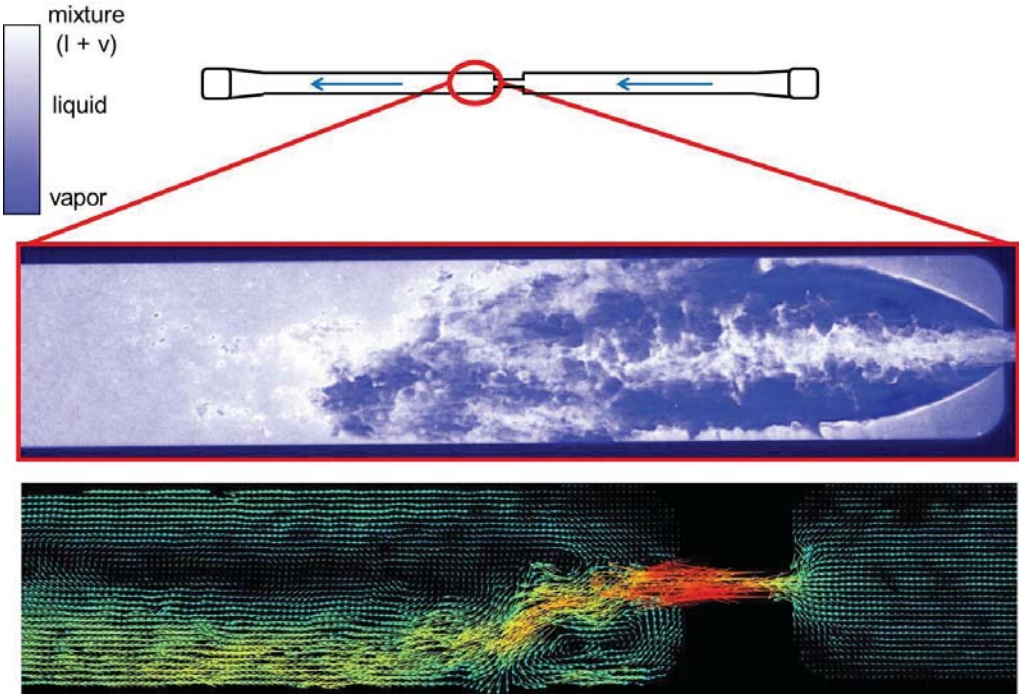
Incorporation of a bifunctional Pd/ZSM-5 catalyst in chip-microreactors

the acting stresses. Hence, a special technique has been developed to visualize the vapor filled cavities in the microchannels (Fig. 1). With a sufficiently high counter pressure the occurrence of vapor phase can be suppressed to a certain extent which depends on the microchannel geometry and the volume stream. In order to analyze the effect of channel geometry on the flow and the occurrence of vapor filled cavities, geometrical variations were applied to the orifice channel. The combination of dispersion experiments with ÂµPIV- and cavitation measurements enables evaluations regarding the main dispersion mechanism: In orifice geometries hydrodynamic stresses seem to be the dominating stress mechanism and cavitation seems to have a minor influence on the dispersion process. Beside the dispersion efficiency, abrasion of the micro systems is an important issue in high pressure dispersion. Therefore, abrasion caused by cavitation and by particles has been analyzed by REM-images which enabled a clear differentiation between the two abrasion sources.



Session: Structured Materials: Reactors
Time: 14:50 - 15:15
Abstract Nr: 241

INCORPORATION OF A BIFUNCTIONAL PD/ZSM-5 CATALYST IN CHIP-MICROREACTORS

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The use of chip-microreactors for fine chemical synthesis is a rapidly growing field worldwide as a result of the advantages of microfluidic technology over conventional chemical synthesis. However, the ability to incorporate a heterogeneous catalyst into these microreactors remains a challenge, specifically in terms of catalyst loading and reproducibility. Zeolite coatings offer a unique solution through the hydrothermal synthesis of the zeolite onto the microchannel walls. The incorporation of zeolites into microreactors typically requires the hydrothermal synthesis to initially be conducted on the individual microchannel plates, followed by an assembly step to form the closed microchannels. However, in the case of chip-based microreactors, post-assembly of the microchannels after coating is not feasible which alludes to the need of an alternative coating method.
In this study, a new zeolite coating method has been developed whereby hydrothermal synthesis of ZSM-5 is conducted within the microchannels (Figure 1). The effect of microchannel pretreatment, hydrothermal synthesis time and temperature, and precursor composition were investigated in terms of zeolite crystal size, morphology and orientation, as well as coating thickness, adherence and uniformity. The ZSM-5 coating was impregnated with various loadings of palladium to form the bifunctional catalyst. The coating was evaluated by XRD, SEM, TEM, and NH3 - TPD methods. The catalytic activity of the bifunctional catalyst for the multiphase reaction of acetone to MIBK was assessed in the microreactor configuration.
It was found that there exists a narrow range of synthesis conditions leading to the preferable zeolite coating formation as opposed to bulk crystallization. Various pretreatments were carried out in order to suppress bulk

crystallization, as well as improve the catalyst loading. Zeolite growth directly onto the microchannel surface resulted in a single layer of b-orientated crystals. However, by introducing an additional source of nutrients, the catalytic loading could be improved. Pretreating with a silica precoating resulted in the transformation of the amorphous silica to a multilayer of randomly oriented ZSM-5 crystals. The effect of various synthesis conditions was further evaluated in order to optimize the coating thickness, crystal size, and acidity. The catalytic performance of the bifunctional Pd/ZSM-5 coatings was evaluated for the production of MIBK.
Hydrothermal synthesis inside the microchannels of chip-microreactors offers a novel method for preparing zeolite coatings. By applying various microchannel pretreatment methods and optimizing the hydrothermal synthesis conditions, a bifunctional Pd/ZSM-5 coating could be prepared and applied to the production of MIBK.

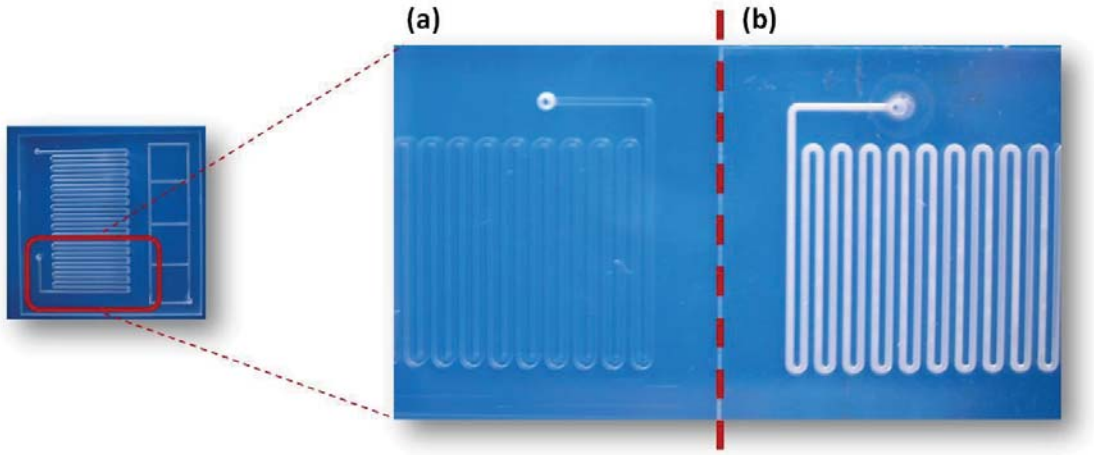

Figure 1: Glass chip-microreactor (a) uncoated, (b) ZSM-5 coated

Session: **Structured Materials: Reactors**
Time: **15:45 - 16:10**
Abstract Nr: **979**

PHOTOCATALITIC PERFORMANCES OF NANOSTRUCTURED/HIERARCHISED TIO2 ELECTRODES

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Background - The effectiveness of solar-driven photo-catalytic processes is dictated to a great extent by the semiconductor capability of absorbing visible and infrared light, as well as its ability to suppress the rapid recombination of photo-generated electrons and holes. Nanotubular arrays have the peculiar characteristic of guiding and accelerating the path of the photo-generated electrons, thus limiting the recombination. 3D or multiscale structures can increase the effect of light scattering, which plays a fundamental role in increasing the overall efficiency of the process.
Aims - In the present work a very recently developed methodology involving the coupling of electrochemical and hydrothermal growth techniques was adopted to obtain a 3D hierachical structures. In the specific, TiO2 nanoparticles were deposited within a nanotubular matrix or over a compact TiO2 layer. The photo-catalytic performance of the resulting samples was analysed and compared with that of the related not hierachised structures.
Methods - The synthesis of samples started from Ti foils which were anodised in organic solvent and in the presence of fluorides. A ramp of potential was applied from the OCV value to the final oxidative potential of at least 20V which was maintained for a due time. Compact oxide layers were also prepared with the same procedure but with solution in absence of fluorides.
In order to perform the hierarchisation, the basic structure was immersed in diluted HCl solution and stirred for 20 min at room temperature. A Ti precursor was then added dropwise to the mixture and stirred for 1 h. The solution was heated at temperatures ranging from 80 to 140Â°C for a due time, under slight stirring. After the reaction, the substrate was withdrawn, rinsed with distilled water and ethanol and finally annealed at 400Â°C.
Results - SEM analyses showed that this procedure was able to originate a hierarchical structure in which