or off-chip circuitry. A PMT is used to provide a baseline comparison. The PMT power consumption is several orders of magnitude greater in comparison to the CMOS BDJ and the PMT requires an optical filter and off detector signal processing.

5. Results and discussion

We verified the BDJ sensor chip functionality by comparing the BDJ chip Stern–Volmer (S–V) plot (I_0/I vs O₂, I_0 = intensity in the absence of O₂, I = intensity at any O₂ concentration) to one recorded by using a PMT in concert with an optical filter (Fig. 10). This is not a straightforward comparison because the BDJ chip produces an output voltage that is a ratio of the logs of currents. A S–V plot of the voltage ratio is not directly meaningful, since a typical S–V plot is a ratio of responses that are linear functions of the optical signal. To obtain data for proper comparison, we used the output voltages from our experiments combined with TSPICE circuit simulations to obtain the actual currents. The current used to generate the BDJ Sensor chip data points is the top junction current only (I_1 in Figs. 5 and 6a, prior to any circuitry). Inspection of the data in Fig. 10 shows that the responses are linear ($r^2 > 0.99$) with statistically equivalent ($p = 0.001$) Stern–Volmer quenching constant (i.e., slopes) of $0.074 \pm 0.04\% \text{O}_2^{-1}$.

To further compare the new BDJ platform performance we recorded the direct voltage from a CMOS photodetector on the chip (see Fig. 11). In this, case a 560 nm long-pass optical filter was used to remove the excitation signal. This filter also removed some of the [Ru(dpp)₃]²⁺ photoluminescence and it introduced additional reflective losses. We calculate the signal to noise ratio (SNR) for the various sensor methods by determining the ratio of the desired fluorescence signal response to the background (excitation and other stray light) optical signal. Under 100% O₂, the SNR is 14 dB, 12 dB and 3.52 dB for a PMT with a filter, the BDJ sensor without any filter, and a CMOS sensor with a filter, respectively. Thus, BDJ platform exhibits substantially better SNR in comparison to a conventional CMOS detector with a filter. Of course, it is possible to improve a non-BDJ CMOS platform further [16], but with the BDJ, we can clearly achieve very good results with no additional components.

6. Conclusion

We have shown that a custom filter-free BDJ-based photodetector chip is an excellent alternative to a PMT for low-power, portable systems, and that its performance is far superior in comparison to a CMOS photodiode with external color filter. The commercially fabricated BDJ chip is very small, consumes very little power and is ideally suited to photoluminescence detection.

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Carrie M. Bartsch works for the Air Force Research Laboratory as a technical co-lead of the Sensors Directorate Biotronics Program and as the program lead for the Electro-Optics Components Branch Biotronics Electronics Initiative. She has co-authored two book chapters along with numerous journal articles and conference proceedings. Dr. Bartsch earned her Ph.D. in electrical engineering from the University of Dayton in 2007, her M.S.E. in EE from the University of Michigan in 2002, and her B.S.E.E. from the University at Buffalo in 2000. Dr. Bartsch has characterized the electrical properties of biopolymers from DC to microwave frequencies. She also demonstrated the first use of DNA-biopolymers as a semiconducting layer in field effect transistors. In addition, Dr. Bartsch has a wide ranging background in optical and electronic devices.

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Fig. 1.

Basic sensor system setup. Light from the blue LED excites the luminophores in the xerogel, causing them to emit orange light upon relaxation. The excitation and fluorescence are detected by the sensor chip, which responds differentially to the orange light.

Fig. 2.
Absorbance and emission spectra for $[\text{Ru}(\text{dpp})_3]^{2+}$ within a xerogel/DNA-CTMA film.

Fig. 3. Silicon wavelength dependent absorption coefficient (α) and penetration depth (x) from Eq. (1).

Fig. 4. Characteristics of BDJ structure in a standard CMOS technology. I_1 is the photogenerated current through the upper pn junction and I_2 is the photogenerated current through the lower pn junction. (a) Side-view of BDJ structure. (b) Current responses of BDJ diodes. From [19].

Fig. 5.
BDJ signal processing topology components on the CMOS sensor chip.

Fig. 6. (a) BDJ sensor signal processing circuit schematic. (b) Photograph of the fabricated chip. Circled are four individual detectors that are each the inputs to four separate and identical circuits as shown in (a). Multiple copies are used for testing and verification. The other detectors seen on the chip are also for testing and not used for this work.

Fig. 7.
Wavelength dependent voltage output from CMOS BDI sensor.

Fig. 8. Wavelength dependent BDJ sensor response from 600 nm to 620 nm. The error bars show the maximum and minimum values measured at each wavelength.

Fig. 9.

Details of the CMOS-based oxygen sensor measurement setup. The xerogel sensor is coated on the fiber in the measurement chamber and the light emitted from the xerogel is carried by the fiber to the BDI sensor chip.

Fig. 10. Stern–Volmer plot for the BDJ CMOS (circles) and PMT/optical filter (triangles) platforms.

Fig. 11.

Actual response from BDJ CMOS chip and PMT as a function of oxygen concentration. The BDJ output is V_{out} in Fig. 6.