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INTEGRATED POLYMER WAVEGUIDES FOR ABSORBANCE DETECTION IN CHEMICAL ANALYSIS SYSTEMS

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ABSTRACT

A chemical analysis system for absorbance detection with integrated polymer waveguides is reported for the first time. The fabrication procedure relies on structuring of a single layer of the photoresist SU-8, so both the microfluidic channel network and the optical components, which include planar waveguides and fiber-to-waveguide coupler structures, are defined in the same processing step. This results in self-alignment of all components and enables a fabrication and packaging time of only one day. The fabrication scheme has recently been presented elsewhere for fluorescence excitation of beads [1].

The emphasis of this paper is on the signal-to-noise ratio of the detection and its relation to the sensitivity. Two absorbance cells with an optical path length of 100 μm and 1000 μm were characterized and compared in terms of sensitivity, limit of detection and effective path length for measurements of the dye Bromothymol Blue. The influence of three different bonding procedures on the spectrally resolved propagation loss of the integrated waveguides between 500 nm and 900 nm was furthermore determined.

INTRODUCTION

Fluorescence detection has been the most popular optical detection method in microfluidic devices, because a good signal-to-noise ratio can be achieved in micrometer-sized channels with the use of free-space optical elements. A disadvantage of fluorescence detection is that labelling of the molecules is typically needed, which complicates the fluidic handling and alters the physical and chemical characteristics of the target molecules. Absorbance detection, on the other hand, is a more universal method, since labelling can be avoided. The disadvantage of this approach, especially in microfluidic channels, is that the limit of detection is higher due to a limited optical path length, when the depth of the channel is used for detection.

The sensitivity can be increased orders of magnitude by detection along a channel segment in the plane of the device. This has been done previously by using free-space optical elements [2, 3], inserted optical fibers [4] and integrated planar waveguides [5, 6].

An increase in the sensitivity is, however, only desirable if it translates into an improvement of the signal-to-noise ratio (S/N) of the detection, which is not necessarily the case, since the sensitivity and the baseline noise are not independent.

THEORY

The signal-to-noise ratio of the detection is typically determined by dividing the measured absorbance value with the noise of the baseline. The relation between the variance in the baseline measured in absorbance units (ΔA) and the variance in the light intensity (ΔI) can be obtained by differentiating, $A=\log(I_0/I)$, with respect to the light intensity (I). The reference intensity (I_0) is kept constant for simplicity:

$$\Delta A = \frac{1}{\ln 10}\frac{\Delta I}{I} \qquad (1)$$

The governing equation for evaluation of the S/N is thus obtained by dividing the absorbance value given by Lambert-Beer's law (A=abc) with the variance (ΔA):

$$\frac{A}{\Delta A} = abc \ln 10 \frac{I}{\Delta I} \qquad (2)$$

a is the molar absorptivity, b is the optical path length (in absence of stray light) and c is the analyte concentration. In previous studies emphasis has mainly been on increasing the optical path length in order to improve the detection [2-6], but it is seen from eq. 2 that a reduction in the relative error of the measured light intensity (ΔI/I) is equally important in order not to get an unpleasant surprise when evaluating the performance of the system.

DESIGN AND FABRICATION

All structures were made in the photoresist SU-8, since it is a well-established process for fabrication of thick polymer layers with a high aspect ratio [7] and because it has been widely used for fabrication of channel sidewalls in microfluidic devices [8, 9].

The mask design of a device is seen in Fig. 1. It consisted of microfluidic channel sidewalls, planar waveguides and fiber-to-waveguide couplers. The channel network was arranged in a cross configuration, which is typically used for capillary electrophoresis experiments. It included an U-shaped absorbance cell with an optical path length of 1000 μm (present in the lower part of the design). Devices with channel widths of 30 μm and 100 μm were fabricated. The design also contained waveguides perpendicular to the channel width in order to have a path length of 30 μm and 100 μm, respectively. Fiber-to-waveguide coupler structures were included in

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order to ease the optical packaging of the device. They consisted of a tapered groove were fibers can be inserted to obtain self-alignment with the waveguides. This is a similar type of approach as has been used previously for both telecommunication [10] and microfluidic devices [1, 11].

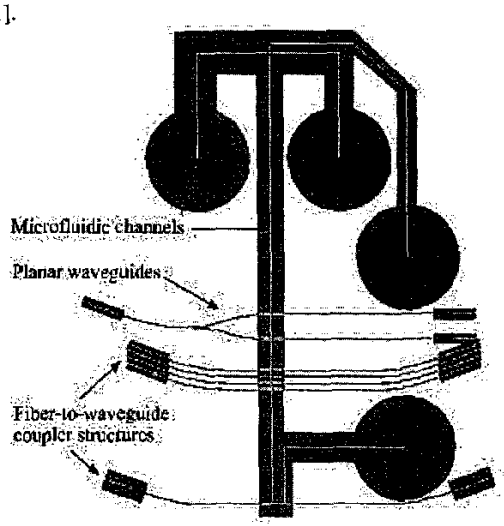

Figure 1. Mask design of a device (3.0 cm x 4.0 cm) consisting of microfluidic channel sidewalls, planar waveguides and fiber-to-waveguide coupler structures. The channel network consisted of an injection cross, a separation channel and an U-shaped absorption cell.

The fabrication procedure is shown in Fig. 2. and is similar to the procedure in [1], where the waveguides were used for fluorescence excitation of beads.

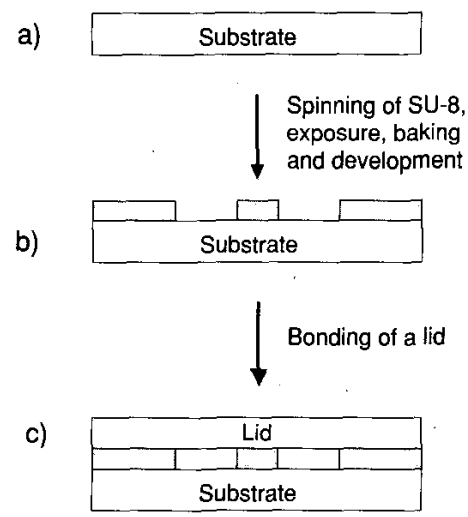

Figure 2. Fabrication procedure for integration of SU-8 waveguides, fiber-to-waveguide couplers and microfluidic channels.

Since both the optical and fluidic elements can be defined in the same layer only a single photoresist process was needed. The substrate was a 500 μm thick silicon wafer with 2.5 μm thick layer of thermally grown silicon dioxide (n=1.458 at 635 nm), which served as the waveguide cladding layer. A SU-8 layer (SU-8-2075, Microchem, U.S.A.) of 90±5 μm was spun onto the substrate (n=1.59 at 635 nm) and patterned by standard UV photolithography. A layer thickness of 90 μm was chosen, because it had to be thicker than the outer diameter of the fiber (68 μm, FVP050055065, Polymicro Technologies. Phoenix, U.S.A.), which otherwise could not be inserted in the coupler grooves. A waveguide with a large core has the additional advantage that more optical power can be coupled into the waveguides. This increases the detected light intensity, which should be favorable in terms of S/N (eq. 2).

The channel network was sealed after patterning of the SU-8 layer by bonding with either a glass or polymer lid. The lid also worked as the waveguide cladding layer. Three different bonding techniques were investigated in order to find the influence of the bonding on the waveguide properties. The three bonding techniques were: a) UV-activated adhesion bonding of a 500 μm thick Borofloat lid, with the use of an intermediate layer of 5-10 μm SU-8, b) lamination of a 60 μm thick polymer foil at 120-125 °C and c) pressing a PMMA substrate with a 100 μm thick intermediate layer of poly(dimethylsiloxane) (PDMS) on top of the structure. It was possible to obtain a hermetically sealed lid with all three processes for channels with a width of 100 μm. Channels with a width of 30 μm were sometimes clogged, especially in the case of the adhesion bonding, due to adhesive in the channels. This is discussed in more detail in [12].

WAVEGUIDE PROPAGATION LOSS

The spectrally resolved propagation loss of the integrated waveguides was calculated by measuring the transmission spectra of 4 waveguides with lengths between 2.52 cm and 4.52 cm. The insertion loss was calculated for each waveguide by normalization with a reference spectrum of the light source. A linear fit with respect to the waveguide length was subsequently calculated in order to obtain the spectrally resolved propagation loss for the three bonding procedures as seen in Fig. 3. The best performance was achieved with the PDMS bonding yielding a propagation loss of 1.4 dB/cm at 633 nm. A few other groups have reported on the propagation loss of integrated polymer waveguides in microfluidic devices. McMullin [11] obtained a propagation loss of 5 db/cm at 633 nm for laser written waveguides based on UV-curable optical adhesive and Lee *et al.* [13] achieved a loss of 4 db/cm for SU-8 waveguides that were fabricated by filling pre-fabricated silicon channels with the polymer solution.

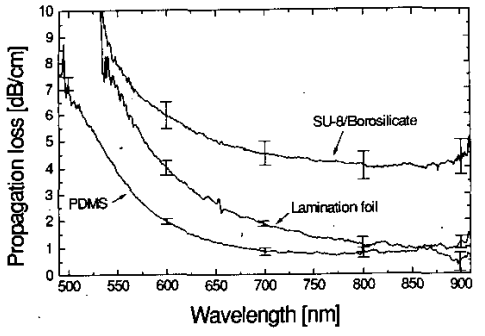

Figure 3. Spectrally resolved propagation loss between 500 nm and 900 nm of integrated waveguides fabricated with three different bonding procedures.

The propagation loss (Fig. 3) is higher at short wavelengths. This is attributed to absorption and is probably a result of the absorption tail of the photoinitiator in the resist. The waveguides with a laminated lid had a higher loss than the PDMS lid, especially at short wavelengths. This is probably caused by thermal degradation or 'yellowing' [10] during lamination, because the lamination temperature was 120-125° C compared to a standard baking temperature of 95° C after development of the SU-8.

The propagation loss of the integrated waveguide fabricated with a 5-10 µm thick SU-8 adhesion layer spun onto a borosilicate lid shows an increase of about 3 db/cm in the whole wavelength range compared to the waveguide with the PDMS lid. The increase is attributed to radiation loss in the upper part of the waveguide, because the adhesion layer and the waveguide core had the same refractive index, so the light was unguided in this region. This can be overcome by using an adhesion layer of a lower refractive index.

CHEMICAL ABSORBANCE CELLS

Fig. 4. shows a microscope picture of a finished device, where light from an Argon-ion laser was coupled across a 1000 µm long absorbance cell through the integrated waveguides. The picture corresponds to the lower part of the design in Fig. 1. The channel width in this case was 30 µm and bonding was achieved by pressing a 500 µm thick borofloat glass wafer with an intermediate layer of PDMS against the SU-8 structure of the bottom substrate. The light path of the absorbance cell was visualized by filling the channel network with a 170 µM fluorescein solution. Excitation of the fluorescein solution was achieved by coupling light with a wavelength of 488 nm from an Argon-ion laser that was connected to the integrated waveguide in the right side of the picture through an inserted optical fiber.

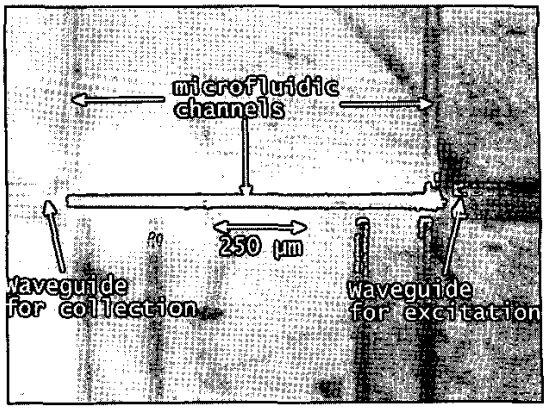

Figure 4. Microscope picture of a 1000 µm long chemical absorbance cell with integrated optical waveguides. The channel was filled with a 170 µM fluorescein solution to visualize the light path, when light from an Argon-ion laser (488 nm) was coupled into the excitation waveguide.

In another absorbance cell, the width (100 µm) of a separation channel was used as the optical path length. The performances of the absorbance cells were investigated by measuring a calibration curve of each cell (Fig. 5) at 633 nm with the dye Bromothymol Blue (Sigma-Aldrich, Denmark).

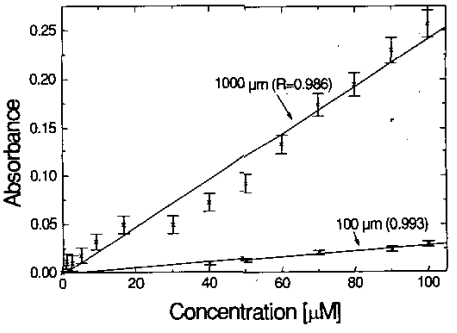

Figure 5. Calibration curves of absorbance cells with a nominal path length of 100 µm and 1000 µm, respectively. The measurements were done with the dye Bromothymol Blue at 633 nm.

Based on these measurements, the sensitivity, limit of detection and effective path length of the absorbance cells were determined (Table 1). The sensitivity was given by the slope of the calibration curve, while the limit of detection was determined for a signal-to-noise ratio of two. The effective optical path length was calculated by normalization with absorbance measurements on a commercial spectrometer (Ultrospec 3000, Pharmacia Biotech, U.S.A.). This procedure is described in detail in [12].

Table 1. Sensitivity, limit of detection (LOD) and effective path length for absorbance cells with a nominal length of 100 μm and 1000 μm.

Nominal path length [μm]	100 μm	1000 μm
Sensitivity x10^3 [M^{-1}]	0.3±0.02	2.4±0.1
Limit of detection [μM]	30±5	15±5
Eff. path length [μm]	113±8	906±40

The decrease in limit of detection was only about two-fold when going from a 100 μm to a 1000 μm long absorbance cell, even though the increase in sensitivity was about eight-fold. The reason for this is due to an increase in the coupling loss from 5 dB (100 μm) to 20 dB (1000 μm), which increases the relative error of the measured light intensity and hence lowers the S/N, as seen by eq. 2. The lowest limit of detection was only 15 μM (1000 μm path length). This is mainly due to noise in the light source, since an unstabilized HeNe laser was used.

CONCLUSIONS

The spectrally resolved propagation loss of integrated polymer waveguides with microfluidic channels was determined for three bonding procedures. The lowest loss was 1.4 dB/cm at 633 nm and was achieved for PDMS bonding.

Two chemical absorbance cells with an optical path length of 100 μm and 1000 μm were furthermore fabricated and tested in terms of sensitivity, limit of detection and influence of stray light by absorbance measurement at 633 nm with the dye Bromothymol Blue. The decrease in limit of detection between the 100 μm and the 1000 μm long absorbance cell was only about two, even though the increase in sensitivity was about 8. This discrepancy is attributed to an increased relative error of the measured light intensity in the case of the long absorbance cell, which in turn was due to an increase in the coupling loss over the channel.

Acknowledgements

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