Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 120 (2015) 1187 – 1190

Procedia Engineering

www.elsevier.com/locate/procedia

EUROSENSORS 2015

Optical measurements by phase shift based technique for high sensitivity and high resolution detection of chemical/biological substances

Andrea De Marcellis*, Mohammed Janneh, Elia Palange

University of L'Aquila, Department of Industrial and Information Engineering and Economics, L'Aquila, Italy

Abstract

In this work we demonstrate that the phase shift detection technique can be applied to reveal, with very high sensitivity and resolution, concentrations of chemical/biological substances optically detectable by measuring power variations of a light source. The system is based on the synchronous demodulation technique and employs a Si photodiode (PD) operating in photovoltaic regime biased through a small modulating sinusoidal waveform. The phase shift variations are measured between this biasing voltage and that one generated by the Si PD illuminated by the light, that interacts with the chemical/biological sample under analysis, providing the phase shift change. Experimental results demonstrate that is possible to achieve phase detection sensitivity, with respect to light power variations, up to 3100°/μW as a function of the Si PD settable operating conditions. By using a commercial lock-in amplifier with a 0.01° phase resolution, a light power variation resolution of about 3pW has been achieved. As a case-example, variations of the molar concentration of a methylene blue solution are detected by performing optical absorption standard measurements. Despite the phase resolution limitation of the used lock-in amplifier, the comparison between the data obtained through the proposed technique and those ones achieved by conventional amplitude measurements demonstrates a high improvement of the detection sensitivity. As a consequence, the measurement of molar concentration variations with a resolution of 80pM is achieved resulting 33 times higher than that one obtained with the amplitude detection.

Peer-review under responsibility of the organizing committee of EUROSENSORS 2015

Keywords: Phase shift detection technique; High sensitivity; High resolution; Chemical and biological substances; Photodiode; Photovoltaic regime; Molar concentration.

* Corresponding author. Tel.: +39 0862 434424; +39 340 3063460.
E-mail address: andrea.demarcellis@univaq.it

Peer-review under responsibility of the organizing committee of EUROSENSORS 2015
doi:10.1016/j.proeng.2015.08.782

1. Introduction

Optical methods are widely used for the detection of chemical and biological compounds by measuring light absorption, scattering and emission [1-3]. An important task is the development of new detection systems and innovative techniques able to reveal the concentration of targets with high sensitivity and resolution. Nevertheless, whatever be the employed optical technique, the light coming from the sample under analysis must be detected by a Photodiode (PD) generating a photocurrent proportional to the light power impinging on its sensitive area. The conventional approach used to improve the signal-to-noise ratio of small and noisy signals is based on the synchronous demodulation technique implemented by lock-in amplifiers [4-8]. However, this approach allows to achieved sensitivity and resolution limited by the selected full-scale for the amplitude measurements. Recently, variations of the light power have been detected with very high sensitivity and resolution by measuring the phase shift changes between the electrical signal generated by the PD and a reference at a specific modulating frequency. This technique has the advantage to be independent from the signal amplitudes and does not suffer of any full-scale limitations [9-11]. With respect to the achievements reported in [11], in this work we present an improvement of one order of magnitude in sensitivity and resolution of the detection system for phase shift measurements as a function of the light power variations impinging on the sensitive area of a Si PD operating in photovoltaic regime and biased through a small modulating sinusoidal waveform. Finally, as a case-example, variations of the molar concentration of a methylene blue solution are detected by performing optical absorption standard measurements achieving a detection resolution of 80pM that is 33 times better than that one obtained with the conventional amplitude detection technique.

2. Measurement set-up, experimental results and discussion

The set-up of the proposed detection system is reported in Figure 1. It has been employed to perform the optical and electrical characterisations and to measure the variations of the molar concentration of a methylene blue solution contained in a 1cm length quartz cuvette by performing standard optical absorption measurements. In this last case, it has been measured the variation of the optical power of a continuous wave p-polarised monochromatic HeNe laser, with an emission wavelength λ=633nm and a 0.5mW maximum power, suitable attenuated by a Neutral Density Filters (NDF) and focussed on the Si PD sensitive area by the lens L. In Figure 2 the data related to the conducted characterizations are reported showing the phase shift detection achieved by employing the commercial lock-in amplifier Stanford Research SRS830-DSP and the VTB8440B by PerkinElmer Optoelectronics as Si PD. This last operates in photovoltaic regime and is biased through a modulating sinusoidal voltage signal $V_{BIAS}(f_0)$ having a small amplitude equal to ±50mV. The reported phase shift has been measured between $V_{BIAS}(f_0)$ and $V_{OUT}(f_0)$, as a function of the laser power impinging on the Si PD sensitive area, with a fixed resistive load R_L=10MΩ and for two different values of the modulating frequencies: f_0 = 7.7Hz and f_0 = 0.77Hz.

More in detail, the measured phase shift is due to the variation of the *pn* depletion layer width and the built-in potential of the Si PD that vary as a function of the light power impinging on its sensitive area.

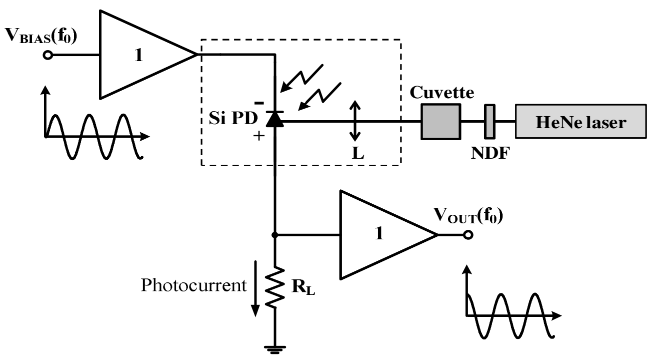

Fig. 1. Experimental set-up showing all the optics and electronics components.

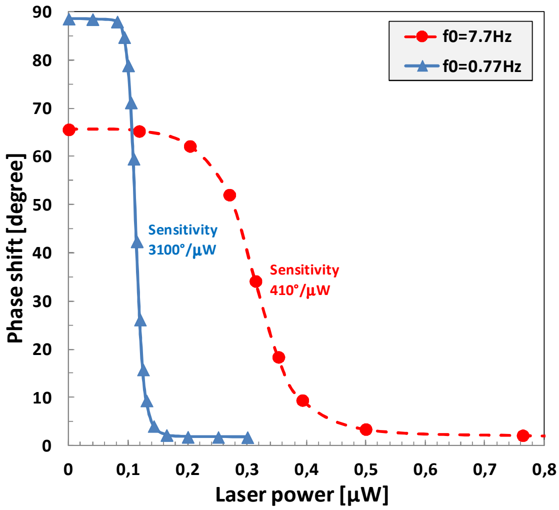

Fig. 2. Electrical characterisations of the Si PD in photovoltaic regime: the phase shift as a function of the laser power impinging on the Si PD sensitive area for two different modulating frequencies f_0.

This causes variations of the junction capacitance C_j that, more in general, can be expressed as follows [12]:

$$C_j = A\sqrt{\frac{q\varepsilon_0\varepsilon_r N_a N_d}{2(V_R+\psi_0)(N_a+N_d)}} \qquad (1)$$

where A is the Si-PD illuminated sensitive area, q the electron charge, ε_0 and ε_r the vacuum and Si permittivity's, N_a and N_d the acceptor and donor concentrations, ψ_0 the built-in potential and V_R the applied reverse bias voltage that in this case is null. As a consequence, a variation of the light power impinging on the Si-PD can be detected by measuring the phase shift, induced by the change of C_j, occurred between the reference signal (i.e., $V_{BIAS}(f_0)$) and that one photogenerated by the Si-PD (i.e., $V_{OUT}(f_0)$). Referring to Figure 2 and considering the 0.01° phase resolution of the employed lock-in amplifier, the resulting resolution value in the laser power detection is about 24pW for f_0=7.7Hz and 3pW f_0=0.77Hz.

Finally, Figure 3 shows the experimental results concerning the detections of the changes of the molar concentration of a methylene blue solution achieved by measuring, through the lock-in amplifier, the variations of the voltage amplitude and the phase shift for f_0=0.77Hz. The initial molar concentration of the employed solution is equal to 8µM that has been suitably diluted with different quantities of pure distilled water. In both the cases, the linear regression well fits the experimental data and the resulting sensitivity is 380µV/µM with a resolution of 2.6nM for the amplitude measurements and 127°/µM with a resolution of 80pM for the phase shift detections. As a consequence, an improvement of a factor 33 of the detection resolution has been demonstrated so allowing the detection of about 80pM concentration variations of the considered substance. Also in this case, since this value is mainly limited by the 0.01° phase resolution of the employed lock-in amplifier, the use of other commercial instruments with 1µ° phase resolution allows to reach concentration resolutions down to 8fM.

3. Conclusions

In this paper we have proposed a very high sensitivity, tuneable, simple phase shift detection technique suitable for the measurement of light power variations in the order of few tens of pW. The implemented experimental set-up employs a continuous laser source and a Si PD operating in photovoltaic regime biased through a small sinusoidal signal. This makes the proposed approach suitable for its integration in portable instrumentations and sensor systems employed for the detection of chemical and biological substances.

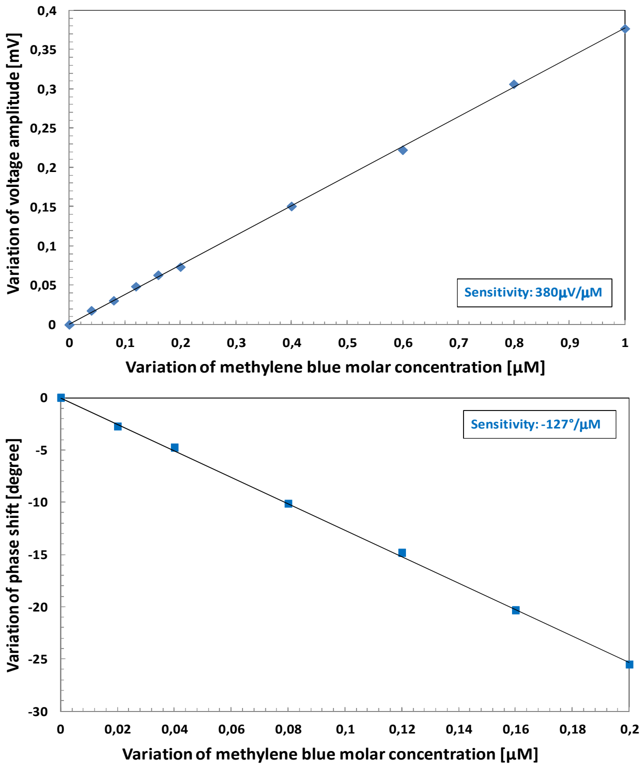

Fig. 3. Experimental detections of the changes of the molar concentration of a methylene blue solution achieved by measuring the variations of: the voltage amplitude (above) and the phase shift for f_0=0.77Hz (below).

References

[1] M.T. Cone, J.D. Mason, E. Figueroa, B.H. Hokr, J.N. Bixler, C.C. Castellanos, G.D. Noojin, J.C. Wigle, B.A. Rockwell, V.V. Yakovlev, E.S. Fry, Measuring the absorption coefficient of biological materials using integrating cavity ring-down spectroscopy, Optica 2 (2015) 162-168.

[2] K. Korena, L. Huttera, B. Enkob, A. Peinc, S.M. Borisova, I. Klimanta, Tuning the dynamic range and sensitivity of optical oxygen-sensors by employing differently substituted polystyrene-derivatives, Sens. & Act. B 176 (2013) 344-350.

[3] S. Zenga, S. Hub, J. Xiac, T. Andersonb, X.Q. Dinha, X.M. Mengc, P. Coqueta, K.T. Yonga, Graphene–MoS2 hybrid nanostructures enhanced surface plasmon resonance biosensors, Sens. & Act. B 207 (2015) 801-810.

[4] A. De Marcellis, G. Ferri, E. Palange, A novel analog autocalibrating phase-voltage converter for signal phase shifting detection, IEEE Sensors Journal 11 (2011) 259-266.

[5] M. Davidovic, J. Seiter, M. Hofbauer, W. Gaberl, H. Zimmermann, Monolithically integrated dual-lock-in optical sensor, IET Electronics Letters 50 (2014) 306-308.

[6] A. Hu, V. P. Chodavarapu, CMOS optoelectronic lock-in amplifier with integrated phototransistor array, IEEE Trans. on Biom. Cir. & Sys. 4 (2010) 274-280.

[7] A. De Marcellis, G. Ferri, C. Di Natale, E. Martinelli, A. D'Amico, A fully-analog lock-in amplifier with automatic phase alignment for accurate measurements of ppb gas concentrations, IEEE Sensors Journal 12 (2012) 1377-1383.

[8] P.M. Maya-Hernandez, M.T. Sanz-Pascual, B. Calvo, CMOS Low-Power Lock-In Amplifiers With Signal Rectification in Current Domain, IEEE Trans. on Instr. and Meas. 64 (2015) 1858-1867.

[9] A. De Marcellis, G. Ferri, E. Palange, A fully-analog high performances automatic system for phase measurement of electrical and optical signals, IEEE Trans. on Instr. and Meas. 64 (2015) 1043-1054.

[10] A. De Marcellis, G. Ferri, E. Palange, Electrical self-modulation of optical sensors for light power measurement in chemical applications by phase detection technique, Sens. and Act. B 193 (2014) 375-383.

[11] A. De Marcellis, M. Janneh, E. Palange, Very high sensitivity tuneable phase detection of light power variations by using electrical modulation of a Si-photodiode in photovoltaic regime, IET Electronics Letters 51 (2015) 282-284.

[12] S.L. Chuang, Physics of Photonic Devices, 2nd Ed. Wiley & Sons, New Jersey, USA, 2009.