

Micromachined Mixing Device for FTIR Spectroscopy

P. Svasek¹, E. Svasek¹, P. Hinsmann², B. Lendl², M. Harasek³
and M.J. Vellekoop⁴

¹ Ludwig Boltzmann Institute of Biomedical Microtechnology, Vienna

² Institute of Chemical Technologies and Analytics, Vienna University of Technology

³ Institute of Chemical Engineering, Vienna University of Technology

⁴ Institute of Industrial Electronics and Material Science, Vienna University of Technology

Time-resolved FTIR-spectroscopy is a powerful method for the investigation of chemical reactions, especially in the field of biochemistry. It allows obtaining structural information of molecular dynamics. This method requires fast mixing of at least two reactants as well as a very short optical pathlength because of the strong IR-absorption of water and organic solvents. To meet these requirements a micromachined mixing device was developed which combines fast diffusion based mixing and an optical pathlength of less than 20 μm . The devices are batch fabricated on 4" CaF_2 wafers.

Introduction

Time-resolved Fourier transform infrared spectroscopy is an efficient technique to study chemical reactions and obtain structural information of molecular dynamics [1], [2]. This method requires fast mixing of the reactants. Additionally, the event under study must be precisely repeatable to provide a good signal/noise ratio. Water and organic solvents show a very high absorption in the mid-IR range, therefore the optical pathlength of the device has to be very short, typically less than 20 μm . The principle of the mixing device is shown in Fig. 1.

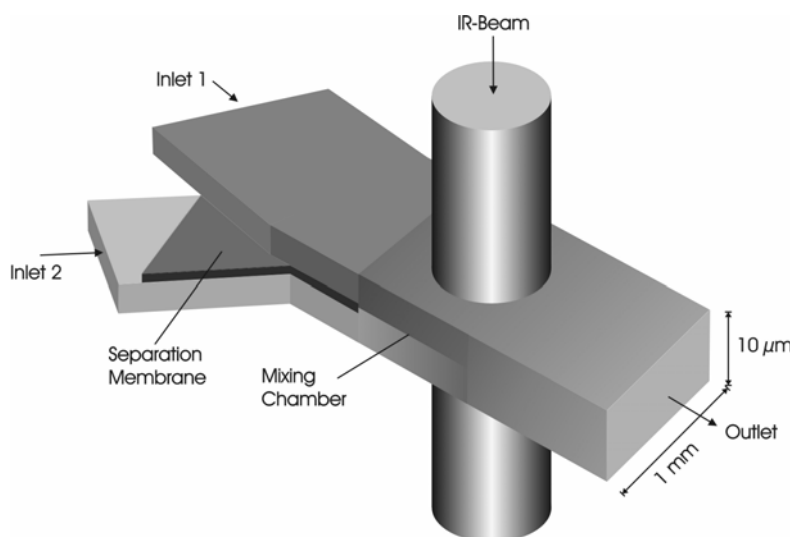


Fig. 1: Schematic drawing of the micromixer (drawing not to scale).

The mixer is operated in the “stopped flow” mode. Two liquids are injected by means of a double piston syringe pump into inlet 1 and inlet 2. These two streams are separated by a metal separation membrane until they enter the mixing chamber. Inside this chamber the liquids form two thin sheets which are superimposed. While the flow is on there is hardly any mixing due to laminar flow conditions in microfluidic devices. As soon as the flow is stopped, which is done by means of a dedicated fluidic setup [2], the liquids are mixed by diffusion. Diffusion is a rather slow process; therefore the distances have to be kept as short as possible. Fluid dynamic simulations performed with FLUENT V5.5 showed as a result a mixing time of 100 ms for water based solutions. The height of the mixing chamber was 20 μm [1]. The mixing chamber of the latest device is 10 μm high, therefore one can assume a mixing time of ~ 25 ms.

Preparation of the Mixing Device

The devices are fabricated using micromachining methods like photolithography and waferbonding. As a substrate material we use CaF_2 because of its superior optical properties. The fluid channels are formed by two layers of the negative-working epoxy based photoresist SU-8. During the last years this material has been widely used because of its very good mechanical and chemical properties.

The devices are built up as follows: A 4 μm thick layer of SU-8 is deposited by spin-coating, softbaked, exposed, post-exposure-baked, but not developed. On top of this layer a 2 μm thick layer of Ag is deposited by evaporation. This metal layer is then covered by positive photoresist AZ 1512 HS. The photoresist is patterned as usual and the Ag layer is etched to obtain the structure of the “separation membrane” (see Fig. 1). As an etchant a 45 % (WT) solution of $\text{Fe}(\text{NO}_3)_3$ in water is used. This solution does not attack the SU-8. Finally the SU-8 is developed with PGMEA. To dissolve unexposed SU-8 under the metal structure takes quite some time (approx. 2 hours for channels of 1 mm length). Wafer #1 (the “bottom” wafer) now carries the structures of Inlet 2 and the separation membrane.

On wafer #2 (the “top” wafer) the structure of inlet 1 is fabricated as usual from a 4 μm thick layer of SU-8. This wafer contains the holes for two inlets and one outlet per mixer. These holes are conventionally drilled by means of a high speed spindle attached to a computer-controlled mill. So far the SU-8 on both wafers is not hardbaked and therefore the polymer is not completely crosslinked. Both wafers are superimposed face to face, and aligned. Hardbaking is done in an EVG 501 wafer bonder. A force of 1700 N is applied and the wafers are heated to 200 $^\circ\text{C}$ for 1 hour. Because of this high temperature the polymer is completely crosslinked and a bond is established between SU-8 and the opposite Ag-structure (see Fig. 2).

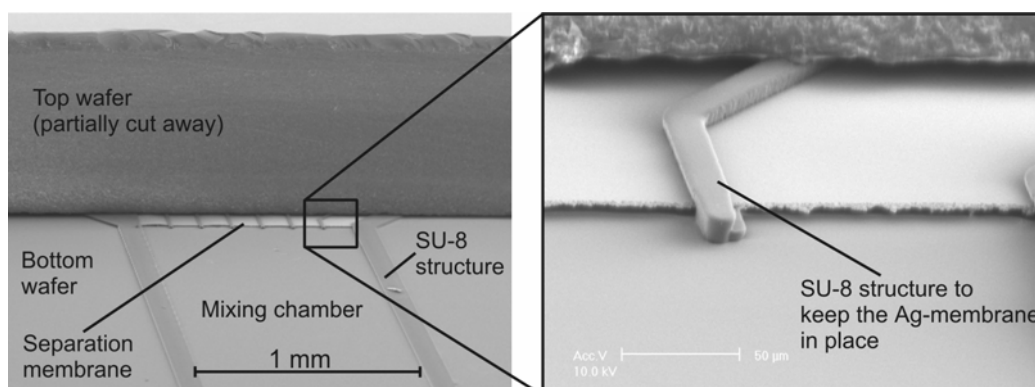


Fig. 2: SEM micrograph of the mixing device. The top wafer is partially cut away to show the SU-8-structures and the separation membrane.

The stream of liquid from inlet 1 flows between the top wafer and the separation membrane, the stream from inlet 2 flows between the separation membrane and the bottom wafer. At the edge of the membrane the streams meet and enter the mixing chamber (see Fig. 2).

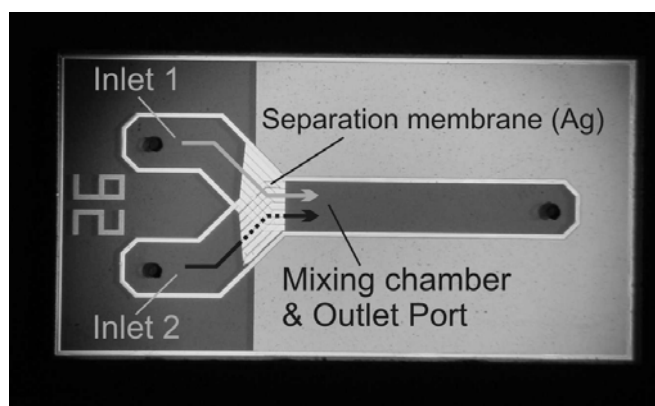


Fig. 3: Micrograph of the mixing device after dicing. The dimension of the device is $6 \times 12 \times 2 \text{ mm}^3$.

Results Obtained from Model Reactions

If reactants are used which react very fast, the reaction is limited by diffusion only. Consequently in this case the experiment is actually a test of the mixer performance (see Fig. 4). It can be observed clearly that the mixing time meets the result of the CFD simulation. Some premixing (i.e. formation of reaction product) can be observed while the flow is on. Premixing cannot be neglected in case of very fast reactions [1]. In case of a slow reaction no formation of the reaction product can be observed, as shown in Fig. 5. This reaction is completed after a few hundred milliseconds.

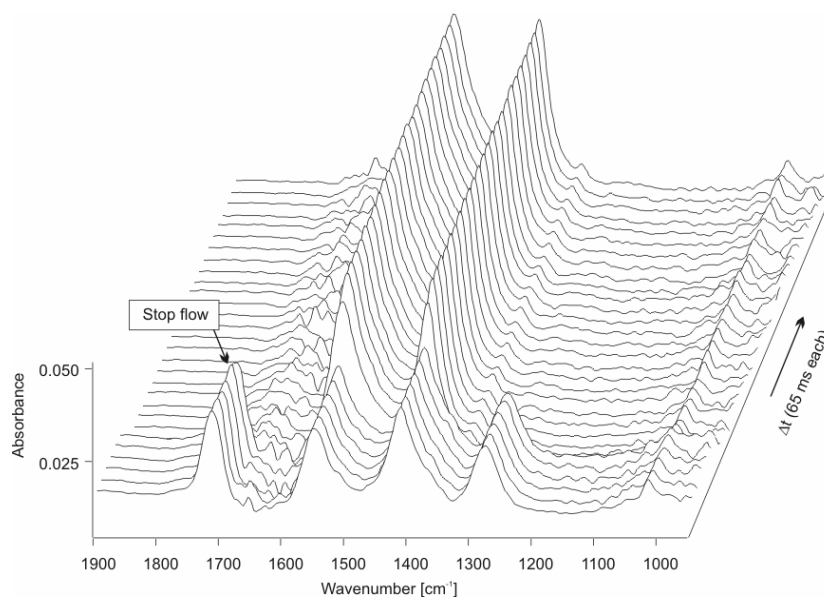


Fig. 4: Stack plot of FTIR spectra obtained from the reaction of CH_3COOH and NaOH . Time delay between subsequent spectra is 65 ms.

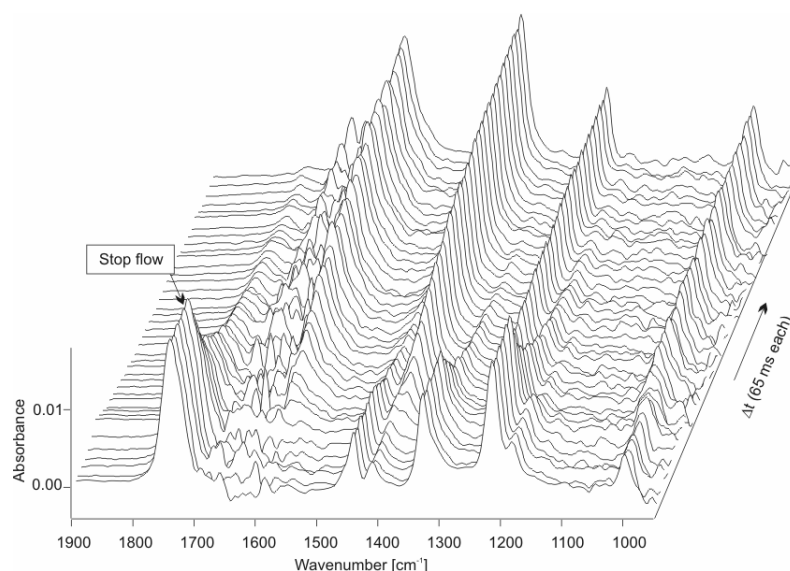


Fig. 5: Spectra obtained from the reaction of methyl monochloroacetate and sodium hydroxide. Time delay between subsequent spectra is 65 ms.

Conclusion

The micromachined mixing device presented in this paper offers the opportunity to investigate the dynamics of chemical reactions, especially in the field of biochemistry [3]. The results of the CFD simulations were confirmed by experiments. To realize this device a special technique was developed that combines two layers of SU-8 photoresist and one layer of metal. This technique is useful for the fabrication of other microfluidic devices as well. The devices are produced in a batch process using 4-inch wafers of Calcium Fluoride, a material that shows very good optical properties.

Acknowledgement

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References

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