MICROREACTORS

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Contents

Summary

Microreactors open a novel way of chemical synthesis in a highly controlled way. Due to their improved mass and heat transfer, these devices are well suited, e.g., for mixing-sensitive, fast and highly exothermic reactions. Endothermic reactions profit as well from the improved thermal management. This leads to a better exploitation of resources and decrease of energy consumption of chemical processes and will hereby increase their costing and eco-efficiency. An additional interesting benefit is the enabling to run reactions in a safe way under unconventional process parameters known as *new process windows*. This includes processing at unusually high temperatures and pressures - high-p-T processing. In this overview, selected examples from the diversity of microstructured components are given and grouped by their scopes of applications. This comprises first microreactors or micromixers for liquid / liquid contacting, followed by microdevices for gas / liquid contacting and then by catalytic gas-phase microreactors.

1. Introduction

This chapter gives a review of the different types of microreactors, their benefits and their usage in chemical engineering. First, general characteristics of microreactors are discussed [37] - excellent mass and heat transfer, safety issues, diffusion transport etc. - in the following, different types of reactors are presented and some examples of use are going to be shown. Then, important aspects for industrial application, like cost analysis, eco-efficiency analysis and process safety, will be shortly discussed.

In the last years, great efforts have been undergone to carry out reactions at small scale. These undertakings led to the field of *Micro Process Technology*, which includes the concept of doing chemistry in a different way by improving and intensifying existing processes. Microreactors are typically continuous systems and the microstructure dimensions have to be small enough to provide the necessary heat transport and mass transfer. The prefix “micro” relates to the inner life of the reactor and not to the components that are build around.

There is a wide range of manufacturing techniques from established methods that stem from microelectronics [1, 4, 5] up to modern ultra precision engineering and micro erosion techniques [1, 6, 7]. These versatile techniques allow a large variation of accessible microreactor materials such as steel, metals, silicon, ceramics, etc. as well as the lab material classes polymers [1, 8, 9] and glass [1, 10].

The main argument for using microstructured components in a chemical process is *process intensification* [37]. This subsumes first of all order-of-magnitude changes in

conversion and selectivity or increased space-time yields, but is more and more used in a broader definition addressing also waste reduction, energy savings and better safety. Totally, this means having much increased performance at the same reactor footprint or, vice versa, shrinking down the reactor dimensions [11]. The preferential way for the usage of such microstructured components is to implement them into a running process at the point where they are needed or where their benefits can intensify the course (plant retrofit approach).

2. General Benefits of Microreactors

There are several benefits of microstructured reactors [12, 37]. The usability of microreactors for highly exothermic reactions is based on their large surface / volume ratio. Thereby, the heat, which is generated by the reaction, can be transferred very efficiently out of the system. This avoids hot spots and the formation of side products, as e.g. evident from coloration of the product. Besides, endothermic reactions can be heated up to reaction temperature more efficiently than in conventional batch vessels.

Another benefit is to achieve small characteristic lengths for transport of matter in the range of a few ten to less than a micron, which accelerates the diffusion transport so that operation under intrinsic kinetic conditions can be achieved. This makes microstructured reactors well suited for very fast reactions, even when being mixing sensitive. Industrial reactors have typically an interface / volume ratio of some 10 m^2 m^{-3} up to 100 m^2 m^{-3}. Conventional lab-scale apparatus comprise typically a few 100 m^2 m^{-3} and in a few cases have at best 2000 m^2 m^{-3}. Opposed to this, the surface / volume ratio for microstructured elements is very large up to 20000 m^2 m^{-3} and higher. Thus, microreactors are ideal tools for multi-phase reactions, e.g. to work under kinetically controlled conditions while keeping the conversion rate at maximum.
An additional benefit of these small devices is the very low inner volume, which is important for process safety, e.g. to limit the damage potential for worst-case scenarios in case of a process upset.

The exact defined and short residence times in such systems make it possible to optimize selectivity and space-time yield.

3. Selected Types of Microreactors

When categorized by the presence of the phase(s) involved, microreactors can be subdivided further by the types of contacting principles typically employed as first step to initiate reaction by mixing. Thus, microreactors are grouped here for contacting liquid and liquid, gas and liquid or gas and gas phases. In the following, a few examples stemming from such classes are given to exemplify the diversity of microreactors for the different applications.

3.1. Liquid / Liquid Contactors (Micromixers)

A large number of mixers with different mixing principles are available for contacting liquids and liquids. Pure mixing tasks are, e.g., given for making disperse liquid / liquid systems such as emulsions or dispersions. Mixing has to be combined to reaction for

single-phase or multi-phase organic systems. If the reaction demands more time than provided by the mixing chamber (which is common), delay loops such as tubes or capillaries are added to the system for completing the reaction. Alternatively, a micro heat exchanger-reactor may follow the mixer in case of higher demands on heat transport. If a catalyst is required for the reaction, the simplest approach is to fill tubes or chambers in microdevices with powders or particles. A more advanced and tailored way of catalyst provision, however, is the coating of the channel walls, e.g. by slurry, spraying, sol-gel or vapor deposition techniques. Some micromixers with different mixing principles are shown in Figure 1. Their special mixing principles are going to be explained in the following sub-sections.

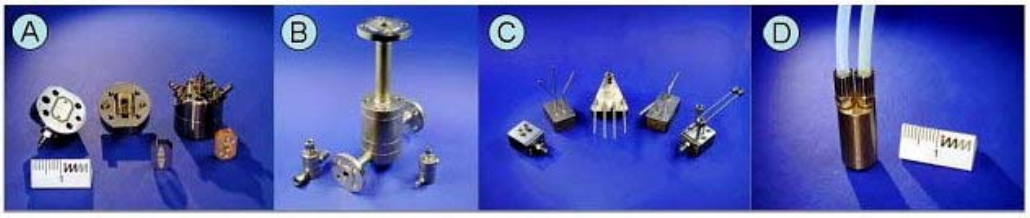
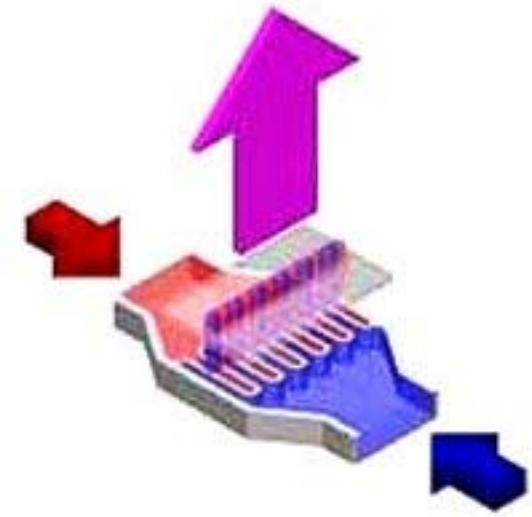


Figure 1: A: Interdigital mixer for diffusion mixing, B: Nozzle ("Star Laminator") mixer for jet mixing, C: Bas-relief ("Caterpillar") mixer for recirculation (convective) mixing (medium and high Reynolds numbers) and split/re-combination mixing (low Reynolds number), D: Impinging jet mixer for collision mixing via jets. (by courtesy of IMM).

3.1.1. Lamination and Hydrodynamic Focusing

Lamination means reduction of fluid distances to an extent that diffusion on its own becomes effective (under laminar-flow conditions). Hydrodynamic focusing, typically done by geometric constraints, is a simple means to further reduce the characteristic dimensions. In this way, mixing times in order of milliseconds and below can be achieved. Laminated streams are easily parallelized (multi-lamination) by grouping several inlet channels or nozzles. In a typical interdigital micromixer configuration, two fluids are fed via two separate channels, split in many sub-streams, which are then re-positioned so that combination can be done in a central mixing chamber. In this way, small lamellae alongside are yielded and further compression of these fluid layers by hydrodynamic focusing can follow the lamellae formation. The thickness of the lamellae can also be affected by setting the two flow rates of the liquids at different levels which uses hydrodynamic compression by virtue of pressure (without geometric constraining) [38 - 40].

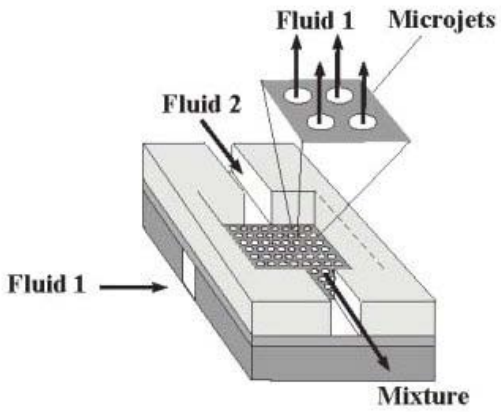


Scheme 1: Multi-lamination mixing principle of an interdigital micromixer, (by courtesy of IMM).

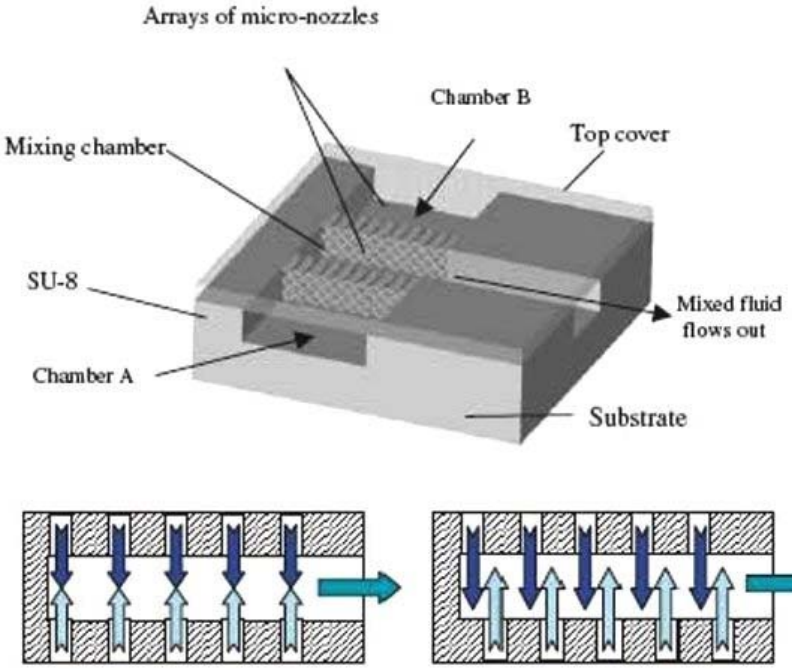
3.1.2. Jet Mixing

One of the first micromixers developed was based on jet mixing. The so called micro-plume injection mixer comprised a microstructured hole array used for multiple jet injection of one fluid into a continuous flowing phase of another in a mixing chamber. The jets enter the flow in an orthogonal (90°) fashion [38, 41].

This concept has been further developed to a two-array configuration with varying position of the impinging jets implemented in one system [38, 42]. The liquid streams were fed through arrays of micro nozzles opposite to each other. These arrays can be arranged in the way that the nozzles are located face to face to each other or in an offset arrangement. Due to this, the generated jets can be brought directly to collision or flew aside each other, inducing convection.

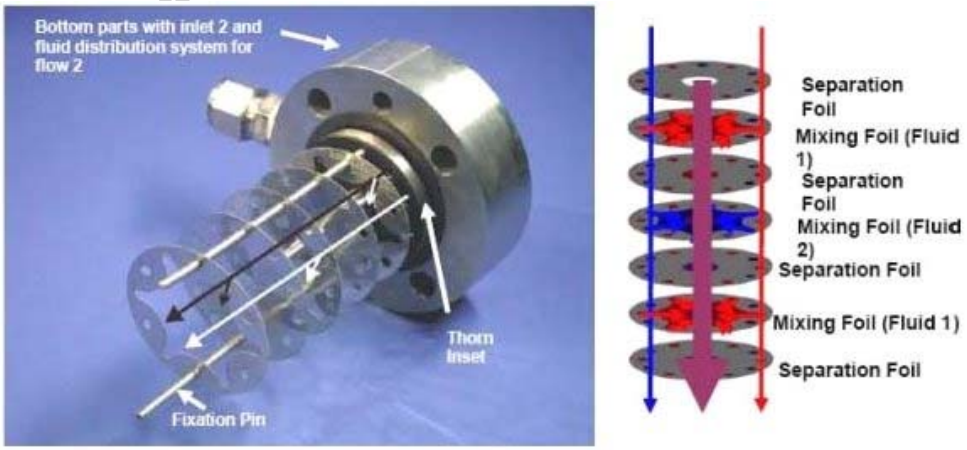


Scheme 2: Schematic of the micro-plume injection mixer design (newly drawn following [41]).



Scheme 4: Top: Schematic design of the impinging-jet array micromixer, Bottom: Face to face and offset nozzle arrays [42] (by courtesy of IOP Publishing Ltd.).

An even higher parallelized arrangement for jet mixing can be achieved when stacking many plates with multiple nozzles, as given in the microstructured mixer shown in Scheme 3. The fluids are fed through ports parallel to the mixing chamber and conducted via openings, centrally arranged in star-shaped format, to the platelet center. A breakout in the second type of platelet defines the whole flow conduit giving a cylindrical mixing chamber for the whole platelet stack [20, 38]. Due to the high number of openings and the low pressure drop by the large diameter of the breakout, very high flow rates can be achieved; the current upper limit being about 30 000 L/h liquid flow. Mixing is then performed under turbulent conditions. The concept of consecutive injection can be simply scaled out by internal numbering-up by increasing the stack of platelets, up to several hundreds. Thus, the latter suffices even bulk-chemical applications.



Scheme 3: Microstructured mixer with multiple jets from stacked star-shaped platelets and corresponding mixing principle Star Laminator [20] (by courtesy of IMM).

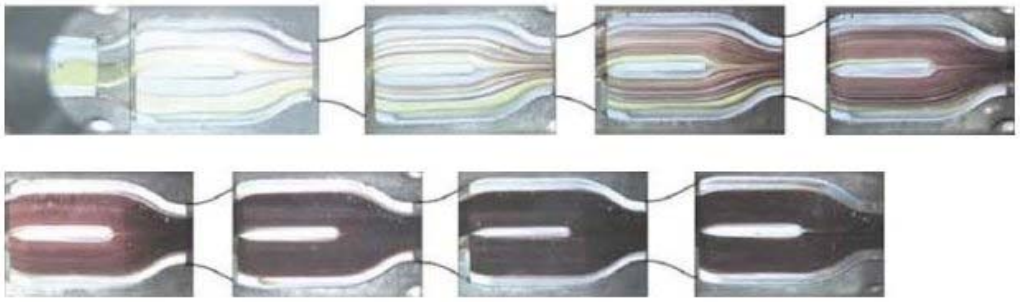
Jet mixers can particularly be applied to precipitations, since a 'wall-free' flow guidance in free space can be realized, e.g. in vertical position as falling jets. This avoids clogging which is otherwise often observed for precipitations in microchannels. When the jets are brought into collision, by having a mirror-imaged inclined flow guidance leading to a merging point, mixing takes place. In Figure 2, an Y-type impinging jet micromixer is shown [38]. The quality of mixing depends on the diameter of the jet formed at the outside of the mixer and also on the applied flow rates.

3.1.3. Split-and-Recombine Mixing

This class of mixers uses splitting and recombination of parts of the fluid flow to generate and rearrange small fluid compartments, preferably lamellae, to increase 'interfaces' for speeding up mixing. In most mixers, this is achieved by physically structuring the flow path so that it itself is split and recombined.

Figure 2: Stable y-type jets at the outlet of an impinging jet micromixer, (by courtesy of IMM).

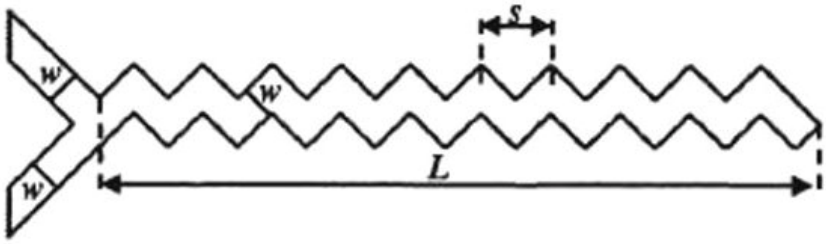
Attempts to simplify the mixer design by performing splitting and recombination of the lamellae in one larger microchannel suffer from inertia forces. With the exception of very low flow rates, these cause deformation of the lamellae so that special measures like the insertion of splitting planes are needed here to partly separate the lamellae from each other and really have split and recombination (and not to induce flow circulation). Thus, this mixing principle will be applicable for high viscous media (very low Reynolds Numbers) [38, 43].



Scheme 5: Mixing principle of split-and-recombine mixing, e.g. constantly increasing the number of lamellae along the flow path [43] (Reproduced by permission of The Royal Society of Chemistry).

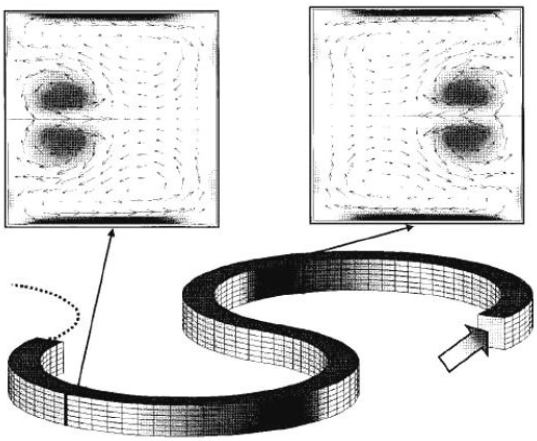
3.1.4. Recirculation-Flow Mixing In Curved Channels

The zig-zag micromixer exploits recirculation flows for mixing [38, 44]. At sufficiently high Reynolds Numbers, recirculation patterns are induced in the edges of the zig-zag channels, since the major part of the flow occurs in an alternately curved main stream which agitates the surrounding wakes. Liquid is transported in this way perpendicular to the original flow direction and improves mixing.



Scheme 6: A zig-zag microfluidic mixing element [38, 44] (by courtesy of ACS)

Recirculation flow can be also induced by using alternately curved micromixer design [38, 45]. Mixing is achieved hereby by the generation of secondary helical flows. When liquids are guided through curved channels, the fluid velocity gets it maximum towards the outer channel wall and so called Dean vortices are formed. Two counter-rotating eddies above and below the channel transverse section of the curved channel and lead to fluid transport outwards in this plane by means of centrifugal forces. By recirculation, back transport along the channel walls is induced. Thereby, interfaces are constantly created and renewed to increase mass transport. A Dean number of ~140 is the threshold for given conditions and given channel design as reported in.



Scheme 7: top: View of secondary flows taken in flow direction; bottom: sketch of a meander channel [38, 45] (by courtesy of AIChE Journal)

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Biographical Sketches

Tobias Illg was born in 1977. He started the apprenticeship for a chemical laboratory worker at the University of Heidelberg in 1994. His main working field was in the organic synthesis of chiral phosphane ligands. After his apprenticeship, he started to qualify for an advanced technical college entrance qualification in the year 1999 and finished it in 2000. He gained his first work experience as chemical laboratory worker at CHESS GmbH, Mannheim in the field of the organic synthesis of piperazine, pyrrolidine and piperidine derivatives.

After that, he started his studies at the University of Applied Science in Mannheim. Specialized in organic

chemistry, he finished his studies in 2005. His diploma thesis was about the hydrosilylation of C,N-Multiple-Bonds. The project work for this thesis was done at the Research Center Karlsruhe at the workgroup of Prof. Dinjus. Parts of Mr. Illg's thesis presented at the XXIIth International Conference on Organometallic Chemistry (Zaragoza, Spain, 27. July 2006) and at the symposium Frontiers in Catalysis (Vizegrad, Hungary 8.-10. September 2005).

Since 2006, he has been working at the Institut für Mikrotechnik Mainz GmbH (IMM) in the department "Mixing and Multiphase Contacting", investigating micro process technology applications in the named fields.

In November 2007 he started his PhD at the Institut für Mikrotechnik Mainz GmbH in cooperation with the Eindhoven University of Technology. His PhD-work is founded by the Deutsche Bundesstiftung Umwelt (DBU) and is about 'New process windows for the save and continuous synthesis of tert.-Butylperoxypivalate with micro process technology'.

Prof. Dr. Volker Hessel, born 1964, studied chemistry at Mainz University. He got the PhD level in the field of organic chemistry in 1993. The topic of his PhD thesis was structure-property relationships of special supra-molecular structures, micelles and lyotropic liquid crystals, constructed by so-called bi- or multi-polar amphiphiles with rigid core unit.

Since 1994 Prof. Dr. Hessel is an employee of the Institut für Mikrotechnik Mainz GmbH and since 1996 he became group leader for microreaction technology. In 1999 he was appointed Head of the Microreaction Technology Department, formed at that time, meanwhile named Chemical Process Technology. His fields of research comprise micro process engineering for mixing, fine chemistry, fuel processing and heterogeneous catalysis. In 2002 Prof. Dr. Hessel was appointed Vice Director R&D at IMM and in 2007 as Director R&D at IMM.

Prof. Dr. Hessel is author or co-author of 129 peer-reviewed publications (with 22 extended reviews) and 160 conference papers with regard to chemical micro process engineering, one respective chapter in Ullmann's Encyclopedia of Industrial Chemistry, 11 book chapters, and 3 books.

In July 2005, Prof. Dr. Hessel was appointed as part-time professor for the chair of "Micro Process Engineering" at Eindhoven University of Technology, TU/e. This professorship is under the umbrella of the Chemical Reactor Engineering group of Prof. Dr.ir. Jaap Schouten in the Department of Chemical Engineering and Chemistry. In October 2007, Prof. Dr. Hessel received an adjunct professorship at Technical University of Darmstadt.

Prof. Dr. Hessel received the AIChE award "Excellence in Process Development Research" in 2007.

A.o. Prof. Dr. Hessel is AIChE chair (US) "Microprocess Engineering"; elected board member of the German industrial platform IPμVT; member of the editorial boards of "Catalysis Today", "Chemical Engineering Journal", "Chemical Engineering Technology", "Recent Patents on Chemical Engineering", and "Current Organic Chemistry"; Topical chair at AIChE Spring conferences 2006+2007 and organizing committee member of the "Process Intensification and Miniaturization" symposia held at CHISA-6 and at ECCE-6 (EPIC) as well as chair of the program committees of the "Conference on Smart Synthesis and Technologies for Organic Processes (SynTOP)" and the "International Conference on Microreaction Technology 10" (IMRET-10).