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A Miniaturized UV-LED Based Optical Gas Sensor Utilizing Silica Waveguides for the Measurement of Nitrogen Dioxide and Sulphur Dioxide †

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Abstract: Continuing advances in the development of light-emitting diodes (LEDs) in the deep ultraviolet spectral range (DUV) enable miniaturized, mobile, and cost-effective DUV gas sensors. Efficient light coupling, guiding, and splitting plays a key role for stable sensor systems and is realized by low-loss silica based waveguides, fabricated by ultrashort pulsed laser etching and laser polishing. The tapered waveguide leads to a significantly higher efficiency with less required space compared to optical fibers or free beam setups, respectively. Toxic gases like NO_2 and SO_2 were successfully measured down to 1 ppm utilizing LEDs with wavelengths of 395 nm and 280 nm.

Keywords: gas sensor; waveguide; UV; LED; absorption spectroscopy; laser processing; glass

1. Introduction

The optical sensors market is growing steadily. Valued over USD 15.4 billion in 2015, a compound annual growth rate of 7.5% is estimated between 2016 and 2024 [1]. One reason for this growth is the successful implementation of semiconductor based LEDs as a lightweight, compact, efficient, and cost-effective light source and the demand for miniaturized sensors. Up to now, many optical sensors are based on LEDs emitting in the visible and infrared region, e.g., heart rate monitors of smartwatches or smoke detectors. In contrast, due to a lack of suitable light sources, current optical sensors working in the DUV have been engineered with gas discharge lamps, limiting the adoption by the mass market due to their long warmup times, large sizes, short lifetimes and high cost.

However, in recent years, several groups have been successfully working on the development of DUV-LEDs with wavelengths below 240 nm [2–4] and even 210 nm [5]. These LEDs are based on the wide bandgap material system AlGaN and their wavelengths can be controlled by the Al mole fraction in the AlGaN quantum well (QW) active region [6]. As a result, absorption peaks of harmful gases in the DUV have been exploited by LED based sensors to selectively measure the concentration of O_3, NO_2 and NO [7,8]. The spectral absorption measurement offers advantages over conventional electrochemical sensors, which are limited regarding selectivity and cross-sensitivity [9]. For instance, NO_x sensors unselectively measure NO and NO_2 and show significant cross-sensitivity to NH_3 [10].

On the other hand, DUV-LEDs still exhibit fairly low efficiencies and emission. Thus, along ongoing optimizations of the LEDs, efficient sensor systems are required to couple, guide and split light. So far, publications on DUV-LED gas sensors base on optical fibers [7] or lens based setups [8] and require much space. In contrast, this work reports on a miniaturized optical gas sensor utilizing silica waveguides and demonstrates its functionality by measuring NO_2 and SO_2 down to 1 ppm using LEDs with wavelengths corresponding to the respective absorption peaks of the two gases.

2. Sensor Design

2.1. Sensor Principle

The presented sensor exploits the Beer-Lambert law, which describes light absorption in a medium like a gas, depending on the path length l through the absorbing gas, the wavelength dependent specific absorption coefficient $\alpha(\lambda)$ of the gas, and the gas concentration c. It is given by

$$\frac{I_1}{I_0} = e^{-\alpha(\lambda)cl}, \tag{1}$$

whereby I_0 and I_1 denote the light intensities before and after absorption, respectively. Two reference methods are used to stabilize the sensor signal and realize high resolutions [11]. First, in order to compensate intensity fluctuations of the LED due to temperature changes and ageing effects, both I_0 and I_1 have to be measured by splitting the light path. In this work, a reference waveguide arm splits the light, which is coupled into the waveguide from several LEDs, into the main measurement path and the reference path (cf. Figure 1). While the reference path guides light to the reference detector, the major part of the light is guided through the waveguide and a silica window into the gas volume. In order to double the path length while keeping the sensor compact, a concave mirror at the end of the 55-mm-long absorption cell reflects and collimates the light back to the measurement detector.

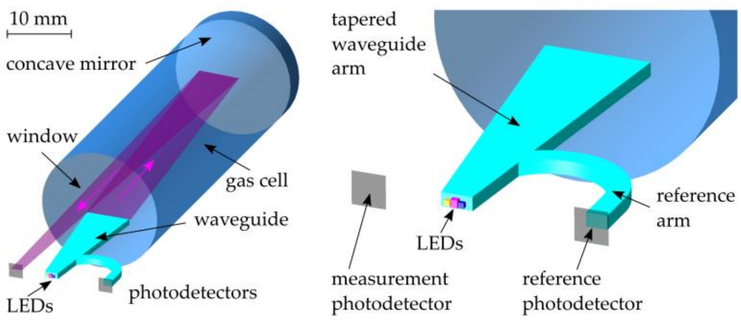

Figure 1. Schematics of the DUV-LED based gas sensor utilizing a tapered waveguide: the light is splitted into a main path and a reference path. The tapering improves the efficiency by light collimation. The absorption length is doubled by use of a concave mirror at the back face of the gas cell.

Second, in order to compensate non-gas-specific transmission changes, e.g., due to a hazed window, a reference LED with a non-gas-sensitive wavelength is used and the two (or more) LEDs are turned on and off alternately. Finally, the referenced relative transmission T is calculated by

$$T = \frac{T_{Gas}}{T_{Ref}} = \frac{\frac{I^M_{Gas} - I^M_{Gas,dark}}{I^R_{Gas} - I^R_{Gas,dark}}}{\frac{I^M_{Ref} - I^M_{Ref,dark}}{I^R_{Ref} - I^R_{Ref,dark}}}, \tag{2}$$

with superscripts M and R denoting intensities measured on the measurement and reference detector, respectively. Subscript indices *Gas* and *Ref* indicate the specific active LED (gas-specific or non-gas-specific) and *dark* the intensity measured without any active LED (dark current measurement).

A 20-bit, two-channel simultaneous sampling measurement system based on the DDC112 analog-to digital converter (Texas Instruments) and a sbRio-9632 development board (National Instruments) was used to control the LEDs and collect data. It is described in detail in Reference [12].

2.2. Optical Simulations for Optimal System Efficiency

The 1-mm-thick waveguide, which is fabricated by ultrashort pulsed laser etching and subsequently laser polishing to achieve smooth sidewalls [13], consists of a DUV transparent fused silica core and ambient air cladding, leading to a high refractive index contrast Δ of 0.28. Using raytracing simulations (Zemax Optics Studio), a much higher coupling efficiency of about 73% is achieved compared to an efficiency of about 4% using a standard glass fiber with a small Δ of 0.01. At the same time, curves with radii down to 0.6 mm can be implemented with the presented system, while the simulated fiber shows the same loss already at 53.6 mm limiting the miniaturization [13].

The efficiency of the measurement path is doubled by light collimation in a tapered waveguide. The front width is 2 mm, while the back is expanded to 7 mm. This kind of collimation has advantages over lens or reflector based collimators due to its integrated light homogenizing properties.

2.3. Optoelectronic Components of the Sensor

The absorption spectra of NO_2 and SO_2 exhibit maxima at emission wavelengths around 400 nm and 280 nm, respectively. Correspondingly LED-chips with wavelengths of 395 nm (EOLC-395-34, Epigap, Berlin, Germany), 290 nm (Research samples, Ferdinand Braun Institut, Berlin, Germany), and 280 nm (EOLC-280-39, Epigap) were implemented. Optionally, one non-gas-selective reference LED-chip at a wavelength of 597 nm (LYP47F, Osram, Regensburg, Germany) was used. TO5-packaged silicon photodetectors (S1226-5BQ, Hamamatsu, Japan) with a photosensitive area of 2.4 mm × 2.4 mm were utilized to measure the light intensity.

One advantage of the presented sensor is its platform idea. Depending on the use-case appropriate gas specific LEDs are chosen. In this work two versions were used to measure NO_2 and SO_2. For measuring NO_2, LED-chips with wavelengths of 395 nm (gas) and 595 nm (reference) and for the measurement of SO_2, with 280 nm/290 nm (gas) and 395 nm (reference) were assembled.

3. Gas Measurements

Measurement of NO_2 and SO_2

A total gas flow of 1000 sccm was applied, consisting of a variable amount of NO_2 in N_2 with 20% O_2 as carrier gas. The two LEDs are activated serially and photodetector currents are integrated leading to a sensor sample rate of about 1 Hz. NO_2 concentrations from 100 ppm down to 1 ppm were applied and successfully measured by the sensor as shown in Figure 2a including drift compensation.

Due to the available gas cylinder, a lower flow rate of 500 sccm was applied for measuring SO_2. SO_2 concentrations of 20 ppm and lower were applied and 1 ppm could be resolved (cf. Figure 2b).

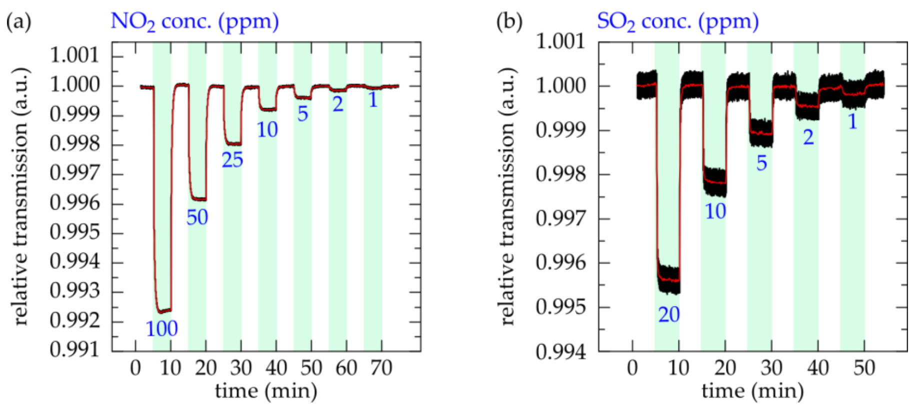

Figure 2. Sensor signal (black) and averaged (n = 10) signal (red) for measuring different concentrations of (**a**) NO_2 (395-nm-LED) and (**b**) SO_2 (280-nm-LED).

4. Conclusions

The use of LEDs and a tapered silica waveguide significantly decreases the sensor size. NO_2 and SO_2 concentrations were successfully measured down to 1 ppm and further measurements in the sub-ppm regime and studies on cross-sensitivities are currently being investigated.

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Conflicts of Interest: The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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