# Silicon low flow microreactor for measuring catalytic activity

Toke R. Henriksen[1]*, Peter Vesborg[2], Jakob L. Olsen[2],
A. Monkowski[1], O. Hansen[1], and I. Chorkendorff[2]
[1]*Department of Micro- and Nanotechnology,*
*Technical University of Denmark, 2800 Kgs. Lyngby, Denmark*
[2]*Center for Individual Nanoparticle Functionality, Department of Physics,*
*Technical University of Denmark, 2800 Kgs. Lyngby, Denmark*
*toke.henriksen@nanotech.dtu.dk (corresponding author)

## Introduction

A microreactor is a chemical reactor with dimensions in the micrometer range. Microreactors can be applied as analytical tools for characterizing the chemical kinetics of catalytic processes. Compared to conventional mini-tubular reactors, microreactors offer a number of advantages, such as the capability of analyzing very small amounts of catalysts, short response time, accurate control of experimental parameters, and improved safety [1][2]. We present a novel continuous-flow microreactor designed for high sensitivity measurements of catalytic activity. The device is intended for reactions with gas-phase reactants and products and is well suited for heterogenous catalysis as well as for photocatalysis. It is possible to conveniently heat the reactor to elevated temperatures.

## Materials and Methods

The microreactor consists of a channel system etched in a silicon chip, which has been hermetically sealed with a Pyrex lid (fig. 1). The reactant gases are introduced into the chip through two inlets, which allow for on-chip mixing. A high gas flow through a main flow channel system ensures that the gas exposed to the catalyst has the same composition as the feedgas mixture. A small fraction of the reactant gas, about $10^{14}$-$10^{15}$ molecules per second, enters the reaction chamber, which contains the catalyst. The product gas leaves the chip through an outlet to a mass spectrometer, which analyzes the gas composition. Between the reaction chamber and the mass spectrometer an on-chip flow-limiting capillary is placed. The microreactor is characterized by directing the entire flow through the catalyst bed to enter the mass spectrometer. This increases the sensitivity significantly compared to microreactors where only a small fraction of the product gas is analyzed. The entire chip has a length of 20 mm, a width of 16 mm, and a thickness of 850 µm. The silicon chip is fabricated using photolithography, reactive ion etching and thermal oxidation. The catalyst is deposited in the reaction chamber, and the channel system is subsequently sealed with a Pyrex lid using anodic bonding. The Pyrex lid contains a temperature sensor consisting of a platinum thin film resistor.

## Results and Discussion

Fig. 2 shows a result of a catalyst characterization using the experimental setup, illustrating an application of the microreactor. The plot shows mass spectrometer currents as a function of time for photocatalytic CO oxidation ($2CO+O_2 \rightarrow 2CO_2$) in the microreactor using 10 µg Degussa P25 $TiO_2$ powder as photocatalyst. The experiment is carried out at room temperature and the reactant gas is a mixture consisting of 90% He, 5.0% CO, 2.5% Ar, and 2.5% $O_2$. The microreactor is illuminated through the Pyrex lid using a mercury lamp with a spectrum centered at 365 nm. It is seen that the concentration of $CO_2$ rises due to the photocatalytic reaction during irradiation from the lamp. The reaction stops during periods of no irradiation. A response time of only ~ 10 s is observed.

## Significance

The microreactor presented here is intended to be a versatile tool for fast and easy measurements of catalytic activity under well controlled conditions. Furthermore, it is capable of characterizing very small amounts of catalysts. This makes it well suited for studies of model systems of catalytic nanoparticles fabricated using e.g. electron-beam lithography, which can only be produced in small quantities [3].

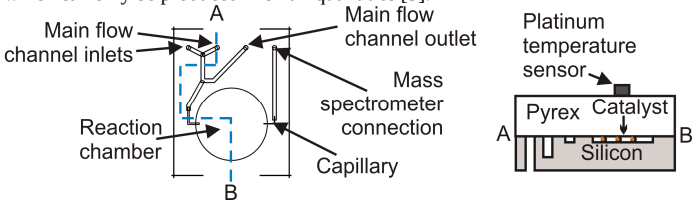


**Figure 1.** Left: Sketch of the microreactor design. The reactant gases are introduced through the two main flow inlets, which allow for on-chip mixing. The dashed, blue line defines the cross section to the right. Right: Cross section of the microreactor.

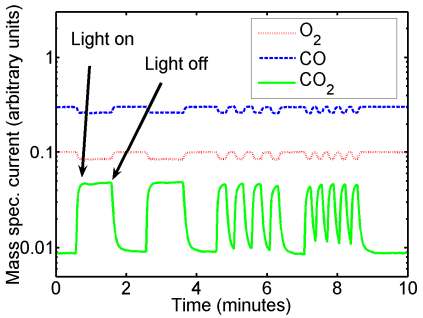


**Figure 2.** Mass spectrometer currents as a function of time. When irradiated by a mercury lamp, $O_2$ and CO react under influence of the $TiO_2$ catalyst to form $CO_2$. This is seen as a rise in the $CO_2$ signal and a corresponding drop in the $O_2$ and CO signals.

## References

1. Ajmera, S.K., Delattre, C., Schmidt, M.A., and Jensen, K.F., *Sensors Actuat. B* 82 297 (2002).
2. Younes-Metzler, O., Svagin, J., Jensen, S., Christensen, C.H., Hansen, O., and Quaade, U., *Appl. Cat. A* 284 5 (2005).
3. Johansson, S., Fridell, E., and Kasemo, B., *J. Vac. Sci. Technol. A*.18(4) 1514 (2000).