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Fundamentals, Devices, Fabrication, and Applications

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Cover:

Top left: mounting a chemical reactor with microstructured elements (Dr. Schirrmeister, Chapter 7; courtesy of Uhde GmbH and Degussa AG, Germany)
Bottom right: mixing of aqueous solutions with Bromothymol Blue pH-indicator (Dr. Kockmann, Germany, Chapter 3)

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Preface

Besides the development of new devices the main goal of engineering activities is to achieve a high performance in technical systems with low effort for optimized processes and products. An incredible performance increase was achieved in communication and information technology by the miniaturization of electronic equipment down to the nanometer scale during the last decades. Moore's law of doubling the number of circuits in electronic devices in 18 months by miniaturization still holds since decades and is expected to last.

Process technology is a wide field where small processes down to the molecular scale happen in devices having a length of several meters. The scale-up of chemical production or power plants has led to high energy efficiencies and affordable consumer products. Around 1920, cryogenic air separation units produced an amount of about 1.3 t/h oxygen with 98–99% purity. 30 years later, the largest air separation units delivered about 5.2 t/h oxygen with 99% purity. Nowadays, the largest air separation units are supplying large customers with about 65 t/h oxygen with 99.5% purity and higher. As the throughput increases, the specific energy consumption decreases from about 1.5 kW/kg oxygen to about 0.4 kW/kg oxygen. Besides the development of large units, the consumer specific supply was also addressed by small and adjusted plants for flexible production satisfying the costumer's demand. Additionally, some branches of the chemical industry are not subjected to the economy of scale like the pharmaceutical industry or fine chemicals; flexibility as well as the product price and quality are the important factors.

The combination of process engineering and micro system engineering with the design, fabrication, and integration of functional microstructures is one of the most promising research and development areas of the last two decades. This is reflected in the publishing of scientific journals like "Sensors and Actuators" (since 1981) as well as in the growing field of international conferences like μTAS (Micro Total Analysis Systems, since 1994), the IMRET (International Conference on Micro Reaction Technology by AIChE and DECHEMA, since 1997), or the ICMM (International Conference on Micro and Mini Channels by ASME, since 2003). This can also be seen in the growing industrial activities using microstructured equipment in process development and production of chemicals. Some activities can be summarized under the concept of process intensification, such as compact heat exchangers or structured packing in separation columns for intensified heat and mass transfer. With characteristic lengths of the devices in the size of boundary layers, the transfer processes can be en-

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hanced and controlled in the desired way. Other activities include modular platforms and entire chemical plants consisting of several microstructure elements and devices, mainly for laboratory and process development.

This book on micro process engineering is divided into four sections: fundamentals (Chapter 1 to 6), the design and system integration (Chapter 7 to 9), fabrication technologies and materials (Chapter 10 to 12), and, finally, the applications of microstructured devices and systems (Chapter 13 to 16). Each chapter has review character and stands on its own, but is also integrated into the whole book. A common nomenclature and index will help the orientation of the reader. In Chapter 1 to 6 the fundamentals and tools of process engineering are presented with single-phase and multiphase fluid flow, heat and mass transfer as well as the treatment of chemical reactions following the concept of unit operations. The equipment and process design is organized by project management methods and assisted by modeling and simulation as well as the integration of sensors and analytical equipment, described in Chapter 7, 8, and 9. The broad fabrication variety of microstructured devices for micro process engineering is illustrated in Chapter 10, 11, and 12 grouped according the materials metal, polymers, silicon, glass, and ceramics. Some typical examples of microstructured devices illustrate the various fabrication methods. Even more examples are given in Chapter 13 to 15 with industrial applications in Europe, Japan and the US. Last but not least Chapter 16 emphasizes the application of microstructured devices in education and laboratory research work. This gives students a deeper insight into the complex behavior of chemical plants and will lead to a more sophisticated view of continuous flow processing in education, laboratory experiments, and chemical synthesis.

The aim of this book is the comprehensive description of actual knowledge and competence for microfluidic and chemical process fundamentals, design rules, related fabrication technology, as well as an overview of actual and future applications. This work is located at the boundary of at least two different disciplines, trying to collect and unify some of the special knowledge from different areas, driven by the hope that innovation happens at the interfaces between the disciplines. From this, the team of authors of various engineers, physicists and chemists, from universities, research institutes, and industry in different countries contributes an embracing part of detailed know-how about processes in and applications of microstructures. I hope that this knowledge will help to look out of the box to other related areas of chemical engineering, micro system engineering and to other engineering, physical, chemical, or biological areas.

Finally, I want to thank all the contributors for their enduring work, besides their actual work and activities. I hope that this enthusiasm can be read throughout the book, will spread further on to the readers and will help to enlarge the knowledge and activities on this new and gap-filling area of micro process engineering.

Norbert Kockmann
Volume Editor
November 2005

Foreword

We hereby present the fifth volume of *Advanced Micro & Nanosystems* (*AMN*), entitled *Micro Process Engineering*.

Usually, when engineering devices get smaller, we expect higher speeds, more accuracy, or less power consumption, but typically we do not associate smaller devices to successfully compete with larger ones when it comes to material throughput. Not so in micro process engineering. This research area has quietly grown in the flanks, and promises to become one of the most profitable areas in microtechnology. Why is this so? It turns out that micro process engineering targets the more efficient manufacture of chemical substances, no less than miniaturized chemical factories that match the throughput of their macroscopic counterparts.

The volume editor, Dr. Norbert Kockmann, has assembled a notable international authors hip to bring to us the state of the art in this very exciting application area. At the microscale, many physical and chemical effects have to be reevaluated as they apply to chemical engineering manufacturing processes, and in this volume six chapters guide us through the most important fundamental concepts. The revised theory implies the need for new design methods, and so three chapters consider simulation, modelling, and system design. Device fabrication sets specific challenges, for all resulting production surfaces must be chemically and thermally resistant, and must target high throughput of liquids and gases. Finally, because micro process engineering is driven by its exciting applications, four chapters cover the most important topics from a completely international perspective.

We are happy to report here that the decision to produce topical volumes such as *CMOS-MEMS* or *Microengineering of Metals and Ceramics* is finding tremendous acceptance with our readers and hence we will continue to plan further relevant topics from either an application area or a specific manufacturing technology.

Looking ahead, we hope to welcome you back, dear reader, to the upcoming sixth member of the *AMN* series, in which we take a close look at the fascinating field of LIGA and its application.

Oliver Brand, Gary K. Fedder, Christofer Hierold, Jan G. Korvink, and Osamu Tabata
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Nomenclature

List of main parameters

If not indicated in the text.

Name	Unit	Description
A	m^2	area or cross section
a	$\frac{m^2}{s}$	temperature conductivity $a = \frac{\lambda}{\rho c_p}$
b	m	geometrical factor, channel width
C	–	constant
C_i	–	ratio of heat capacity fluxes
c	$\frac{m}{s}$	absolute velocity
c_i	–	concentration of component i
c_p	$\frac{kJ}{kg\ K}$	isobaric specific heat capacity
c_v	$\frac{kJ}{kg\ K}$	isochoric specific heat capacity
D	$\frac{m^2}{s}$	diffusion coefficient
D	m	diameter
d_h	m	hydraulic diameter $= \frac{4A}{U}$
E	J	energy
e	$\frac{J}{kg}$	specific energy
F	N	force
f	–	probability distribution function
G	$\frac{kg}{m^2 s}$	mass velocity (US literature)
g	m/s^2	gravity constant

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g	m	temperature jump coefficient
H	J	enthalpy
H_i	Pa	Henry coefficient
h	$\frac{J}{kg}$	specific enthalpy
h_{LV}	$\frac{J}{kg}$	latent heat of vaporization
h	$\frac{W}{m^2K}$	heat transfer coefficient (US literature)
h	m	geometrical factor, height
I	A	electrical current
J	J/s	general energy current
K	–	general coefficient
k	$\frac{W}{m^2K}$	overall heat transfer coefficient
k	$\frac{W}{mK}$	thermal conductivity (US literature)
k	*)	reaction rate constant, *) unit depends on reaction order
k_M	$\frac{kg}{sPa}$	membrane conductivity
k	$\frac{J}{K}$	Boltzmann constant $\left(1.380662 \times 10^{-23} \frac{J}{K}\right)$
L	m	length
L_{ij}	–	general transport coefficient
L_p	mol/s	rate of production
l	m	length, characteristic
M	$\frac{kg}{kmol}$	molar mass
M^*	kg	mass of an atom or molecule
m	kg	mass
m	–	reaction order
$\dot{m}$	$\frac{kg}{s}$	mass flow
N	–	number
N_i	–	ratio of transferred heat to heat capacity, number of transfer units
n	1/s	rotation speed
n	mol	amount of substance
$\dot{n}$	mol/s	molar flow rate
P	W	power
p	bar, $Pa = \frac{N}{m^2}$	pressure

p_i	bar, $\text{Pa} = \frac{\text{N}}{\text{m}^2}$	partial pressure
Q	$\text{J} = \text{Nm} = \frac{\text{kg m}^2}{\text{s}^2}$	heat
$\dot{Q}$	$\text{W} = \frac{\text{J}}{\text{s}}$	heat flux
q	$\frac{\text{J}}{\text{kg}}$	specific heat
$\dot{q}$	$\frac{\text{W}}{\text{m}^2}$	specific heat flux
R	m	radius
R	$\frac{\text{J}}{\text{kg K}}$	individual gas constant $= \frac{R_m}{M}$
R_i	$\frac{\text{mol}}{\text{m}^3\text{s}}$	transformation rate
R_m	$\frac{\text{J}}{\text{kmol K}}$	universal gas constant $\left(8.314 \frac{\text{kJ}}{\text{kmol K}}\right)$
R_{el}	$\frac{\text{V}}{\text{A}}$	electrical resistance
r	m	location coordinate, radius
r	1/s	reaction rate
S	$\frac{\text{J}}{\text{K}}$	entropy
S'	–	heat production potential
s	$\frac{\text{J}}{\text{kg K}}$	specific entropy
T	K	temperature (Kelvin)
t	s	time
U	J	inner energy
U	m	circumference, perimeter
U	V	electric voltage
u	$\frac{\text{J}}{\text{kg}}$	specific inner energy
u	$\frac{\text{m}}{\text{s}}$	velocity in x-direction
V	m^3	volume
$\dot{V}$	m^3/s	volume flow rate
v	$\frac{\text{m}}{\text{s}}$	velocity in y-direction
$\vec{v}$	$\frac{\text{m}}{\text{s}}$	vector of velocity
W	J	work
W_{diss}	J	dissipation work

w	$\frac{\text{m}}{\text{s}}$	velocity in z-direction
w	$\frac{\text{J}}{\text{kg}}$	specific work
X	–	general transport variable
X	–	conversion
$\vec{x}$	m	position vector
x	m	cartesian coordinate
x	–	vapor quality or void fraction
x_i	–	liquid concentration of component i
y	m	cartesian coordinate
y_i	–	vapor concentration of component i
z	m	cartesian coordinate, main flow direction in a channel

Greek letters

α	$\frac{\text{W}}{\text{m}^2\text{K}}$	heat transfer coefficient
α	–	mixing quality
β	$\frac{\text{m}}{\text{s}}$	mass transfer coefficient
γ	–	Arrhenius number
γ	$\frac{\text{kg}}{\text{m}^2\text{s}}$	momentum transfer coefficient
γ	$\frac{\text{N}}{\text{m}}$	interfacial tension
γ_i	–	relative amount of substance, mole fraction
δ	m	boundary layer thickness
ε	$\frac{\text{m}^2}{\text{s}^3}$	specific energy dissipation
ε	–	porosity
ζ	–	friction factor of channel fitting or installation
ζ	m	slip length of rarefied gas flow
η	$\frac{\text{kg}}{\text{m s}} = \frac{\text{Ns}}{\text{m}^2}$	dynamic viscosity
Θ	–	dimensionless temperature
θ	deg	contact angle
θ	–	heat exchanger efficiency
κ	–	isentropic exponent
Λ	m	mean free path length
λ	$\frac{\text{W}}{\text{mK}}$	heat conductivity

λ_R	–	channel friction factor
μ	Pa s	dynamic viscosity (US literature)
ν	$\frac{m^2}{s}$	kinematic viscosity $= \frac{\eta}{\rho}$
ν_i	–	stoichiometric coefficient
π_i	Pa	mole fraction acc. Raoult's law
ρ	$\frac{kg}{m^3}$	density $= \frac{m}{V}$
σ	m	diameter of an atom or molecule
σ	$\frac{N}{m}$	surface tension
$\dot{\sigma}$	$\frac{J}{Ks}$	dissipation function or local entropy production
τ	s	space time
τ	$\frac{N}{m^2}$	shear stress
φ	–	probability
χ	–	dispersion
ω	1/s	angular velocity

Sub- and superscripts

$\vec{x}$	vector description of x
$\bar{x}$	mean value of x
x^*	dimensionless form of x

Dimensionless numbers of fluid mechanics, heat and mass transfer

Name	Description
$\mathrm{Bi} = \frac{\alpha l}{\lambda}$	Biot number ($\lambda = \lambda_{\mathrm{solid}}$)
$\mathrm{Bo} = \frac{wl}{D_{\mathrm{ax}}}$	Bodenstein number
$\mathrm{Bo} = \frac{q''}{G h_{fg}}$	boiling number (US literature)
$\mathrm{Bo} = \frac{\mathrm{We}}{\mathrm{Fr}} = \frac{d^2 g \rho}{\sigma}$	Bond number (US literature)
$\mathrm{Ca} = \frac{\eta w}{\sigma}$	capillary number

$\mathrm{Co} = \left(\frac{1-x}{x}\right)^{0.8}\left(\frac{\rho_\mathrm{V}}{\rho_\mathrm{L}}\right)^{0.5}$	convection number
$\mathrm{DaI} = \frac{t_r}{\tau_\mathrm{res}}$	1st Damköhler number
$\mathrm{DaII} = \frac{k_s}{k_D} = \frac{t_r}{t_D}$	2nd Damköhler number
$\mathrm{Dn} = \mathrm{Re}\left(\frac{D}{R_c}\right)^{1/2}$	Dean number
$\mathrm{Ec} = \frac{w^2}{c_p \Delta T}$	Eckert number
$\mathrm{Eu} = \frac{\Delta p}{\rho w^2}$	Euler number
$\mathrm{Fo} = \frac{at}{l^2}$	Fourier number
$\mathrm{Fr} = \frac{w^2}{dg}$	Froude number
$\mathrm{Gr} = \frac{g_z \beta_p s^3 \Delta T}{\nu^2}$	Grashoff number
$\mathrm{Kn} = \frac{\Lambda}{L}$	Knudsen number
$\mathrm{Le} = \frac{a}{D}$	Lewis number
$\mathrm{Ma} = \frac{w}{c}$	Mach number
$\mathrm{Ne} = \frac{P}{\rho n^3 d^5}$	Newton number
$\mathrm{Nu} = \frac{\alpha l}{\lambda}$	Nußelt number ($\lambda = \lambda_\mathrm{Fluid}$)
$\mathrm{Pe} = \mathrm{RePr} = \frac{wl}{a}$	Péclet number
$\mathrm{Pr} = \frac{\nu}{a}$	Prandtl number
$\mathrm{Ra} = \mathrm{GrPr}$	Rayleigh number
$\mathrm{Re} = \frac{wl}{\nu}$	Reynolds number
$\mathrm{Sc} = \mathrm{LePr} = \frac{\nu}{D}$	Schmidt number

$\mathrm{Sh} = \frac{\beta l}{D}$	Sherwood number
$\mathrm{We} = \frac{w^2 d\rho}{\sigma}$ $= \frac{n^2 d^3 \rho}{\sigma}$	Weber number

Mathematical operators

D	substantial differential
d	general differential
∂	partial differential
Δ	delta, divergence, difference
$\nabla = \vec{i}\,\frac{\partial}{\partial x} + \vec{j}\,\frac{\partial}{\partial y} + \vec{k}\,\frac{\partial}{\partial z}$	Nabla operator
grad $\varphi = \nabla\varphi$	gradient of φ
div $\vec{v} = \nabla\,\vec{v}$	divergence of $\vec{v}$
rot $\vec{v} = \nabla \times \vec{v}$	rotation of $\vec{v}$
$\Delta\varphi = \frac{\partial^2\varphi}{\partial x^2} + \frac{\partial^2\varphi}{\partial y^2} + \frac{\partial^2\varphi}{\partial z^2}$	Laplace operator

Main conferences and their abbreviation

IMRET 1: W. Ehrfeld (Ed.), *Microreaction Technology, Proc. of the 1st Int. Conf. on Microreaction Technology 1997*, Springer, Berlin, **1998**.

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Process Engineering Methods and Microsystem Technology

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Abstract
The fundamentals of chemical engineering are presented with the aim of applications in microsystem technology, microfluidics, and transport processes in microstructures. After a general overview about both disciplines and common areas the concept of unit operations is briefly introduced. The balance equations are derived from statistical mechanics and applied to other relevant systems of process engineering together with the kinetic description of main transfer processes. Engineering tools like dimensional analysis, order of magnitude estimations, or lumped element modeling are explained, which are very helpful for dealing with complex nonlinear systems. Concluding this chapter, the benefits and limits of miniaturization of various unit operations and typical issues are explained that might serve as a plentiful source for the future development.

Keywords
Unit operations, balance equations, transport equations, engineering modeling, scaling process

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1.1 Introduction

Process technology and microsystem technology are both interdisciplinary engineering and natural science branches connecting physics, chemistry, biology, engineering arts, and management techniques to an enabling toolbox for various applications. Process engineering embraces orientating calculations for process and equipment design under general orientation, and system-orientated, cross-linked thinking. Process engineers are working in various areas ranging from the food industry through biotechnology to pharmaceutical products, from analytical and laboratory equipment through energy conversion to industrial chemistry for the production of millions of tons of chemicals [1, Chapter 1]. Chemical process engineering covers not only the design and implementation of chemical production and analytical processes but also deals with the equipment design, the appropriate materials, the fabrication, and operation of various chemical production processes. The aims of process technology are the economical and safe production of the desired products with the intended form and composition.

Microsystem technology, coming from information technology and miniaturization of data-processing devices, has now entered many fields in our daily life. Silicon chips and sensors can be found in cars, washing machines or smart cards with various functions. Besides the data-processing function, microsystems have taken over other tasks like sensing and analyzing, actuating or controlling larger systems. Microsystem engineering comprises besides engineering skills like design, simulation, or material knowledge also a deep physical and chemical knowledge for the fabrication and functional design issues. Also medical and biological skills are useful for the growing application fields for analysis, diagnostics, and therapeutics. A good overview about the state-of-the-art in microsystem technology is given in [2]. For the control and manipulation of still smaller systems, microsystem technology is a major link to nanotechnology [3, 4].

Figure 1.1 gives an impression of the wide field and complexity of both disciplines, but also illustrates the multiple interfaces and common fields. The fruitful ideas from both sides may inspire the further development in both disciplines and result in an enlargement of possibilities and applications for the innovation across the borderlines.

Chemistry in *miniaturized equipment* is an emerging discipline coming together from microsystem technology and from chemical engineering, but also an established discipline of chemical analytics. Starting at the end of the nineteenth century a group of researchers at the University of Delft around Behrens [4a] and at the Technical University of Graz around Prof. Emich and Prof. Pregl developed the chemical analysis of very small amounts of reagents. In 1900 Prof. Behrens wrote his book "Mikrochemische Technik" [4a] about micro chemical techniques. In 1911 Prof. Friedrich Emich published the textbook "Lehrbuch der Mikrochemie" [5] and Prof. Fritz Pregl was rewarded in 1923 by the Nobel price for his fundamental work in microchemical analysis. In the middle of the last century in nuclear science small structures were developed for the separation of isotopes, see [6]. From this work, among others, the LIGA technology emerges at German research institutes.

Dealing with very small geometrical structures is also a well-known area in process engineering. The adsorption technology and chemical reactions at catalytic surfaces are based on the flow and adhesion processes in nanoscale pores [7, Chapter 4]. Transformations and transfer processes on the molecular scale are called "micro processes" in contrast to a "macro process" where convection

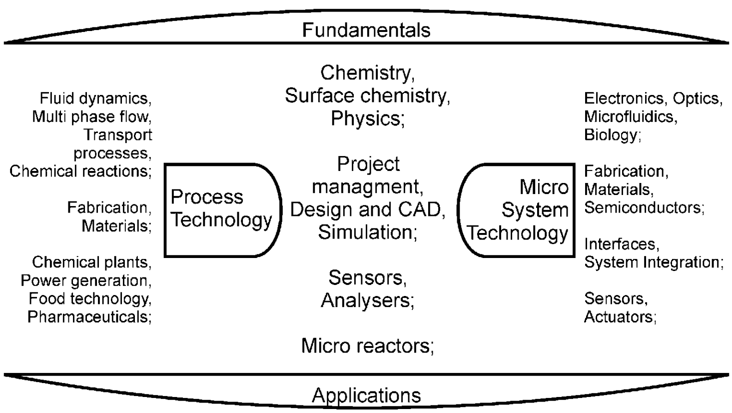

Fig. 1.1 Disciplines of process engineering and microsystem technology, differing and common overlapping areas (middle column). The lists are not complete and the future will certainly bring new applications and new common fields and applications.

plays the major role. Some typical length scales for process technology, chemistry and microtechnology are given in Fig. 1.2.

Figure 1.2 illustrates the different wording in process engineering, microsystem technology, and nanotechnology, especially the different meaning of "micro". The micropores in adsorption media are one characteristic example on the nanometer scale. Microstructured equipment has internal characteristic dimensions like channel diameter or gap height within the micrometer range. A clear definition of "micro" does not exist, but it is not necessarily required for all applications and areas.

In the process industry, there are several applications of structures with typical dimensions below 1 mm, like compact plate and fin heat exchangers or structured packings in separation columns for enhanced heat and mass transfer. This is often summarized under the key word of process intensification. However, the miniaturization of conventional technology is limited by two major restrictions: the fabrication possibilities for the small structures at reasonable costs and the increased fouling probability, the high danger of blocking, and total failure of these structures. The first restriction has been widened with the enhanced fabrication possibilities, but the risk of fouling and blocking is still there and should not be underestimated.

The elementary setup of microstructured and conventional equipment is similar and displayed in Fig. 1.3. Process plants consist of process units, which themselves are made of equipment like heat exchangers or vessels with internal structures. The basic geometrical elements of the internal structures in conven-

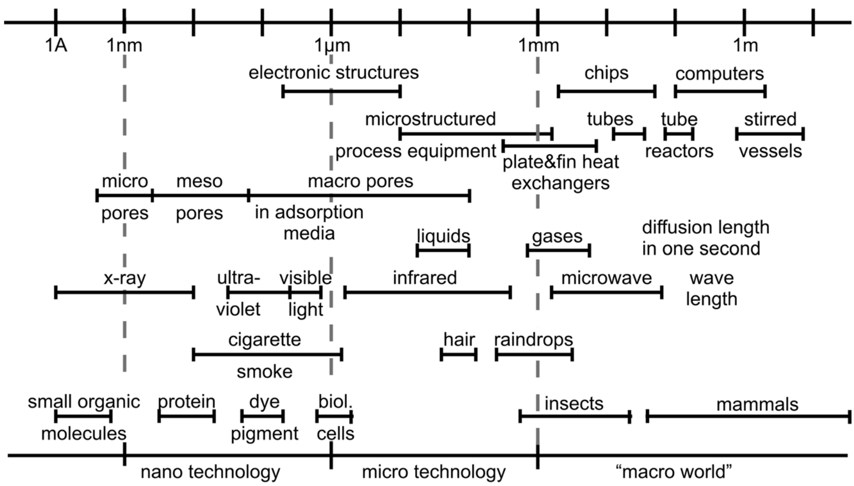

Fig. 1.2 Characteristic length of important processes and equipment in chemical engineering and microsystem technology. The top and bottom line indicate also the different wording of micro processes in the two disciplines, adopted from [8].

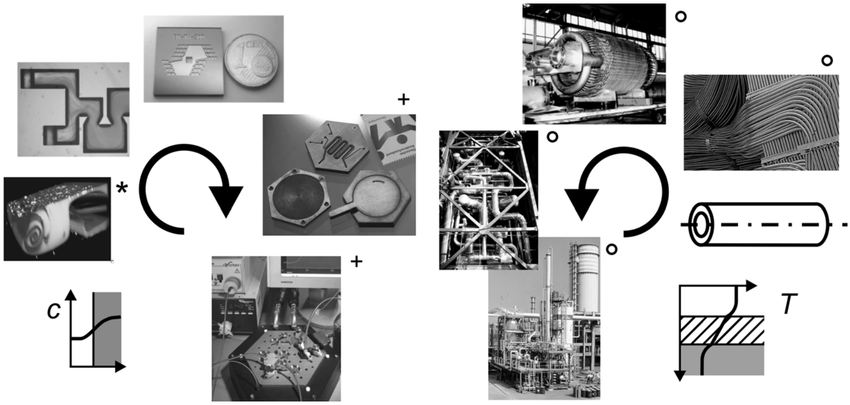

Fig. 1.3 Setup of microstructured and conventional equipment from single microstructures through combined elements to an entire plant. The principle of the active area can be applied in both cases (Sources: * from [9]; + courtesy of FhG-ICT, Pfinztal, Germany; o courtesy of Linde AG, Wiesbaden, Germany).

tional technology are the tube, the plate, and the film, on or in which the transport processes and transformations happen. The layout of process equipment and process steps follow this scheme from small elementary active areas ("micro process") over the process space of the device ("macro process") to the balancing of the complete process.

The parallel arrangement of microstructured channels or elements is called internal numbering-up, which is the most frequent way to increase the throughput of an apparatus. The parallelization of microstructured devices is called external numbering-up, applied to bypass the flow distribution problems within the equipment. A relatively new concept is the equal-up concept, the parallelization of similar effects [8]. The numbering-up and equal-up concepts facilitate the scale-up process from laboratory equipment to production equipment, but still have their own problems of flow distribution in manifolds, see Chapter 8.

1.2 Unit Operations and Beyond

The consecutive groups or steps in a process plant can frankly be named for many cases as

- pretreatment or conditioning of the incoming substances,
- transformation of the reagents in chemical, physical, or biological processes,
- separation of the received components, and
- purification and conditioning of the products, see Fig. 1.4.

The physical and chemical processes in the various steps may be the same or similar, like heat transfer or extraction. They are called *unit operations* that are playing a major role in the research and development of process engineering. The unit operations can be combined and connected in different forms. The concept of unit operation combines a macro process with the apparatus to a process unit. It allows us to treat all micro processes within the process space in the same manner and to derive scientifically based design rules and calculation instructions. For an entire process plant the unit operations are combined and switched in a proper way and integrated for efficient material and energy use. Besides the energy and mass flow integration the appropriate process control and automation determines the economical performance and safety of the plant. This gives a very complex picture of a chemical or process technology plant, which is illustrated in Fig. 1.4. For a proper design and operation of a plant, many disciplines have to work closely together.

The unit operations can be categorized into three major groups according the employed physical effects and major driving forces for combination or separation of substances: the mechanical, the electromagnetic, and the thermal unit operations (molecular driving forces) see Table 1.1. This list does not claim to be complete, especially the separation processes from analytics are only shown schematically. Probably in the next years further operations will be developed enabled by enhanced fabrication and integration possibilities. In adsorption of species or membrane separation, chemical processes may also be involved for mass-transfer processes in microstructures, see [11, Chapter 3]. The consequent treatment of unit operations allows the methodological design with help of the following principles. The *principle of continuity* of substances, phases, energy and momentum includes the preference of continuous processes opposite to batch processes. The *principle of balancing* of the relevant transport processes gives the energy, momentum, and mass fluxes in differential or black-box form. The *principle of scaling* and *similarity* of processes gives a calculation tool for transferring experimental, analytical, and numerical results to processes on different scales with the help of dimensionless numbers and groups.

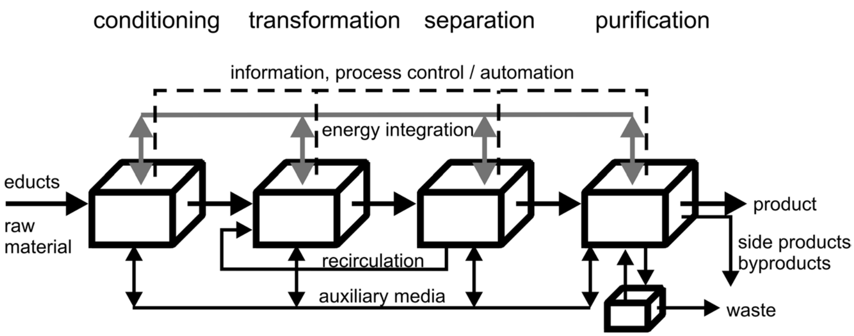

Fig. 1.4 Main process steps in a chemical production plant with pretreatment, conversion, separation, and purification of the products, adapted from [10]. The system integration includes the energy management, auxiliary media as well as information for the process control and automation.