Optical absorption sensing with dual-spectrum silicon LEDs in SOI-CMOS technology

Satadal Dutta\textsuperscript{1,}\textsuperscript{*}, Peter G. Steeneken\textsuperscript{1}, and Gerard J. Verbiest\textsuperscript{1}
\textsuperscript{1}Precision and Microsystems Engineering, Delft University of Technology, Delft, The Netherlands
\textsuperscript{*}Email: s.dutta-1@tudelft.nl

Abstract—Silicon p-n junction diodes emit low-intensity, broad-spectrum light near 1120 nm in forward bias and between 400-900 nm in reverse bias (avalanche). For the first time, we experimentally achieve optical absorption sensing of pigment in solution with silicon micro LEDs designed in a standard silicon-on-insulator CMOS technology. By driving a single LED in both forward and avalanche modes of operation, we steer it’s electroluminescent spectrum between visible and near-infrared (NIR). We then characterize the vertical optical transmission of both visible and NIR light from the LED through the same micro-droplet specimen to a vertically mounted discrete silicon photodiode. The effective absorption coefficient of carmine solution in glycerol at varying concentrations were extracted from the color ratio in optical coupling. By computing the LED-specific molar absorption coefficient of carmine, we estimate the concentration (~0.040 mol L\textsuperscript{-1}) and validate the same with a commercial spectrophotometer (~0.030 mol L\textsuperscript{-1}). With a maximum observed sensitivity of ~1260 cm\textsuperscript{-1} mol\textsuperscript{-1} L, the sensor is a significant step forward towards low-cost CMOS-integrated optical sensors with silicon LED as the light source intended for biochemical analyses in food sector and plant/human health.

Keywords—Silicon, Avalanche breakdown, CMOS, Optical sensing, light-emitting diode.

I. INTRODUCTION

Silicon (Si) photonics is emerging as a key player in the development of CMOS-integrated optical devices for applications in bio-chemical sensing and data communication links \cite{1}-\cite{10}. State-of-the-art optical sensors, popular in biochemical analyses in both medical and food sector, use expensive lasers or quasi-monochromatic LEDs made of III-V compound semiconductors \cite{11}. This prevents the monolithic integration with driver/read-out electronics designed in Si CMOS technology. Interestingly, Si p-n junction diodes exhibit broad-spectrum electroluminescence (EL) near 1120 nm in forward mode (FM) and in the range of 400 nm - 900 nm in avalanche mode (AM) of operation, although at a very low quantum efficiency (~10\textsuperscript{-3}-10\textsuperscript{-5}) \cite{12}-\cite{17} due to the indirect bandgap of Si. Recent advancements \cite{18}-\cite{20} have successfully highlighted the Si LED as a promising candidate for monolithically integrated optical interconnects due to the high responsivity of Si photodiodes (PDs) for wavelengths ($\lambda$) < 1000 nm.

In this work, we experimentally show for the first time that Si LEDs designed in a standard SOI-CMOS technology are viable for optical absorption sensing by driving a single LED in both FM and AM operation. The vertically transmitted light propagates through a pigmented micro-

Fig. 1. Schematic block diagram illustrating the optical sensing method. Optical coupling from the Si LED on the CMOS chip to an externally mounted Si PD (reverse biased at 1 V) is measured in air and in presence of the same glycerol droplet specimen in both (a) forward and (b) avalanche modes of LED operation. The droplet contains dissolved carmine pigment absorbing light of $\lambda$ in the 400 nm - 600 nm interval, emitted by the LED in avalanche mode. Post-measurement of each droplet, the chip surface is cleaned with laboratory grade iso-propanol (IPA) and to re-use the LED for the next droplet measurement.

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for any process modification or device replacement in the optical sensor. The 400 nm-1300 nm spectral range is highly suitable in bio-sensing e.g. photosynthetic pigments [23], leaf-water status [24][25], coloured contaminants in water [26]-[28] and blood oxygen levels [29]. Our work, therefore, constitutes a major step forward in realizing low-cost, and micro-volume CMOS-integrated optical sensors with silicon light source.

II. EXPERIMENTAL MATERIALS AND METHOD

A. LED design and electroluminescent spectra

Figs. 2a, 2b show the top-view layout of the two test LEDs D1 and D2 respectively, designed in a standard 130 nm silicon-on-insulator (SOI) CMOS technology [21]. Figs. 2c shows their vertical cross-section. D1 is a vertical n+p junction at a depth of ~0.25 μm with a breakdown voltage of ~17 V [18] and peripherally placed electrode contacts. D2 is a lateral p+nn+ junction reaching the Si-SiO2 interface, with ~17 V [18] and peripherally placed electrode contacts. D2 is therefore a higher spatial uniformity and quantum efficiency interdigitated (comb-like) layout [30] of alternating cathode anode pairs. (Fig. 3c) by imaging the chip surface at an inclination of ~45° via a micro-manipulator. The LED is driven in avalanche (~19 V) and forward (~1 V) operation. Further, at a given reverse bias of 1V using a Keysight B2912A precision SMU with dc offset currents < 1 pA. Commercial carmine (E120 in glycerol (E422) and water). (c) Top-view and (d) slanted view (25 degrees w.r.t. horizontal) of a droplet from sample c2. (e) Schematic cross-section showing the light-rays being focussed by the plano-convex micro lens formed by the droplet.

b) Solution specimens of carmine in laboratory-grade glycerol with the indicated concentrations (% by volume) relative to a reference commercial sample of liquid food color (carmine (E120) in glycerol (E422) and water). (f) Optical absorption coefficient (∆η) of carmine that overlaps with the AM EL spectrum, 400 nm < λ < 600 nm.

B. Measurement set-up and sample preparation

As illustrated in Fig. 3a, the on-chip Si LED, placed on a vacuum chuck, is electrically probed by tungsten needles. The Si PD (BPW34 from Vishay semiconductors), is mounted vertically above the chip at a centre-height ~ 5 mm tilted at ~45° via a micro-manipulator. The LED is driven in a constant current (sweep) mode and the PD is driven at a fixed reverse bias of 1V using a Keysight B2912A precision SMU with dc offset currents < 1 pA. Commercial carmine (E120 food colour) solution (of unknown concentration (c)), mounted vertically above the chip at a centre-height ~ 5 mm tilted at ~45° via a micro-manipulator. The LED is driven in avalanche (~19 V) and forward (~1 V) operation. Further, at a given reverse bias of 1V using a Keysight B2912A precision SMU with dc offset currents < 1 pA. Commercial carmine (E120 in glycerol (E422) and water). (c) Top-view and (d) slanted view (25 degrees w.r.t. horizontal) of a droplet from sample c2. (e) Schematic cross-section showing the light-rays being focussed by the plano-convex micro lens formed by the droplet.

Fig. 3. (a) Schematic of the measurement set-up (not to scale). The on-chip Si LED and the external Si photodiode (BPW34) are driven by a 2-channel Keysight B2912A precision source-measure unit (SMU) in constant current mode and constant voltage mode respectively. (b) Solution specimens of carmine in laboratory-grade glycerol with the indicated concentrations (% by volume) relative to a reference commercial sample of liquid food color (carmine (E120) in glycerol (E422) and water). (c) Top-view and (d) slanted view (25 degrees w.r.t. horizontal) of a droplet from sample c2. (e) Schematic cross-section showing the light-rays being focussed by the plano-convex micro lens formed by the droplet.

by volume relative to c_ref (see Fig. 3b). Sample c0=0 refers to only glycerol (no pigment). PD photocurrent (ΔI_PD) is measured in air and in presence of a micro-droplet (diameter: 250 μm – 500 μm), which is transferred from each solution sample to the chip with a hydrophilic tip of a ~100 μm silica fiber (Figs. 3c,d) masking the Si LED entirely. The same droplet is used to measure ΔI_PD for AM and FM LED.

III. RESULTS AND ANALYSIS

The glycerol droplet height h is primarily governed by the angle of contact [31][32] at the local liquid - chip (SiO2) interface, and was estimated to be within 180 μm - 280 μm (Fig. 3c) by imaging the chip surface at an inclination of ~25° with a VHIX microscope from Keyence. The same was verified by droplet imaging with a Attension Theta Lite optical tensiometer by experimenting with multiple droplet samples on the chip surface. A droplet acts as a microscopic plano-convex lens (Fig. 3e) with a good matching of refractive index (~1.47) with that of the back-end oxide layer (~1.45), which enhances the vertical transmission coefficient of light. Optical gain due to lensing is evident from Fig. 4 which shows the measured ΔI_PD versus ΔI_LED in AM and FM operation. For both D1 and D2, ΔI_PDAM > ΔI_PDFM primarily due to the higher PD quantum efficiency (η_PD) for light emitted in AM [18]. ΔI_PDFM and ΔI_PDAM are respectively ~1.2 times and ~1.5 times higher in presence of a glycerol (c0) droplet as compared to in air. A mismatch in the gain in AM and FM is likely due to electrostatic effects on glycerol refractive index [33] at different V_LED applied in AM (~19 V) and FM (~1 V) operation. Further, at a given V_LED, both ΔI_PDFM and ΔI_PDAM is higher for D2 than for D1 due to the higher external quantum efficiency of D2. Since the pigment absorption window overlaps only with the AM EL spectrum, we can express the photocurrent in presence of the droplet as:

ΔI_(PD(Gly))AM(c) = η_(LED)AM ΔI_(PD(Gly))FM(c) exp{|-α(c0) h|} (2)

Here η_Gly is the light-extraction efficiency from the chip through the droplet. The colour ratio (COR) of optical coupling in AM to FM can then be expressed as:
α = corresponding to our broadband AM Si LED as reference (undiluted) solution, cm⁻¹. To obtain a quantitative estimate of absorbance through a standard quartz cuvette with a 10 mm optical path of a solution sample with a concentration of the target specimen. Subsequently, we obtain the spectrum and the molar absorption coefficient spectrum of the carmine, the effective absorption coefficient and carmine concentration was determined and validated by a commercial spectrophotometer.

We reported the very first proof-of-principle of optical absorption sensing of pigment in solution with broad-spectrum silicon micro LEDs designed in a standard silicon-on-insulator CMOS technology. Vertical optical transmission through a glycerol micro-droplet containing carmine pigment was measured with a silicon PD while driving the on-chip silicon LED in both forward and avalanche modes of operation, and thereby steering it’s electroluminescent spectrum between visible and near infrared. Hence, the same droplet can be used to measure optical transmission for both visible and near-infrared light from the same LED. The effective absorbance of the solution was obtained from the ratio of photocurrent in avalanche-mode to that in forward-mode LED operation. Further, from the observed droplet height and the known molar absorption coefficient of carmine, the effective absorption coefficient and carmine concentration was determined and validated by a commercial spectrophotometer.

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