AN OPTICAL INJECTION SYSTEM USED FOR OPTIMALIZATION STUDIES OF HIGH ASPECT RATIO LC COMOSS COLUMNS

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Abstract

A method was developed to evaluate the influence of the pillar configuration on the dispersion characteristics in pillar arrays intended for use in liquid chromatography. This method consists in uncaging a narrow plug of dye through a micromachined window. A proof-of-principle of the method is demonstrated and a first plate height determination was performed.

Keywords: uncaging dye, comoss, liquid chromatography, pillar array, injection

1. Introduction

Recent computational fluid dynamics (CFD) studies have shown that it would be highly desirable to replace the packed bed columns used in liquid chromatography by 2D structured pillar arrays [1]. Dimensional parameters such as pillar and wall spacing and pillar shape appear to be crucial for achieving higher efficencies [2]. Even though optimal dimensional values are available by CFD studies, no experimental validation of these parameters has been performed so far. This is in part due to the fact that it is not straightforward to convert a proposed design into the desired pillar array without altering the original dimensions. Different process steps such as lithography, Bosch etching and even bonding give rise to minute alterations of an original design, which lead to large deviations of the predicted fluidic behaviour. Another issue is that in order to study band broadening in detail, a good injection system should be available. A challenging aspect is the high aspect ratio of the channels that are needed, making it extremely difficult to inject undisturbed and rectangular plugs [3].

2. Experimental

A virtual injection system is presented here which allows to create very wide and perfect rectangular plugs by means of an uncaging dye [4]. We fabricated a 30 µm × 1 mm window inside a pillar array channel, through which laser light could in situ create a fluorescent plug (Fig.1). Due to the positioning of the window in contact with the channel, no increase in peak width due to light diffraction effects occurs (Fig.2). Because of the configuration of our system (the laser is situated at the silicon side and the objective lens is at the glass side), the plugs could be observed from the exact moment that they are being generated. For the fabrication of the chip, we first etched the pillars by Bosch etching. Subsequently, 300 nm stress free silicon nitride was deposited to provide a transparent etch stop for subsequent KOH etching of the (100) silicon wafer. The thickness of the silicon nitride layer can be easily varied in order to increase the pillar dimensions and to decrease the microchannels between the pillars. This provides an easy method to vary submicron distances with a normal UV lithography equipment. Finally the pillar channel was sealed by anodic bonding to a Pyrex® substrate.

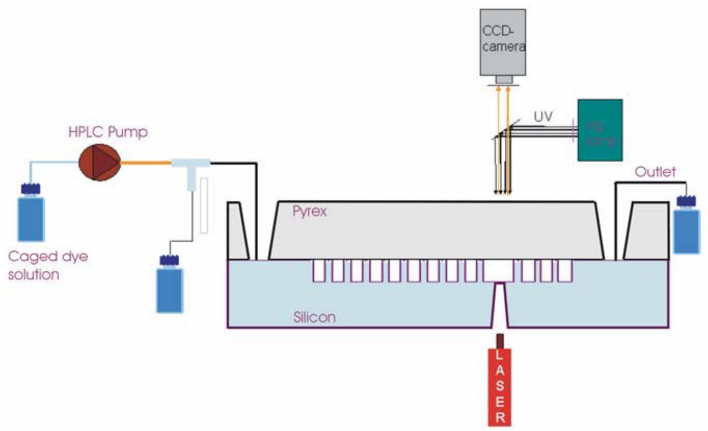

Figure 1. Setup showing operation principle. A solution containing 2 mg/ml of the caged dye bis-(5-carboxymethoxy-2-nitrobenzyl) ether dikalium salt in DI water is pumped via a flow splitter through the pillar channel. Applying the laser light uncages a plug defined by the window by chemical modification. This generated plug is subsequently monitored by a CCD camera. On the bottom side of the wafer a 2 µm thick layer of PECVD SiO_2 and a 1 mm thick layer of PMMA was applied for improving the mechanical strength of the window (not shown).

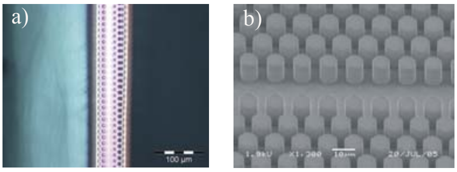

Figure 2. a) Bottom view of the injection window. The inclined walls are due to the KOH etching. The width of the window is about 30 µm, which is larger than the intended 10 µm due to errors on the wafer flat orientation and alligning.

3. Results and discussion

A plug was created in one of the channels with pillar diameters of 10 µm (Fig.3). A theoretical plate height value of 9,7 ± 0,3 µm (n=4) was determined for the peak zone (Fig.4), based on a flow velocity of 0.94 mm/s. Experimental work is under way to evaluate and optimize the performance of different pillar configurations using the presented technique.

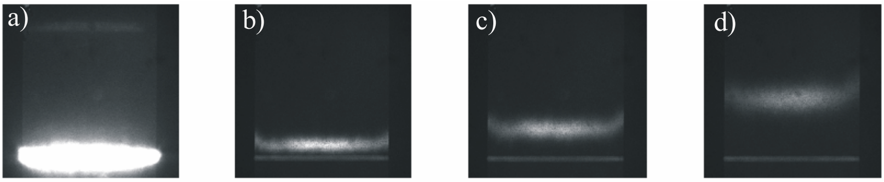

Figure 3. a) Optical injection at time 0 s by applying the laser source, b) a fluorescent plug becomes visible after turning of the laser at t= 0.938 s, the peak moves along the channel: c) t=2.189 s, d) t=4.534 s. It can clearly be seen that the flow velocity is faster at the side of the channel. This is due to a local low flow resistance and can be cancelled by optimizing the distance of the side wall to the closest pillar.

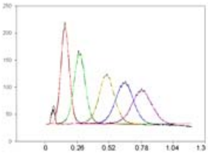

Figure 4**:** Plot of the intensity (a.u.) versus the distance travelled in the channel (mm).The intensity profiles were determined by discarding the region close to the side wall and averaging the intensities along the width of the channel.

4. Conclusions

We have fabricated a pillar array column with an integrated functionality that allows the creation of fluorescent plugs as narrow as 30 µm. This device can be used to evaluate the performance of different pillar configurations in terms of band broadening with the prospect of optimizing liquid chromatography on a chip.

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