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Ratiometric, filter-free optical sensor based on a complementary metal oxide semiconductor buried double junction photodiode



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HIGHLIGHTS

GRAPHICAL ABSTRACT

- A detector for measuring signals at two wavelengths simultaneously.
- No optical filters, no moving parts. • Functionally equivalent to a slew
- scanned spectrofluorimeter.
- Consumes orders of magnitude less power.



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ABSTRACT

We report a complementary metal oxide semiconductor integrated circuit (CMOS IC) with a buried double junction (BDJ) photodiode that (i) provides a real-time output signal that is related to the intensity ratio at two emission wavelengths and (ii) simultaneously eliminates the need for an optical filter to block Rayleigh scatter. We demonstrate the BDJ platform performance for gaseous NH₃ and aqueous pH detection. We also compare the BDJ performance to parallel results obtained by using a slew scanned fluorimeter (SSF). The BDJ results are functionally equivalent to the SSF results without the need for any wavelength filtering or monochromators and the BDJ platform is not prone to errors associated with source intensity fluctuations or sensor signal drift.

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Abbreviation: BDJ, buried double junction; CMOS IC, complementary metal oxide semi-conductor integrated circuit; SSF, slew scan fluorimeter; ([P_{14,666}⁺]), trihexyl(tetradecyl)phosphonium cation; HPTS³⁻, 8-hydroxypyrene-1,3,6-trisulfonate: SNARF-1. seminaphthorhodamine-1 carboxylate.

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1. Introduction

Myriad detection strategies exist for chemical sensing [1,2]. Optical sensing is attractive because it can be minimally invasive and it allows stand-off detection [1,3–5]. However, optical sensing is not without its limitations. In most cases, one must operate under conditions where the source, sensor, and/or detector do not exhibit appreciable drift/decomposition over time and the sensor per se must respond reversibly to the target analytes. In situations where there are system instabilities, one often resorts to kinetic or ratiometric methods [6-9]. Kinetic methods are often implemented within fluorescence-based schemes by using the sensing reporter excited-state fluorescence lifetime (τ) as the analytedependent information carrier [10-13]. Because τ often occurs on a time scale that is orders of magnitude shorter in comparison to system fluctuations and τ is generally intensity independent, this approach is an intrinsically less bias-prone detection modality. The down side of this approach is that the detection system is often substantially more complex and costly. Ratiometric methods are commonly exploited when the sensor reporter chemistry exhibits spectroscopically distinct absorbance and/or emission spectral bands that change in an analyte-dependent manner. One then need only measure the analyte-dependent absorbance and/or intensity ratio at two wavelengths to yield an analyte-dependent response. Thus, in ratiometric methods, one needs to discriminate the absorbance/intensity at two wavelengths. There are essentially three strategies for accomplishing this: (1) a spectrometer coupled to an array detector (CCD, photodiode array); (2) slew scanning to rapidly "jump" a monochromator between two wavelengths using a platform with a single photodetector (e.g., photomultiplier tube); or, (3) a system having two discrete detection channels for monitoring at two wavelengths. Each of these platforms can be costly, complicated, and/or consume substantial electrical power (e.g., CCDs, photomultiplier tubes). There are certainly advantages to platforms that have no moving parts (e.g., gratings) and that operate at low power.

In this paper, we report a complementary metal oxide semiconductor integrated circuit (CMOS IC) with a buried double junction (BDJ) [14–16] photodiode combined with on-board signal processing circuitry that provides a direct measurement of the intensity ratio at two emission wavelengths and eliminates the need for any moving parts, external circuitry, or optical filters. Although BDJ platforms have been used previously to realize color-

sensitive detection [14-16], this strategy has been used just one other time in chemical sensing to minimize Rayleigh scatter and perform detection at a single emission wavelength [17]. Here, we report a BDJ platform for real-time ratiometric intensity measurements at two emission wavelengths coupled with dual wavelength ratiometric chemical sensors for detecting gaseous NH₃ and determining aqueous solution pH. The NH₃-responsive sensor is based on a recently developed photonic ionic liquid consisting of trihexyl(tetradecyl) phosphonium ($[P_{14,666}^+]$) cations paired with 8-hydroxypyrene-1,3,6-trisulfonate (HPTS³⁻, pyranine) [18]. The $[P_{14,666}^{++}]_{3}[HPTS^{3-}]$ fluorescence emission spectrum exhibits two well-resolved bands that we previously used in a ratiometric mode for NH₃, MeNH₂, EtNH₂, *n*-PrNH₂, Et₂NH, and Et₃N detection by using slew scan fluorescence (SSF) with a conventional monochromator as the wavelength selection device and photomultiplier tube as the detector. The aqueous pH sensor is based on seminaphthorhodamine-1 carboxylate (SNARF-1) sequestered within a sol-gel-derived xerogel film [19]. SNARF-1 exhibits a fluorescence emission spectrum with two pH-dependent fluorescence bands that have been exploited for ratiometric pH measurements in a variety of complex systems [20,21].

2. Materials and methods

2.1. Sensor fabrication and sample preparation

 $[P_{14,666}^+]_3[HPTS^{3-}]$ was prepared as described elsewhere [18]. Thin films were formed by spin coating (500 RPM, 100 s) 250 µL of a 10 mM $[P_{14,666}^+]_3[HPTS^{3-}]$ solution (in EtOH) onto clean quartz microscope slides. SNARF-1 (Life Technologies) doped xerogels were prepared by using a protocol adapted from Dobmeier et al. [19]. Briefly, a tetramethoxysilane (TMOS, Sigma–Aldrich) sol was prepared by mixing 1000 µL of doubly-distilled deionized H₂O (ddH₂O), 500 µL of TMOS, and 10 µL of 0.04 M HCl (J.T. Baker) followed by 30 min sonication on ice. A SNARF-1-doped sol was prepared by mixing 75 µL of the aforementioned TMOS sol, 32 µL of 5 mM SNARF-1 dissolved in ddH₂O, and 300 µL of 0.01 M pH



Fig. 1. Measurement systems used in this research. (Panel A) Slew scan spectrofluorimeter. (Panel B) BDJ detector chip. (Panel C) Cross sectional view through a BDJ detector chip structure. (Panel D) Basic BDJ detector chip signal processing topology.

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