Ethylene monitoring in the "fingerprint" region using a mini White-cell and a micromachined IR-emitter

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Summary: *Precise and continuous ethylene detection is needed in various fruit storage applications. One of the main goals of the presented work is the development of gas sensor systems for ethylene monitoring based on miniaturized low cost infrared spectroscopy. The developments concentrate on systems based on small multi pass reflection cells, novel micromachined IR-emitters and thermopile arrays in order to measure at a wavelength of 10.6µm, a rotational vibrational region (fingerprint region) of ethylene. First ethylene measurements were carried out.*

Keywords: *C_2H_4 , optical gas sensor, long path cell*

1 Introduction

In today’s food industry quality monitoring plays an integral part during the whole food chain. The consumer demands controlled quality of agricultural products, particularly ripeness, origin, use by date and contamination levels. One of the most relevant substances in fruit monitoring is ethylene. Fruits are usually stored unripe in low oxygen, low ethylene atmospheres to keep them fresh. Ethylene is produced by fruits during the ripening process and is an indicator that some ripe fruits need to be removed out of the storage container in order to not ripen all the other fruits as well. Ethylene is also added during storage when accelerated ripening is desired. In the case of apples, ethylene concentration during storage of 1-100 ppm are normal. The amount of 400 ppm can be considered as an alarm level.

2 Experimental

Food monitoring systems have to be low cost units compared to spectroscopic lab equipment. Hence, for example IR-laser spectroscopy systems are not suitable for mass use in food industry as they are much too expensive. Low cost components, like thermal emitters and non cryogenic detectors need to be used. Of course there are several micro sized thermal emitters commercially available, but compared with an ideal black body radiator, their emissivity and thus the emitted radiation is moderate. This was the motivation to develop a novel type of micromachined thermal IR-emitters. The main difference compared with common thermal micro emitters is the use of 3D structured bulk silicon (fig. 1). The regular ordered macropores of the emitters are obtained by electrochemical etching of prepatterned silicon substrates. The fabrication technology is described in detail in [1] and [2]. Typical pore diameter of the photonic-crystal-like structures are 5 µm. The macroporous silicon shows a black-body-like emission profile for a wide wavelength range. The device is heated to his operation temperature using a thin film Pt-heater structured onto the backside of the substrate.

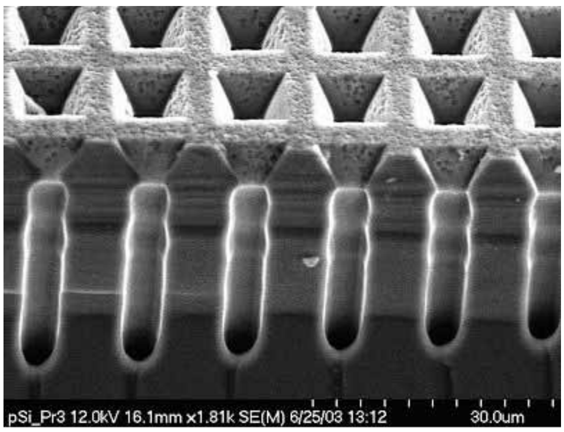

Fig. 1: SEM-picture of the IR-emitter structure based on regular ordered macropore arrays obtained by photo-electrochemical etching of prepatterned silicon substrates. The Pt- heater is at the backside of the chip

The lower detection limit of the optical ethylene monitoring system is mainly defined by the optical path length between emitter and detector. For monitoring the ripeness state of e.g. apples, the required detection limit is below 10 ppm. For this purpose a low cost “mini” White cell with an optical path length of 1.6 m was designed and implemented [3]. The main components of the White cell are an Aluminium body, three gold coated mirrors and a gas in- and outlet, respect-tively. The IR-emitter as well as the IR-detector are directly mounted at the White cell (fig. 2).

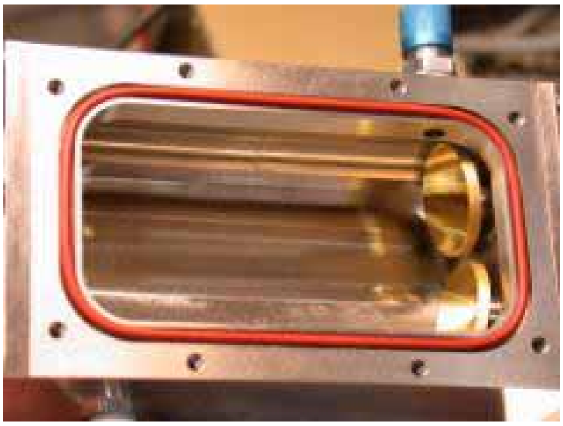

Fig. 2: Top view of the open mini-White cell with a 1,6m path lenght. The dimensions of the cell are 10,5cm x 4,5 cm x 6 cm.

In the first stage of the work commercial IR-detectors were used. The used IR-detector module from Perkin Elmer consists of two thermopiles with optical filters mounted in a TO5 housing. One for the ethylene signal detection at 10.6µm wavelength and one for the reference signal at 3,95µm. The emitter was chopped with 12Hz and the detector signals were evaluated with a lock in amplifier.

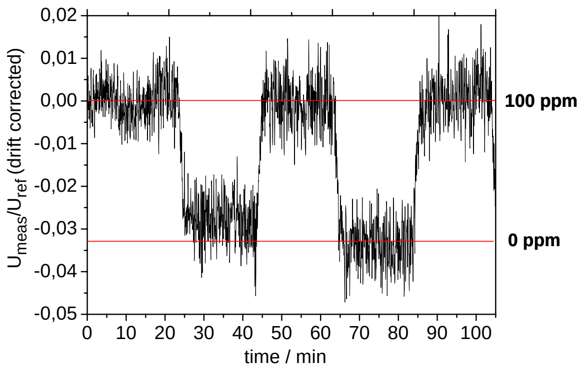

Fig. 3: Ethylene measurement using a 1,6m-white cell and a macro-porous-silicon-IR-emitter as infrared source and commercial twofold thermopile detector array (Perkin Elmer reference at 3,95 µm, ethylene at 10,5µm).

Figure 3 shows one of the first measurements, plotted is the relative signal ($V_{ethy.}$ /Vref.). Due to a not optimized packaging the housing of the emitter is heated up during operation which results a drift of the sensor signal Hence the measurement showed in figure 3 is drift corrected.

3 Summary and outlook

A small ethylene monitoring system for surveil-lance and control of fruit storage has been developed. The operation principle of the system is based on the IR-adsorption in fingerprint region of 10,6 µm. First C_2H_4 measurements have been carried out. Currently a 2x2 thermopile-detector array with integrated optical filters as detection unit and microstructured Fresnel lenses for the measurement of up to three gases and one reference channel is under development.

A first run of thermopile-detector arrays were processed for system integration. The characterised thermopiles showed good detectivities of about 10^8cm·(Hz)$^{0.5}$/W, which are comparable with commercial available thermopile-detectors. Next to ethylene ammonia, ethanol and acetaldehyde were considered for a first parameter set of the optical filters. The corresponding commercial optical filters were obtained. The first run of micro-struc-tured Fresnel lenses based on a binary structure were designed, processed and characterised, where-as the determined focal lengths fit well to the aimed values.

Furthermore the specifications of the electronics were worked out. In addition to a power supply for the modulation of the thermal emitter, it will contain a multiplexer for the signal processing of the 2x2 thermopile-detector array with pre-amplification stage and Lock-in-amplifier.

Acknowledgement

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