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Chemical Micro Process Engineering

Fundamentals, Modelling and Reactions



**WILEY-
VCH**

WILEY-VCH Verlag GmbH & Co. KGaA

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Cover Illustration

Upper left: Production- and pilot-scale gas/gas counter-flow heat exchanger comprising microstructured channel arrays. The device (including flanges about 36 kg heavy and 54 cm long), made of stainless steel, is designed for gas throughput in the range of m³/min at 100 mbar pressure drop for a power of about 10 kW. The internals consist of a stack of microstructured plates having multi-channel arrays of a channel width of 2 mm, depth of 250 µm, and length of 240 mm. Totaling, 6685 micro channels are operated in parallel in this device. The flange-type connection allows installation in large-scale industrial plants (IMM Mainz-Hechtsheim, Germany).

Center: CFD simulation of streamlines of a liquid flow in a caterpillar micro mixer. This device utilizes the split-recombine principle leading to distributive mixing. It is seen that by multiple repetition of this principle the entanglement of the streams increases (IMM Mainz-Hechtsheim, Germany).

Lower right: Cross-flow catalyst screening device with multiple short mini-fixed beds. The fixed-bed catalyst section is fed by bifurcation-channel flow architectures that serve for flow equipartition. This device is a typical example for the class of smart chip reactors, widely employed for analytical-chemistry, kinetic studies and process/catalyst screening purposes on a lab-scale level, and is fabricated using MEMS technology based on silicon micromachining (Courtesy K. S. Jensen, MIT Cambridge, USA).

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Library of Congress Card No.: Applied for.

A catalogue record for this book is available from the British Library.

Bibliographic information published by

Die Deutsche Bibliothek

Die Deutsche Bibliothek lists this publication in the Deutsche Nationalbibliografie; detailed bibliographic data is available in the internet at <http://dnb.ddb.de>.

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Printed in the Federal Republic of Germany.

Printed on acid-free paper.

Composition Manuela Treindl, Laaber

Printing betz-druck GmbH, Darmstadt

Bookbinding Buchbinderei J. Schäffer GmbH & Co. KG, Grünstadt

ISBN 3-527-30741-9

Preface

Carrying out chemical reactions in volumes as small as possible is *a priori* not a completely new idea. In the beginnings of chemical experimentation, dating back to the age of alchemy, chemical substances like sulphuric acid or ammonia were much more valuable than gold, and very small reaction vessels were used to economize on the precious materials. When analytical chemistry was established as a second, independent discipline, the desire to make do with ever less material was very strong in order to avoid consuming large portions of the product for analysis. Establishing increasingly sensitive analytical techniques has therefore been one of the most significant driving forces in analytics research.

The beginning of the industrial age saw a substantial increase in demand for basic materials and chemicals, and the chemical industry was established to satisfy these demands for high production volumes. The tall and impressive silhouettes of modern chemical plants dominate industrial estates, visible from afar as symbols for the vast capabilities and capacities of today's chemical industry. Without this industry and its equipment of enormous proportions, our economic wealth would be quite inconceivable.

Bearing all this in mind, what is the purpose of Chemical Micro Process Technology?

Conventionally, the development of chemical manufacturing processes takes place subsequently *via* a sequence of different intermediate stages. Approaching the final process design, the reaction volume is successively increased from laboratory scale to reaction vessel dimensions suitable for production outputs of several kilotons *per annum*. This procedure, known as "scale-up", is expensive and time-consuming. During the scale-up, new and previously unencountered problems often crop up and have to be solved. It may even occur that the complete development process has to be re-initiated in order to circumvent severe obstacles. Furthermore, the developed industrial process is laid out for a specific, predefined throughput, a fact which constrains the later flexibility of production significantly.

The solution of these problems is based on a simple idea: the developed laboratory-scale process is used for manufacturing of a chemical product by parallelization of many small units. Although promising great advantages over scale-up, this procedure, denoted "numbering-up", is not trivial by far. It cannot be carried out in a simple way due to the tremendous technological effort necessary: a chemical plant with hundreds or even thousands of small-scaled vessels, stirrers, heaters, pumps,

etc. would be impractical. A new way of engineering and new technologies had to be developed to combine the advantages of lab-scale processing with the necessities associated with production-scale throughput. First steps into this direction have been taken, and despite some remaining throughput restrictions, first successes have become visible. Also, economical and ecological reasons create increasing demand for further steps in process intensification and sustainable development.

The present book is devoted to both the experimentally tested micro reactors and micro reaction systems described in current scientific literature as well as the corresponding processes. It will become apparent that many micro reactors at first sight “simply” consist of a multitude of parallel channels. However, a closer look reveals that the details of fluid dynamics or heat and mass transfer often determine their performance. For this reason, besides the description of the equipment and processes referred to above, this book contains a separate chapter on modeling and simulation of transport phenomena in micro reactors.

Using specific examples of gas-phase, gas/liquid and liquid-phase reactions, the advantages of microstructured reactors are highlighted in comparison to conventional equipment. At the same time, known problems are pointed out and some processes are listed for which micro reactors so far failed to show superior performance. Furthermore, the book is conceived as a compendium. Processes, microstructured reactors and chemical reactions are described in an integrated manner, providing in each case the relevant original citations. Equipped with the data given in this book, readers will be able to identify the most suitable reactor to successfully perform a given chemical reaction on the micro scale.

By now, Chemical Micro Process Technology has been established as an independent discipline, bringing forth over 1500 publications in the last few years, and an end is not foreseeable. The surge of scientific cognitions encouraged the authors to write this book, which should provide a deeper insight into this new and fascinating subject.

We are very grateful to those who helped this project become reality. In particular, we would like to mention K. Bouras, T. Hang, C. Mohrmann, and L. Widarto, who prepared electronic versions of many of the figures appearing in this book. We also wish to thank C. Mohrmann and L. Widarto for handling the copyright transfer formalities and T. Hang for taking pictures of some of IMM’s micro devices. A special thanks goes to B. Knabe and R. Schenk for helping us with literature retrieval. Last but not least, we are indebted to K. S. Drese and F. Schönfeld for the thorough checking of parts of our manuscript.

Mainz, November 2003

The authors

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List of Symbols and Abbreviations

$\langle \dots \rangle$	Ensemble average
A	Cross sectional area
A	Coefficient matrix
a	Chemical species
A'	Coefficient matrix
a_p, a_i	Numerical coefficients
ASE	Advanced silicon etching
a_{spec}	Internal surface area
ATR	Attenuated total reflection
$a_l^{u_i}$	Numerical coefficient
b/a	Channel aspect ratio
$b_{i,p}$	Source term in Navier-Stokes equation
$(b_i)_k$	Transformation vector
Bo	Bodenstein number
Boc	tert.-Butyloxycarbonyl
c	Species concentration
Ca	Capillary number
c_a	Concentration of species a
CAD	Computer aided design
c_b	Concentration of species b
CCD	Charge-coupled device
c_f	Fluid specific heat
CFD	Computational fluid dynamics
CGC	Constrained-geometry catalyst
c_i	Concentration at node i
c_i	Concentration of species i
$C_{i\pm 1/2}$	Flux limiter
c_p	Specific heat
CSTR	Continuous-stirred tank reactor
CVD	Chemical vapor deposition

\bar{c}	Concentration averaged over the cross section of a tube
$\tilde{c}_i^e(\omega)$	Laplace transform of the effective concentration field
\tilde{c}	Smoothed volume-fraction function
d	Bubble diameter
d	Typical length scale
D	Diffusion constant
D	Channel diameter
D	Distance
Da	Damköhler number
DBU	1,8-Diazabicyclo[5.4.0]-undec-7-ene
DCC	1,3-Dicyclohexylcarbodiimide
DCM	Dichloromethane
D_e	Dispersion coefficient
D_h	Hydraulic diameter
D_i	Species diffusion constant inside a pore
Dmab	4-[N-(1-(4,4-Dimethyl-2,6-dioxocyclohexylidene)-3-methylbutyl)-3 amino]benzyl
DMAP	4-Dimethylamino pyridine
DMF	N,N-Dimethyl formamide
DNDA	N,N'-Dialkyl-N,N'-dinitro-urea
DRIE	Deep reactive ion etching
DSMC	Direct simulation Monte Carlo method
D_e^{cur}	Dispersion coefficient in curved ducts
e	Channel depth
E	Activation energy
e	Thermal energy density
E	Magnitude of electric field strength
EDCI	3-Ethyl-1-(3-dimethylaminopropyl)-carbodiimide hydrochloride
EDDA	Ethylenediamine diacetate
EDL	Electric double layer
E_i	Electric field strength
EMA	Effective-medium approximation
EOF	Electroosmotic flow
f	Indicates the fluid phase
F	Number of molecules per unit area and time hitting a surface
F	Cost function
f_0	Maxwell distribution
FCT	Flux-corrected transport
FDM	Finite-difference method
FEM	Finite-element method
FEP	Fluorinated ethylene propylene
FFMR	Falling film micro reactor

Fmoc	9-Fluorenylmethoxycarbonyl
f	Friction factor
FTIR	Fourier transform infrared
FVM	Finite-volume method
GC	Gas chromatography
GHSV	Gas hourly space velocity
g_i	Gravity vector
GPC	Gel permeation chromatography
h	Channel height
h	Perturbation function
HPLC	High performance liquid chromatography
i, j, k, l, m, n	Summation indices
ID	Inner diameter
IR	Infrared
J_i	Thermodynamic flux
K	Dean number
k	Thermal conductivity
k	Reaction rate constant
k	Heat transfer coefficient
K	Permeability
k_0	Pre-exponential factor of Arrhenius equation
k_B	Boltzmann constant
k_L	Specific interface in gas/liquid systems
$k_1\alpha$	Mass-transfer coefficient
k_n	Time-dependent dispersion coefficient
Kn	Knudsen number
K_w	Reaction rate constant
L	Characteristic length scale of the flow domain
L	Length of a tube
l	Length of a plug
L	Length of a channel
L	Length of a channel segment
Lab-Chip	Lab-on-a-chip
LBC	Laboratory column
LC	Liquid chromatography
LC-MS	Liquid chromatography coupled with mass spectrometry
L_{hy}	Hydrodynamic entrance length
LIGA	German acronym for lithography, electroforming, moulding (Lithographie, Galvanik, Abformung)
LPCVD	Low pressure chemical vapour deposition

L_s	Slip length
L_{s0}	Reference slip length
L_{th}	Thermal entrance length
m	Molecular mass
MBC	Micro bubble column
MCR	Multi-component reaction
MD	Molecular dynamics
MS	Mass spectrometry
MSE	Micro-strip electrodes
\dot{m}	Mass flow rate
n	Coordinate normal to a wall
N	Number of molecules
n_a	Molar amount of a
n_i	Unit vector normal to an interface
n_i	Outward normal vector
n_i	Number of moles of species i
NIR	Near infrared
Nml	Standard milliliter
NMR	Nuclear magnetic resonance
NPT	Normal pressure and temperature
Nu	Nusselt number
OAOR	Oxidation and outgassing reduction
P	Grid node
p	Pressure
p	Partial pressure
P	Poincaré map
P	Channel perimeter
PDE	Partial differential equations
PDMS	Polydimethylsiloxane
Pe	Peclet number
Pe*	Modified Peclet number containing the Taylor-Aris dispersion
PLIC	Piecewise-linear interface construction
PMMA	Poly methylmethacrylate
Pr	Prandtl number
PTFE	Poly tetrafluorethylene
PVD	Physical vapour deposition
Q_f	Orthogonal subspace
\dot{q}	Heat source
\dot{q}_v	Heat source due to viscous dissipation

R	Tube radius
r	Source term due to chemical reactions
r	Distance between molecules
R	Gas constant
R	Mean radius of curvature of a channel
r	Radial coordinate of a tubular geometry
Re	Reynolds number
R_{ij}	Matrix defining how a specific reaction contributes to a change in concentration of the chemical species involved
r_j	Rate of the j th reaction
R_s	Radius of curvature along an interface
RTD	Residence time distribution
\bar{r}	Mean radius of a pore
s	Indicates the solid phase
s_{abs}	Adsorption probability at an active site of the surface
Sc	Schmidt number
s_{des}	Site-specific desorption probability
SDS	Sodium dodecyl sulphate
SEM	Scanning electron microscopy
s_i	Unit vector
S_{ij}	Surface of a computational cell
S_{kat}	Surface area of a catalyst
SLIC	Single-line interface construction
slpm	Standard liter per minute
SOI	Silicon-on-insulator
SPOS	Solid-phase organic chemistry
STP	Standard temperature and pressure
S_{Φ}	Source term
\dot{S}	Entropy generation per unit time
$(S_{\Phi})_P$	Value of a source term at node P
T	Temperature
T_c	Critical temperature
TEM	Transmission electron microscopy
THF	Tetrahydrofurane
t_i	Unit vector
TOF	Turnover frequency
TOF-MS	Time-of-flight mass spectrometry
u	Magnitude of velocity
u	Line velocity
u	Typical velocity scale
U	Mean flow velocity
u_i	Flow velocity vector

u_{\max}	Maximum velocity
u_p	Velocity at the wall
UV	Ultraviolet
u_i^{int}	Velocity of an interface
\bar{u}	Average velocity
u_i^m	Velocity field at time step m
V	Volume flow
Vi	Viscous number
V_{ij}	Volume of computational cell (i,j)
VIS	Visible
V_{kl}	Interaction potential between molecules k and l
VOF	Volume-of-Fluid
w	Channel width
W, P, E	Computational nodes
W_c	Micro channel width
w_c	Channel width
WGS	Water-gas-shift reaction
W_{ij}	Transport coefficient
x_i	Spatial coordinate vector
X_i	Thermodynamic force
$x_i^{(k)}$	Spatial coordinate i of particle k
$(\dot{Y}_s)_j$	Expansion coefficient for chemical reaction kinetics
z	Coordinate along the axis of a pore
Z_{eff}	2×2 tensor related to the slip flow on a grooved surface
Δp	Pressure drop
Δx	Grid spacing
Φ	Field quantity
Γ	Diffusivity
Λ_e	Effective thermal conductivity tensor
Λ_{ij}	Kinetic coefficients
Ψ	Electric potential
α	Heat transfer coefficient between a fluid and a solid
α	Aspect ratio of a channel
β	Dimensionless parameter representing a pseudo-homogeneous reaction
δ_{ij}	Kronecker symbol
ε	Porosity
ε	Dielectric constant

ε	Energy scale
γ	Ratio of specific heats
γ	Liquid/vapor surface tension
γ_{SL}	Solid/liquid surface tension
γ_{SV}	Solid/vapor surface tension
$\dot{\gamma}_c$	Critical shear rate
κ	Local curvature of an interface
λ	Thermal conductivity
λ	Mean free path of a gas molecules
λ_a	Eigenvalue
λ_e	Effective thermal conductivity
λ_f	Fluid thermal conductivity
λ_{nc}	Correction factor accounting for the non-circularity of a channel
λ_s	Solid thermal conductivity
$\lambda_{s,eff}$	Effective thermal conductivity
μ	Dynamic viscosity
μEDM	Micro electro discharge machining
μTAS	Micro-total-analysis system
ν	Critical exponent
ν_i	Stoichiometric coefficient of species i
θ	Contact angle
θ	Angle to the main flow direction
θ	Surface coverage
θ_a	Advancing contact angle
θ_d	Dynamic contact angle
θ_r	Receding contact angle
ρ	Density
ρ_f	Fluid density
ρ_n	Residual
σ	Correlation length for density fluctuations in a fluid
σ	Interfacial tension
σ	Liquid conductivity
σ	Range of a potential
σ_T	Thermal accommodation coefficient
σ_v	Tangential momentum accommodation coefficient
τ	Intrinsic time scale
τ_{ij}	Stress tensor
ξ_i	Computational space curvilinear coordinates
ζ	Zeta potential