



**Small is the next  
big thing**

# Micro Process Engineering – An Interdisciplinary Approach

Micro Process Engineering, the science of enhanced transport process in microstructured devices

## Basic Understandings

Scaling down to micrometer dimensions changes not only the length or volume of a device, but also influences the performance of unit operations.

## **Relevant Books**

Microflows and Nanoflows - Fundamentals and Simulation by George Karniadakis, Ali Beskok, Narayan Aluru; Springer, 2005.

MICRODROPS AND DIGITAL MICROFLUIDICS by Jean Berthier, William Andrew Inc. 2008.

Transport Phenomena in Micro Process Engineering, by Norbert Kockmann, Springer, 2006.

# Topics to be covered

Overview – length and time scales

Manufacturing practices – top down and bottom up

Transport in microchannels – heat pipes

Continuum, scaling

Balance and transport equations, convective flow and heat transfer in microchannels

Microfluidic networks

Electrowetting and digital microfluidics

# Differences between fluid mechanics at microscales and in the macrodomain :

- *Non-continuum effects*: Both viscosity and the no-slip condition are concepts developed under the framework of continuum. Slip / Contact line motion?
- *Surface-dominated effects*: Friction, electrostatic forces, viscous effects
- *Low Reynolds number effects*:
- *Multiscale and multiphysics effects*.

Microdevices tend to behave differently from the objects we are used to handling in our daily life.

The **inertial forces** tend to be quite small, and **surface effects** tend to dominate the behavior of these small systems.

Friction, electrostatic forces, and viscous effects due to the surrounding air or liquid become increasingly important as the devices become smaller.

Example: Forces present in a thin film of liquid

## Navier Stokes Equation

$$\rho \left( \frac{\partial v_z}{\partial t} + v_x \frac{\partial v_z}{\partial x} + v_y \frac{\partial v_z}{\partial y} + v_z \frac{\partial v_z}{\partial z} \right) =$$
$$- \frac{\partial P}{\partial z} + \mu \left[ \frac{\partial^2 v_z}{\partial x^2} + \frac{\partial^2 v_z}{\partial y^2} + \frac{\partial^2 v_z}{\partial z^2} \right] + \rho g_z$$

$$\rho \left( \frac{\partial v_z}{\partial t} + v_x \frac{\partial v_z}{\partial x} + v_y \frac{\partial v_z}{\partial y} + v_z \frac{\partial v_z}{\partial z} \right) = - \frac{\partial P}{\partial z} + \mu \left[ \frac{\partial^2 v_z}{\partial x^2} + \frac{\partial^2 v_z}{\partial y^2} + \frac{\partial^2 v_z}{\partial z^2} \right] + \rho g_z$$

the z - momentum equation

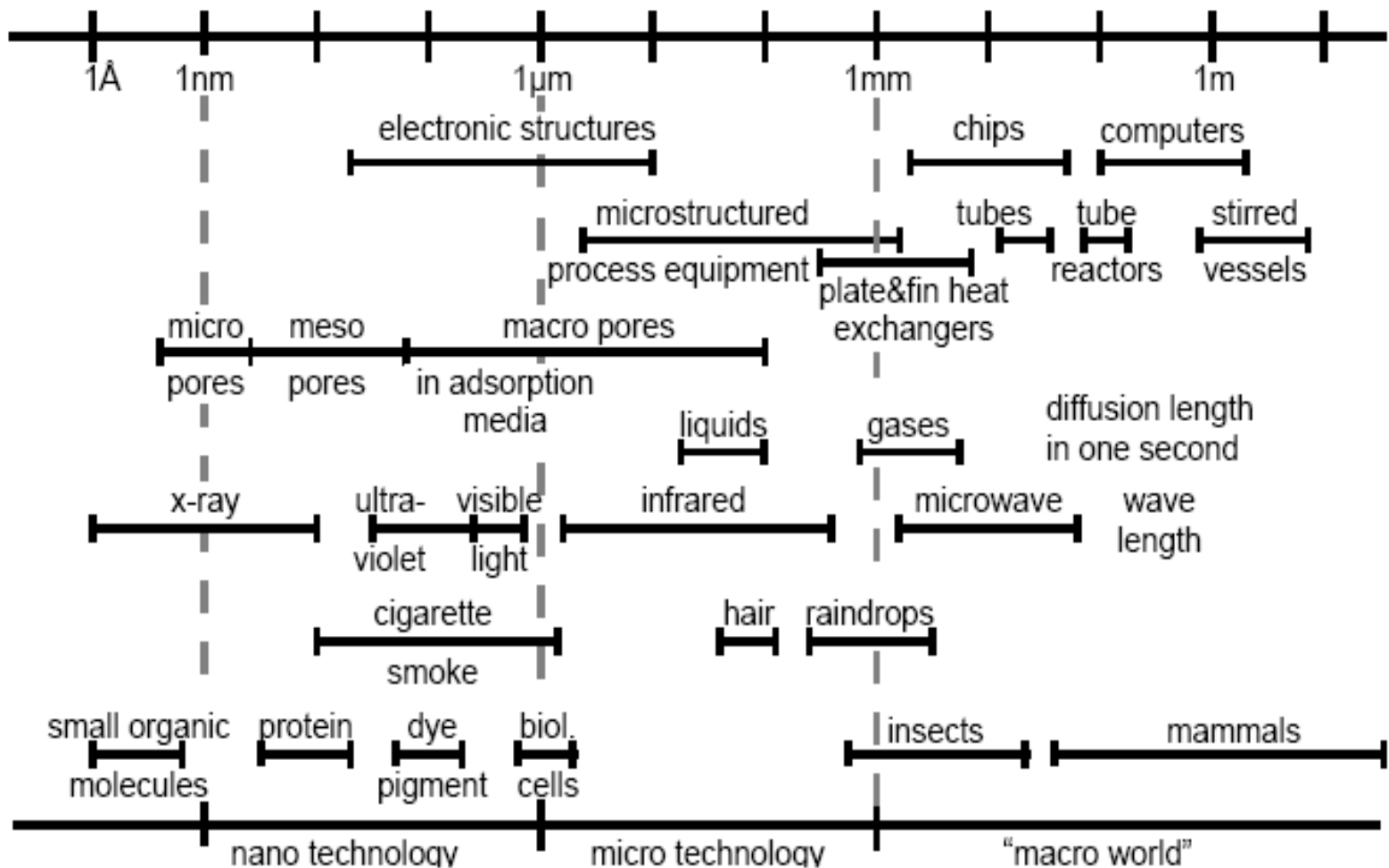
$$v^* \frac{\partial w^*}{\partial y^*} + w^* \frac{\partial w^*}{\partial z^*} = - \frac{\partial p}{\partial z^*} + \frac{\partial}{\partial y^*} \left[ \eta^* \frac{\partial w^*}{\partial y^*} \right] + \frac{1}{\text{Re}^2} \frac{\partial}{\partial z^*} \left[ \eta^* \frac{\partial w^*}{\partial z^*} \right],$$



Micromechanical systems started to enter technical systems in the 80s and 90s, enabling fluidic systems to be developed.

Starting from data processing, microsystems have now integrated mechanical, optical, fluid mechanical, and chemical functions for tasks like sensing and analyzing, controlling larger systems, or producing suitable goods and growing application fields for therapeutics and diagnostics.

Due to the reduced length scale of microstructured process equipment, the transfer lengths are short and precisely defined, and the areas are small, but high surface-to-volume ratios and tiny volumes dominate everything.



Characteristics lengths of important processes and equipment in chemical engineering and microsystems technology

Overview of miniaturization effects and beneficial phenomena e.g. in microchannels with characteristic dimensions from 10 to 1000  $\mu\text{m}$ .

Process intensification in chemical engineering benefits from the miniaturization of channels and ducts within devices, where the characteristic lengths reach into the scale of boundary layers.

The higher transport rates can be used for many different purposes such as rapid mixing, temperature-sensitive reactions, temperature homogenization, nanoparticle precipitation.

## Square-Cube Law

When an object undergoes a proportional increase in size, its new volume is proportional to the cube of the multiplier and its new surface area is proportional to the square of the multiplier.

### Example from Biology

If an animal were scaled up by a considerable amount, its relative muscular strength would be severely reduced, since the **cross section of its muscles would increase** by the ***square*** of the scaling factor while its **mass would increase** by the ***cube*** of the scaling factor.

Cardiovascular and respiratory functions would be severely burdened.

In general, properties (p) that are a function of the area of interaction (A) decrease more slowly than properties that depend on the volume (V ), as expressed by the “*square-cube*” law:

$$\frac{p_1(A)}{p_2(V)} \propto \frac{L^2}{L^3} \propto \frac{1}{L}$$

L is the characteristic dimension of the microdevice. This has interesting application in microdevices.

A typical order of magnitude of the ratio is  $10^6 \text{ m}^2/\text{m}^3$ .

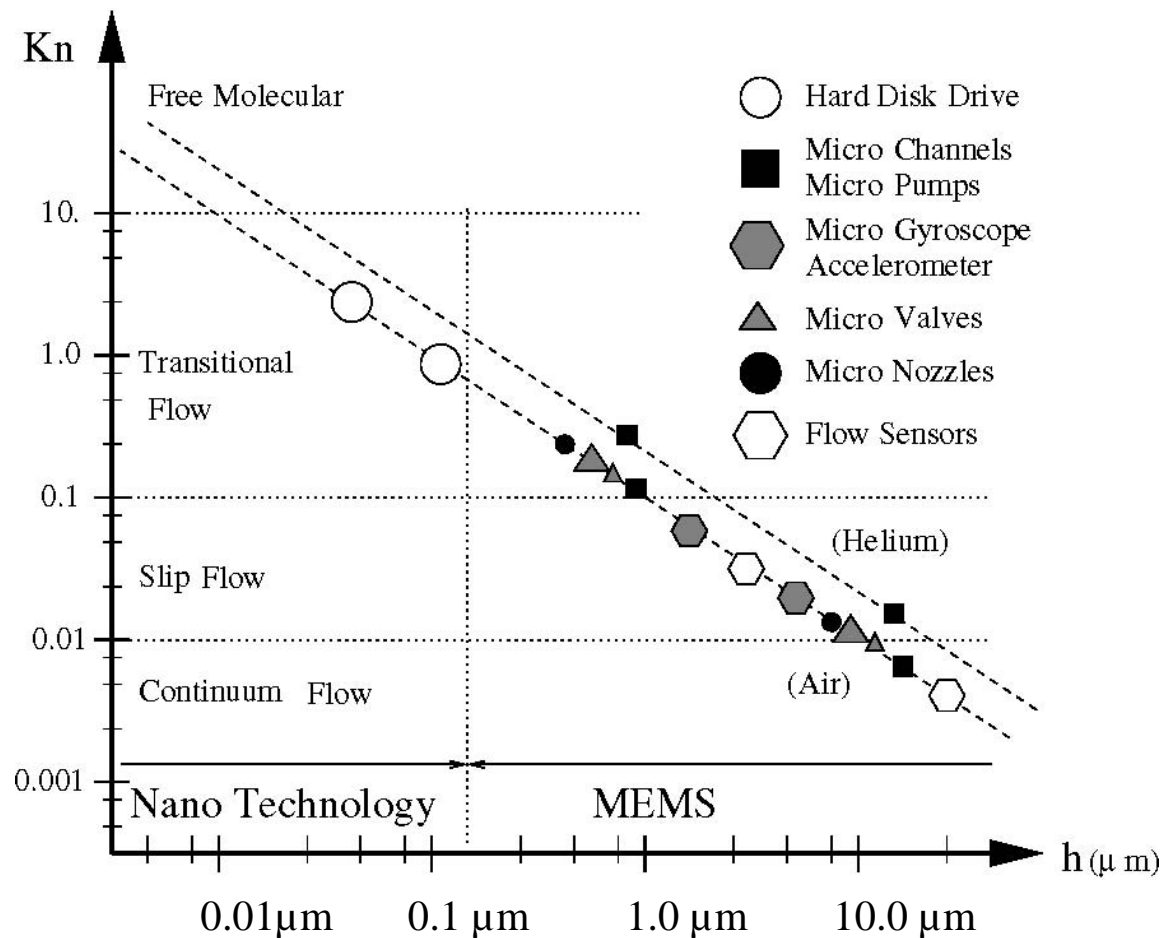
Surface tension effects are dominant at these scales, and micropumps and microvalves have been fabricated taking advantage of this principle

Early applications in computer components – the Winchester-type hard disk drive mechanism, where the read/write head floats 50 nm above the surface of the spinning platter. Smaller the gap - greater the recording capacity.

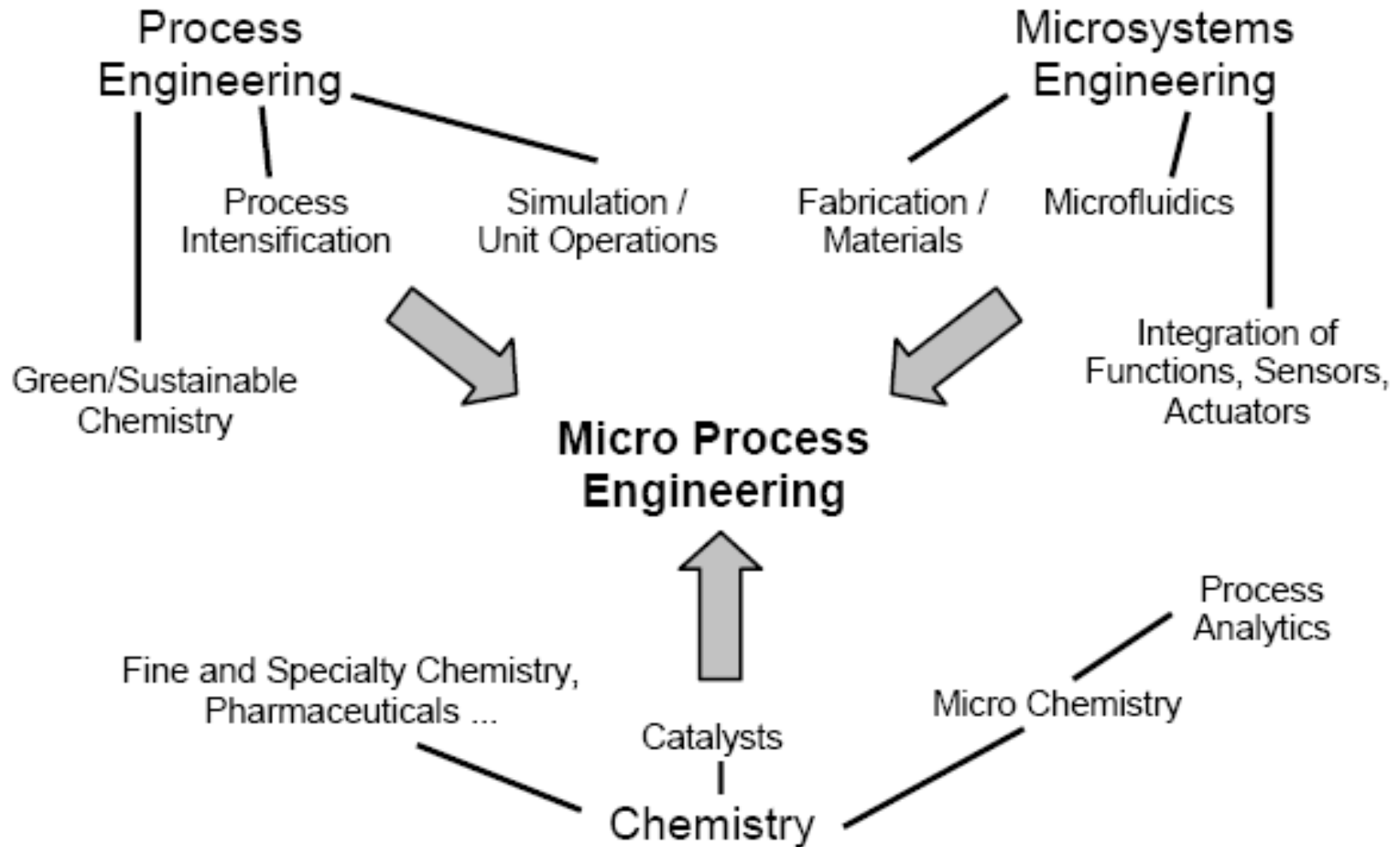
Correspond to low Reynolds and Mach number, e.g., less than 0.6 and 0.3, respectively.

*The corresponding Knudsen number, ( $\lambda / L$ ), is relatively large. –*

**Continuum Limit?**



Typical MEMS and nanotechnology applications in standard atmospheric conditions span the entire Knudsen regime (Continuum, slip, transition, and free-molecular flow). Here  $h$  denotes a characteristic length scale for the microflow.



Microprocess Engineering – an interdisciplinary field  
with inputs from various disciplines



# **Issues to be looked into**

Characteristic length and time scales

Transport phenomena in microstructures

Continuum range

Micro process engineering

Momentum and heat transfer in microchannels  
- Micromixers, Micro heat pipes

Coupled transport processes

Micro fabrication technology – process intensification

**Transfer lengths are short, areas are small but high surface-to-volume ratios and tiny volumes dominate everything**

**Small channels allow short transport lengths for heat and mass transfer.**

This results in high transfer rates, as described for diffusive mass transfer with the mean transport length from the

**Einstein-Smoluchovski equation**

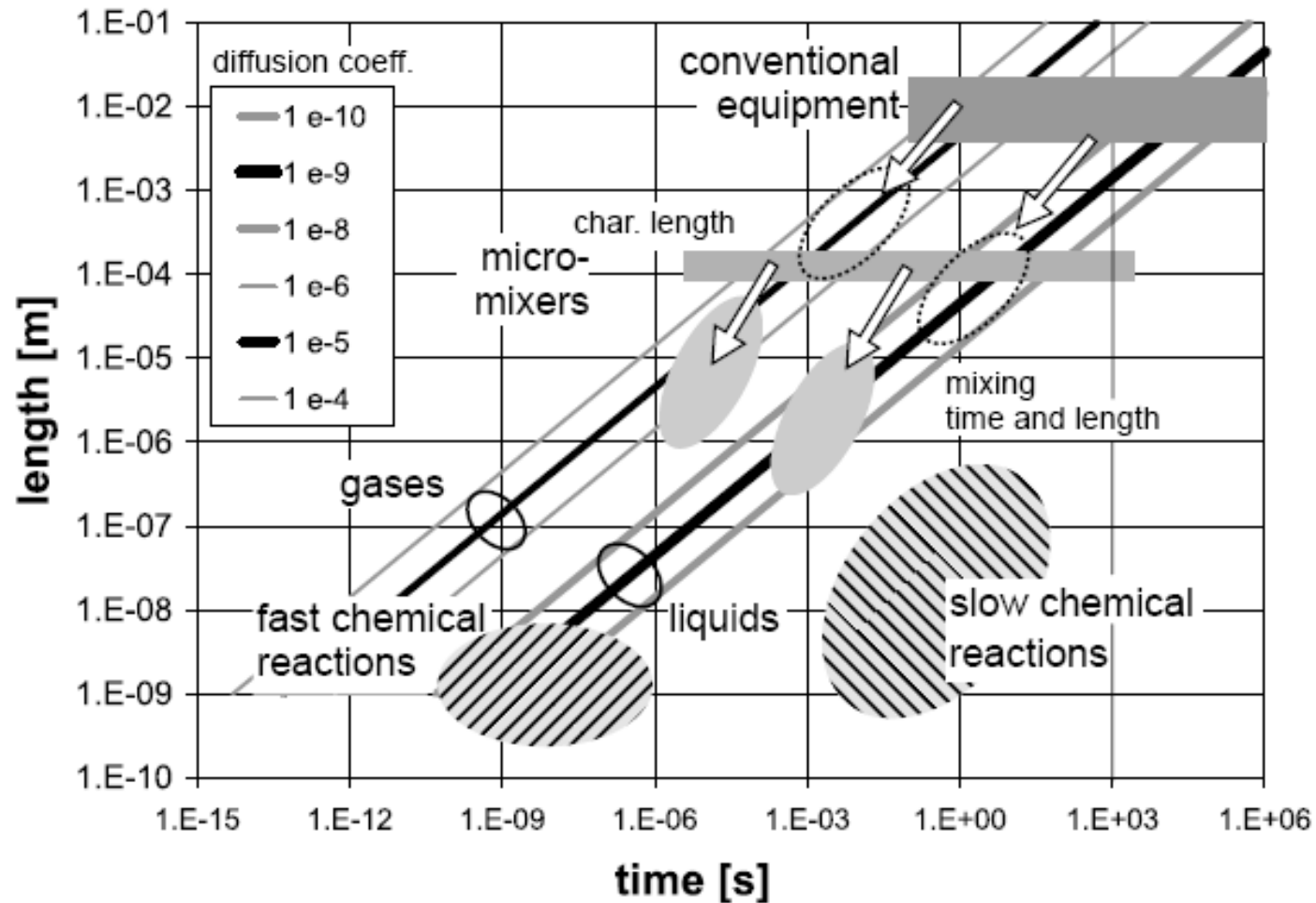
$$x^2 = 2 D t$$

The transport lengths by diffusive mixing in gases ( $D = 10^{-6} \text{ m}^2 / \text{s}$ ) and in liquids with low viscosity ( $D = 10^{-10} \text{ m}^2 / \text{s}$ ) are shown in the next figure.

In general, the shorter the length, the shorter the characteristic time for transport processes will be, and the higher the transformation frequencies.

$$x^2 = 2 D t$$

For diffusion of a species in a surrounding fluid, the typical diffusion length within one second is approx. 7 mm in gases (air) and approx. 70  $\mu\text{m}$  in liquids such as water.



Characteristics length and time scales for mixing in microstructured devices with chemical reaction

Conventional equipment has typical geometries in the range of centimeters and produces fluid structures in the range from 100  $\mu\text{m}$  to 1 mm. The corresponding diffusion time in gases is approx. 1 ms and in liquids in the range of 1 s.

Microstructured devices with typical length scales from 100  $\mu\text{m}$  to 1 mm provide fluid structures with length scales of approx. 1  $\mu\text{m}$ . These small fluid structures lead to mixing times shorter than 100  $\mu\text{s}$  in gases and approx. 1 ms in liquids.

This is the main reason for the enhanced selectivity and high yield of chemical reactions in microreactors.

The conduction length - from the basic balance equation

$$\text{momentum} \quad x_p = \sqrt{2\nu t} \quad \text{heat transfer} \quad x_q = \sqrt{2\alpha t}$$

$$\text{Thermal diffusivity} \quad \alpha = \frac{k}{\rho c_p} \quad \text{m}^2/\text{s}$$

k = Thermal conductivity

$$\nu = \text{Kinematic viscosity} = \mu / \rho, \text{ m}^2/\text{s}$$

Einstein-Smoluchovski equation

$$x^2 = 2 D t$$

The characteristic time is proportional to the square of the length variation and to the transport coefficient

Information about typical length and time scales for fast chemical reactions like neutralizations or slow chemical reactions such as polymerization are required to compare the processes.

The mass transfer in micromixers acts on a length scale of a few microns within milliseconds or less.

Different time scales are typical for partial reactions in complex reaction systems.

With properly designed micromixers and an adjustment of the component concentration, the selectivity of a complex reaction can be increased.

Important - the scale of fluid residence time within the device.

Within small devices, the fluids rest only briefly (seconds or less), which can be detrimental to slow reactions. A slow reaction may be incomplete at the channel outlet.

Fast reactions require short and small channels and a sufficiently high number of channels. They benefit from the rapid mixing and heat exchange.

Reactions with slower side-reactions or unstable intermediates show higher selectivity and higher yield in microstructured devices.

Reactions with high energy demand or release are suitable for micro devices.



The heat transfer in a straight channel with laminar flow is described by a constant Nusselt number  $Nu$ ,

$$Nu = \frac{h d_h}{k} = 3.65$$

for constant wall temperature.

With smaller channel diameter  $d_h$ , the heat transfer coefficient  $h$  increases.

Additionally, convective effects in bent channels can increase the Nusselt number for better performance but also increase the pressure loss

The fluid temperature  $T$  in the channel quickly approximates the wall temperature according to the following equation

$$T(x) \propto e^{-x/l_h}$$

with the characteristic length of

$$l_h = \frac{\dot{m} c_p}{3.65 \pi k}$$

Combining the channel distance and the mean residence time

with the mean velocity  $x = \overline{w} t$

Solve the wall temperature relation for the time-dependent temperature change to obtain the fluid temperature

$$T(t) \propto e^{-t/t_h}$$

The characteristic time,  $t_h$  is defined as

$$t_h = \frac{\rho c_p d_h^2}{3.65 \pi k}$$

With **decreasing channel diameter**, the fluid temperature exponentially approaches the wall temperature.

Efficient heat transfer is also important for **high exothermic chemical reactions** to transport the heat away from the reaction zone and to avoid hot spots.

The **high surface-to-volume ratio** is also responsible for the fast heat transfer in microchannels.

Additionally, a high surface-to-volume ratio is beneficial for **surface reactions** such as heterogeneous catalysis, emulsification or transport-limited processes

With unsteady heat transfer, the characteristic time  $t$  for heating or cooling of a body is proportional to the temperature difference and the ratio of the heat capacity  $mc_p$  to the heat transfer  $h A$  within the environment.

$$t = \frac{m c_p}{h A} = \frac{\rho c_p V}{h A} = \frac{\rho c_p}{h a_v} \propto \frac{\rho c_p}{h} \cdot d_h$$

$h$  is the heat transfer coefficient,  $A$  the surface area

With a smaller length scale  $d_h$ , the surface-to-volume ratio  $a_v$  increases and this characteristic relaxation time  $t$  becomes shorter.

# Lecture 2

## Microscale Manufacturing Practices

# Review

Mass transfer in micromixers acts on a length scale of a few microns within milliseconds or less.

Fast reactions require short and small channels and a sufficiently high number of channels, problems for slow reactions.

Combined reactions with slower side-reactions or unstable intermediates will show a higher selectivity and higher yield in micro-structured devices.

Reactions with high energy demand or release are suitable for micro-devices.

In the case of chemical reactions in microchannels, the mixing patterns can be controlled very effectively.

Hence mass transfer limited reactions are well-suited.

The characteristic dimensions of microreactors are in the range from 50 to 500  $\mu\text{m}$ , which produces a high specific surface and allows effective heat and mass transfer.

For continuously operating microstructured reactors, heat transfer, temperature control, mixing and residence time characteristics, are optimal for a channel diameter of approx. 200  $\mu\text{m}$ .



# Two Approaches in Micro/Nano Manufacturing

Top-down processes: one starts on the macro scale and proceeds to create fine features by processing the bulk on a fine scale - Microelectronics

More expensive as the feature dimensions become smaller

Bottom-up processes: starts at the smallest possible scale, at the atoms and molecules themselves, and builds complexity up from there. Processes controlled by self-assembly

Embryology is the ultimate bottom-up process of producing a macro-scale complex entity by manipulation at the smallest scale possible.

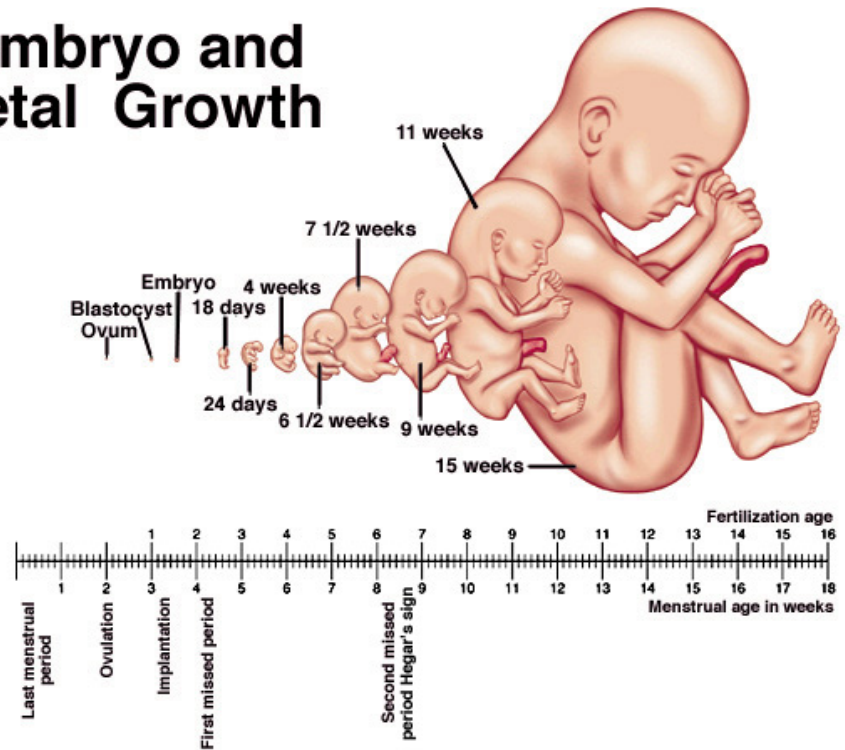
# Micro-Nano Manufacturing



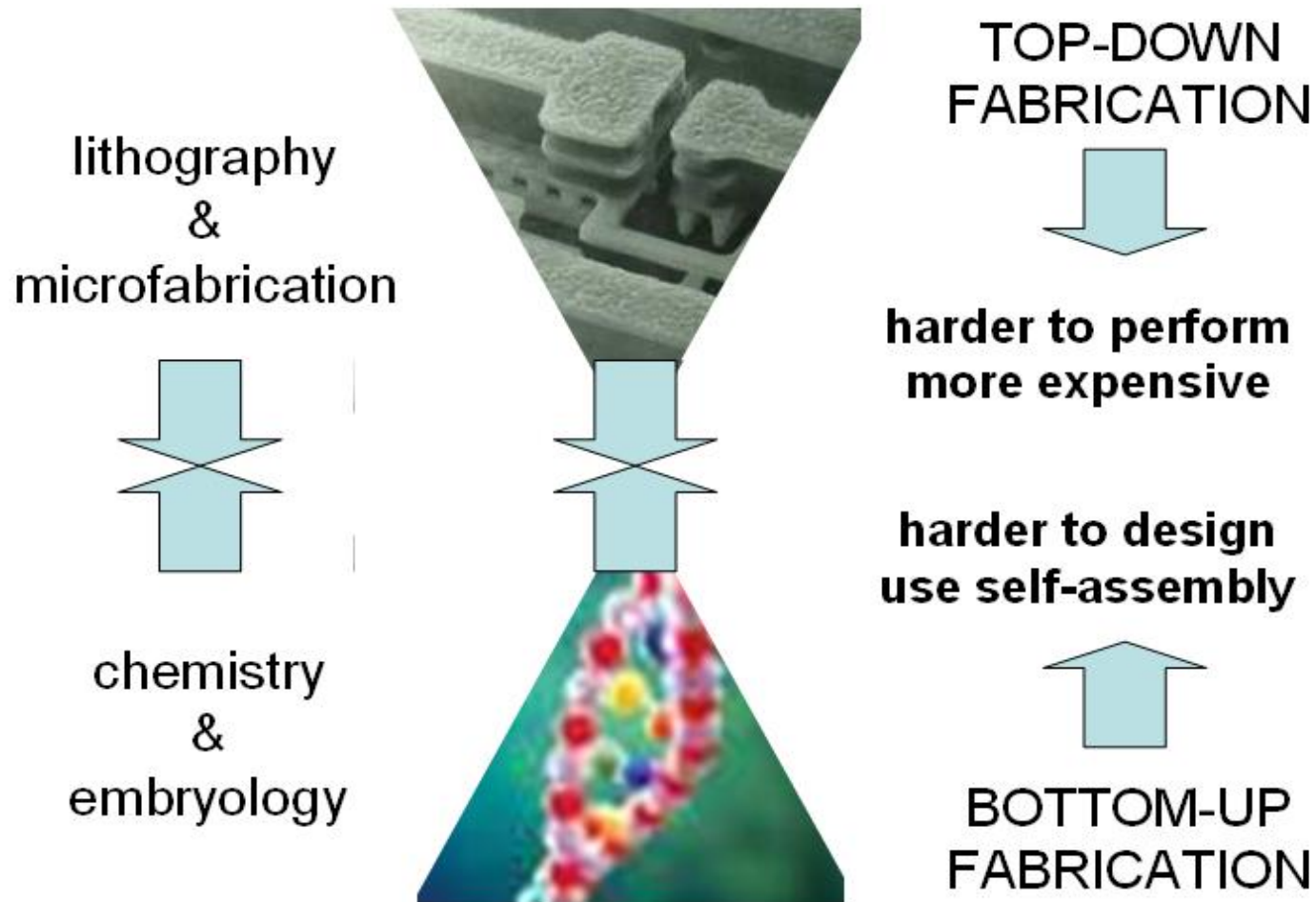
Kailash Temple, Ellora

Byer/Shainberg/Galliano *Dimensions Of Human Sexuality*, 5e. Copyright © 1999. The McGraw-Hill Companies, Inc. All Rights Reserved.

## Embryo and Fetal Growth



# Top-down and Bottom-up Processes



# Lithography – Basics

## Photolithography –Printing with Light

- Lithography is a process that uses focused radiant energy on chemical films that are affected by this energy to create precise temporary patterns in silicon wafers or other materials.
- These temporary patterns can be used to add or remove material from a given area

# Lithography - Basics

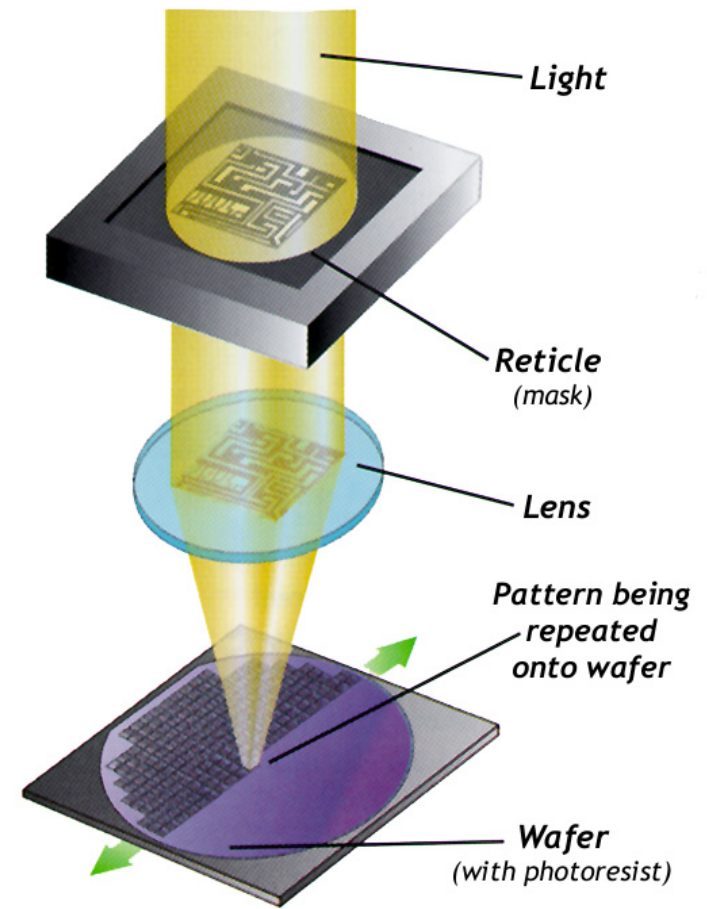
- Lithography in the top-down approach
  - Lithography
  - Etching
  - Deposition
  - Doping
- In order to perform the other 3 processes, we must precisely define where to perform these operations
  - Lithography Does This

# Role of Lithography in the Process

- With multiple etch, deposition, and doping processes taking place in the fabrication of a device, the lithography process is repeated many times.
- The precision and accuracy of lithography in the manufacturing process controls the success in building a device.

# Overview of the Photolithography Process

- Photolithography uses light energy passing through a patterned mask
- The light is focused onto the photosensitive surface
- Chemical changes in the surface coating occur
- Subsequent chemical development creates a temporary pattern on the surface.



# Photolithography –Printing with Light

## STEPS

1. Cleaning - RCA cleaning/Peroxide cleaning
2. Preparation – Heat + adhesion promoter
3. Photoresist application – Spin coating + baking -  $\mu\text{m}$
4. Exposure and developing – UV + Metal ion free developers + post exposure bake
5. Etching – wet (liquid, isotropic) and dry (plasma, anisotropic)
6. Photoresist removal – resist stripper or plasma with  $\text{O}_2$

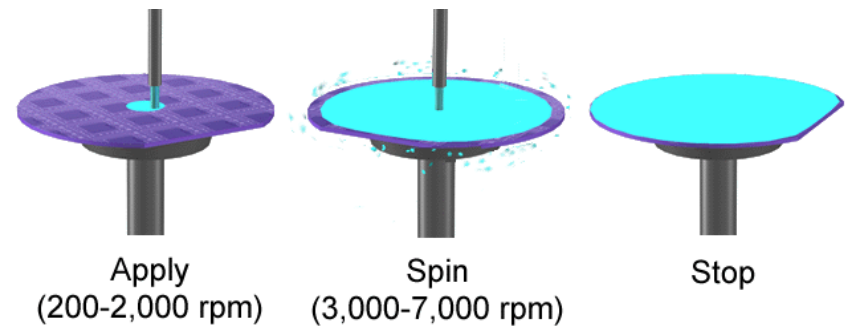
Limited by the wavelength of the light and the lens system  
minimum feature sizes 50 nm.



# Steps in Lithography

- Silicon wafers are commonly used substrates in the top-down process.
- The first step is to coat the clean surface of the wafer with a light sensitive chemical emulsion known as photoresist

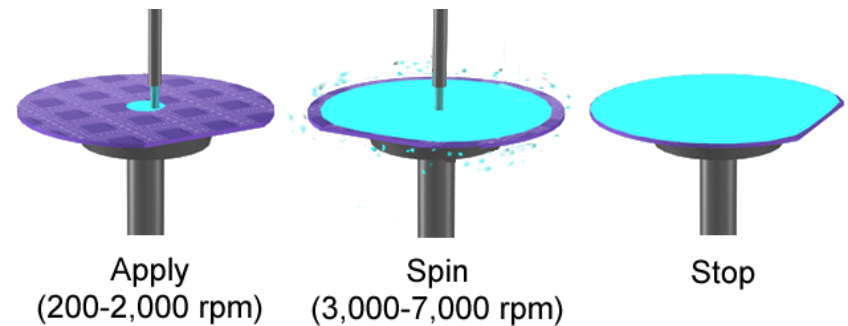
## Photoresist Dispensing (Spinners)



# Steps in Lithography

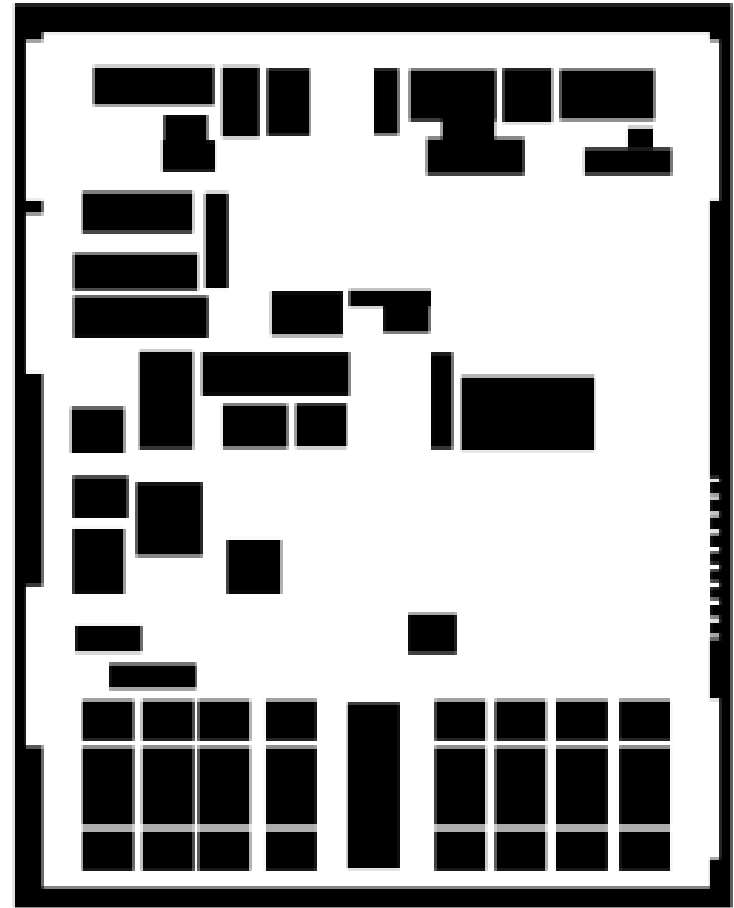
- Baking the resist causes it to form a solid layer.
- The chemical properties of the photoresist define what wavelengths of light will affect it.

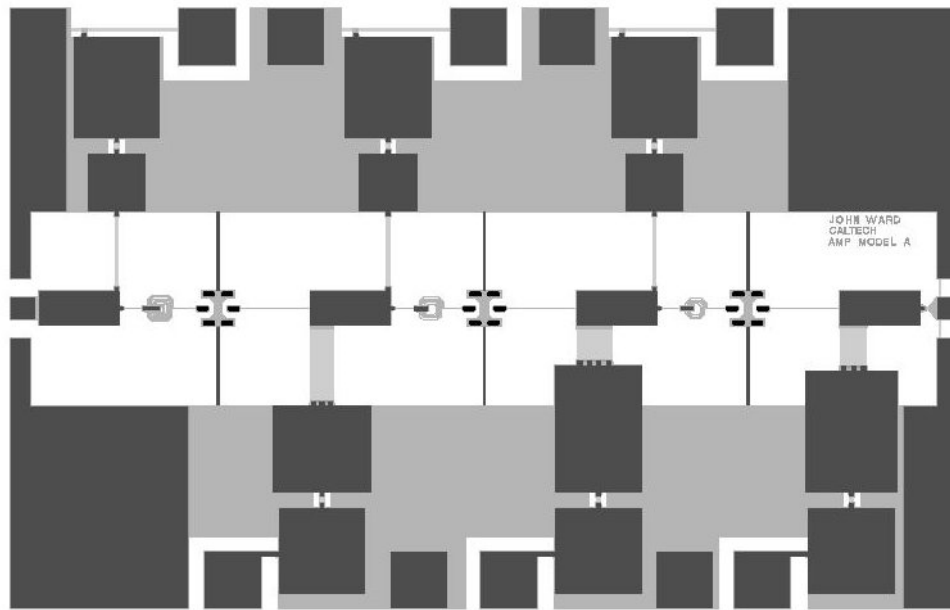
## Photoresist Dispensing (Spinners)



# Steps in Lithography

- A photomask, typically made of quartz with a chrome plating, controls where the radiant energy will strike the photoresist.
- Photomasks are often made with electron beam patterning tools



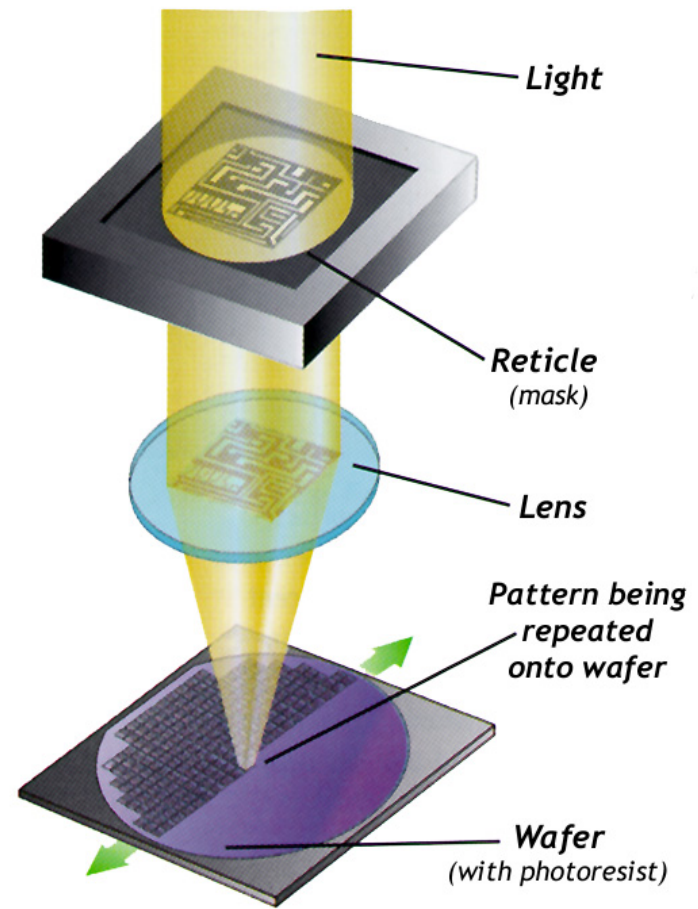


CAD layout of an entire circuit, eventually going to be reduced to fit on a chip 2.5 x 3.8 mm

On a single chip, the width of those connecting lines in there is going to 0.014 microns or so. That's the limiting factor in device density today — the width on the chip of the smallest line we can transfer from a CAD layout pattern into actual metal on silicon.

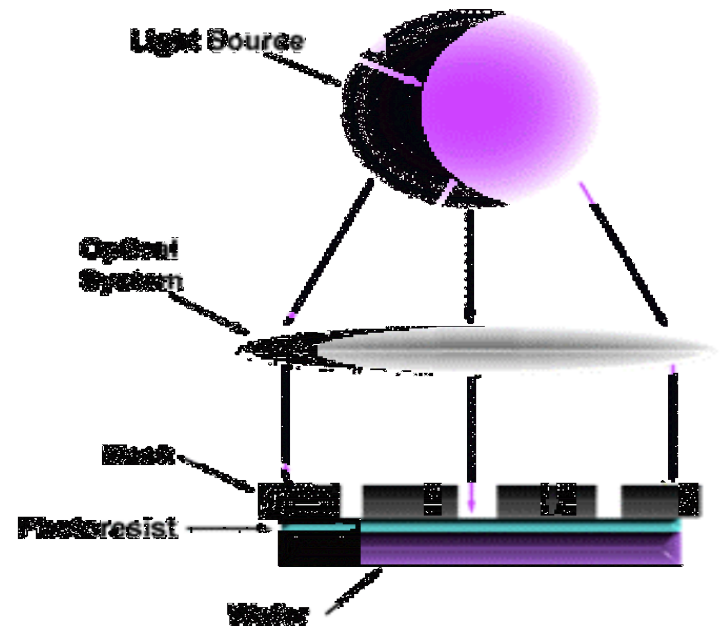
# Steps in the Lithography Process(3) - Exposure

- Exposure of the photoresist to the radiant energy pattern occurs next
- There are several ways to do this
  - Contact/proximity printing
  - Projection printing (shown here)
  - Projection scanning



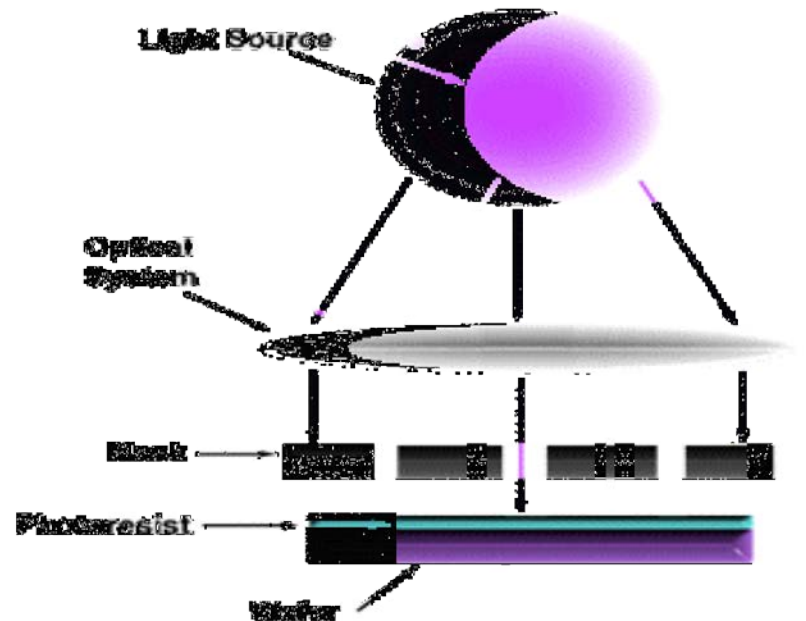
# Contact Printing

- The mask is directly in contact with the wafer
- Advantages
  - Simple
  - Low Cost
- Disadvantages
  - Poor for small features
  - Mask damage may occur from contact
  - Defects from contaminants on mask or wafer due to contacting surfaces



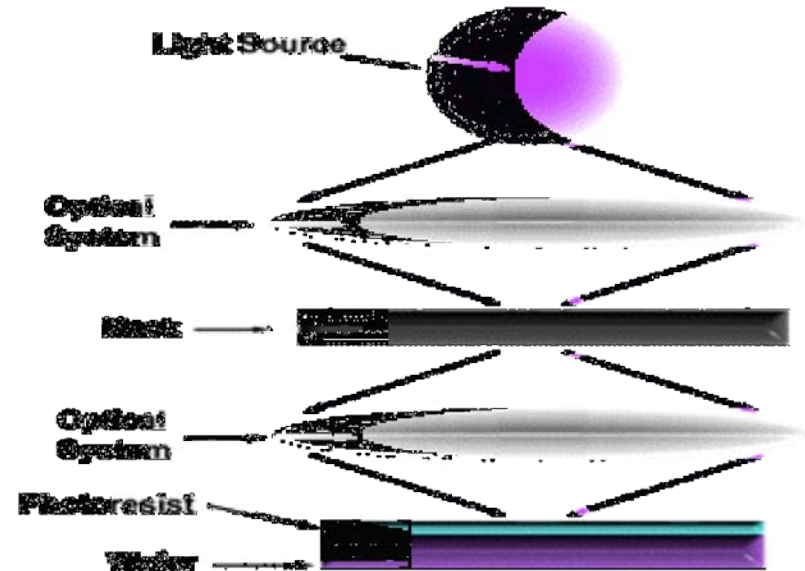
# Proximity Printing

- The mask is above the wafer surface
- Advantages
  - Mask damage is minimal
  - Good registration possible
- Disadvantages
  - Poorer resolution due to distance from the surface
  - Defects from contaminants on mask or wafer due to contacting surfaces
  - Diffraction errors



# Projection Printing (1)

- An optical system focuses the light source and reduces the mask image for exposure on the surface
- Advantages
  - Higher resolution
  - Lens system reduces diffraction error
- Disadvantages
  - Errors due to focus of lens system may occur
  - Limiting factor in resolution can be due to optical system

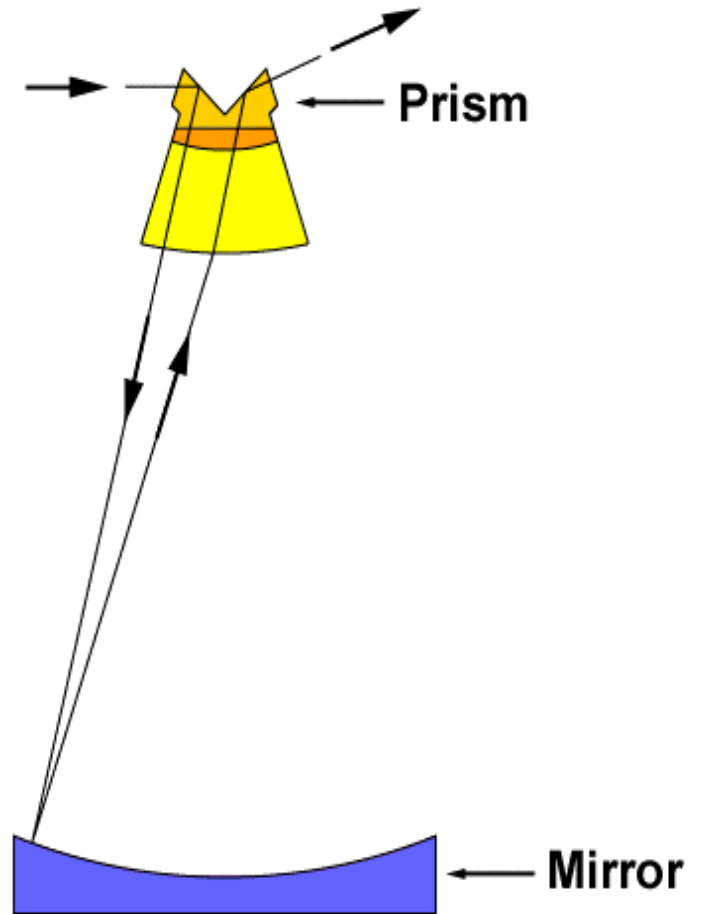


The minimum feature size  $F = K (\text{wavelength}/\text{NA})$  where  $K$  = process constant typically about 0.5. Numeric aperture is typically less than 1.



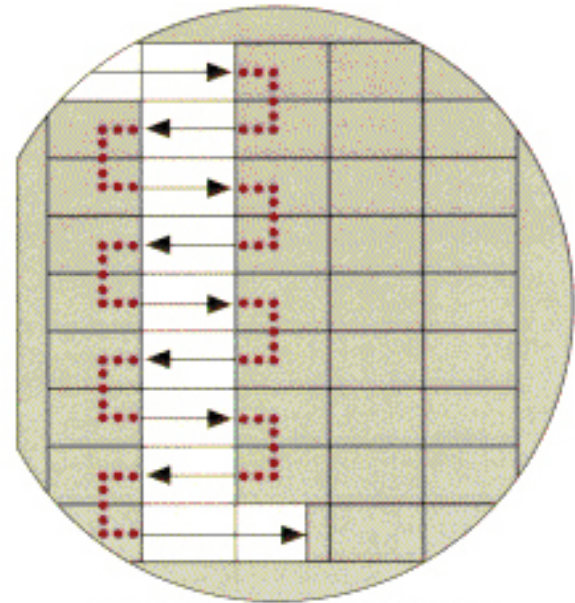
# Projection Printing (2)

- Step and repeat aligner
  - Lens reduction
  - Good throughput but resolution limited to about 0.35  $\mu\text{M}$
- Cadotrophic System
- Mirror, folding prisms and lenses 1:1 ratio
- Less common than steppers



# Step and Scan Aligner

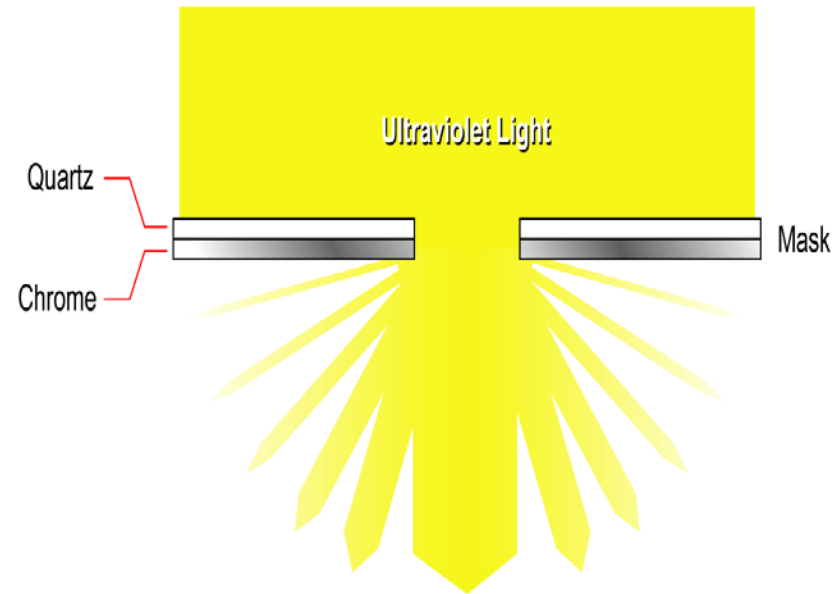
- Uses a spherical mirror and a scanning pattern
- Advantages
  - Improved throughput
  - Lens system aberration minimized
- Disadvantages
  - Complex motion system is required for alignment and precise tracing
  - Light source wavelength is still a factor limiting feature size



Courtesy of Silicon Valley Group

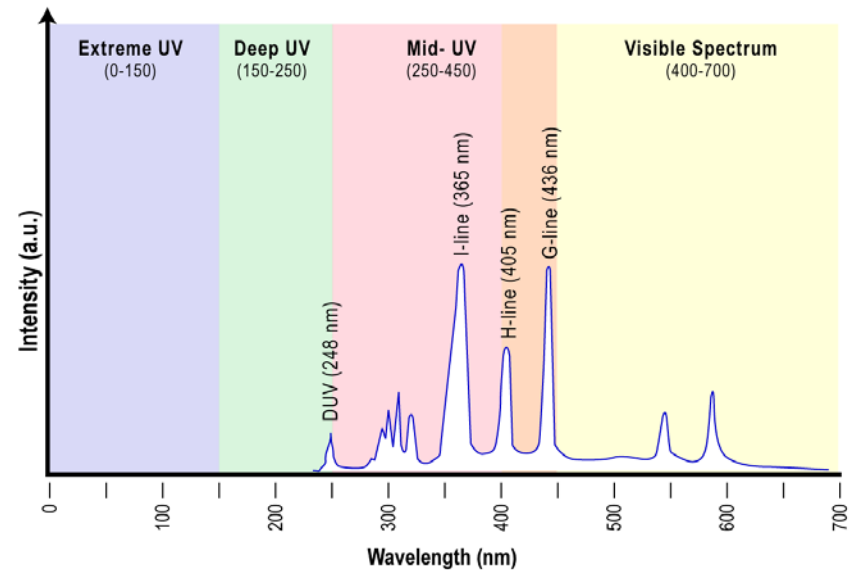
# Diffraction

- As feature sizes shrink in the mask, the wavelength of the light used as a source becomes a factor.
- Shrinking feature sizes require shorter wavelengths of light
- The photoresist must be optimized to match the light source used.



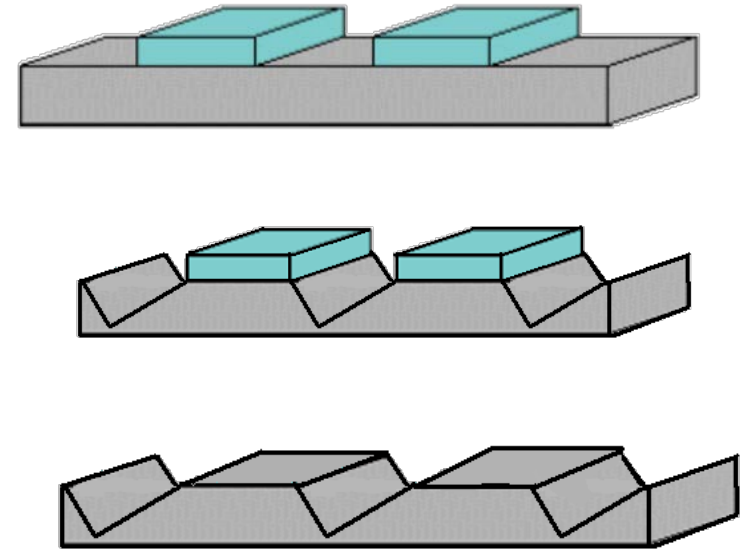
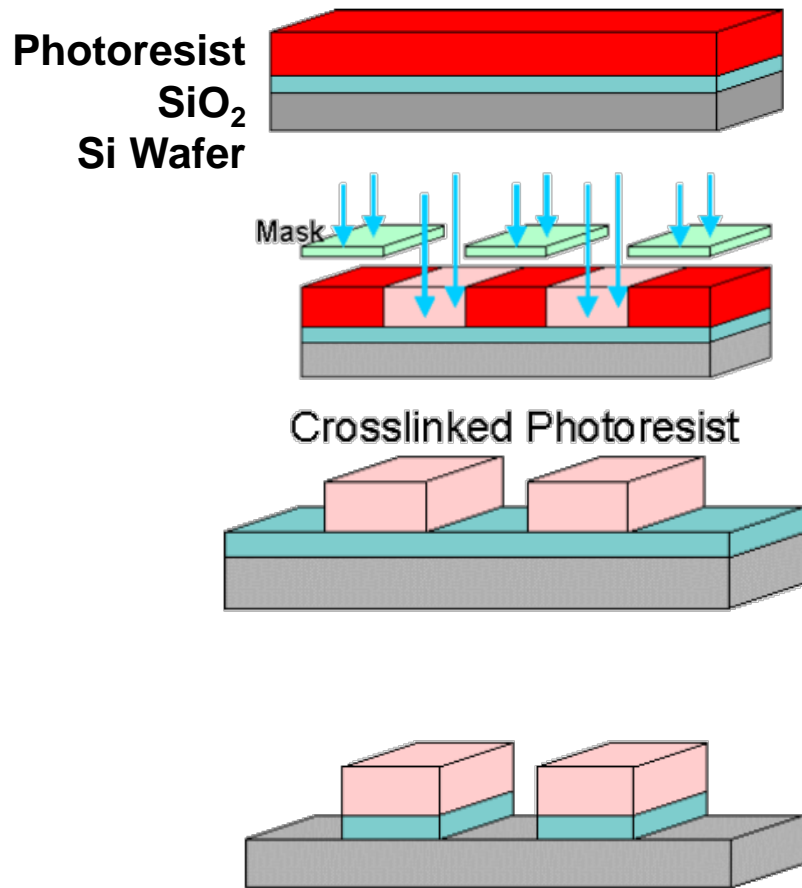
# Diffraction (2)

- The traditional mercury vapor lamp has peaks in certain ranges.
- The intensity of some UV peaks is low
- The photoresist must be optimized to match the light source used.



# Top-Down Approach

## Fabrication of V-grooves on Si wafer



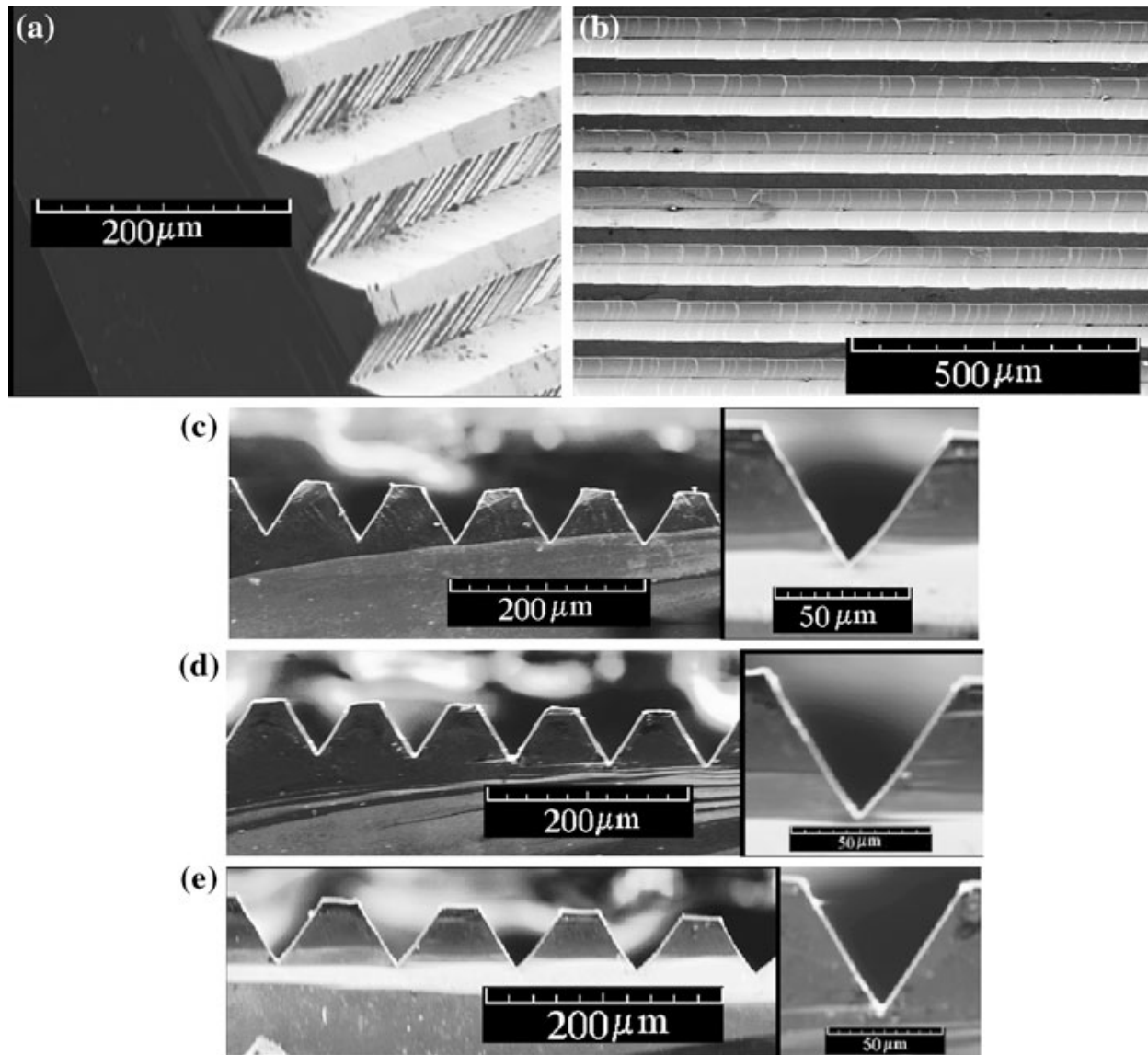
### Photoresist

+ve - whatever shows, goes

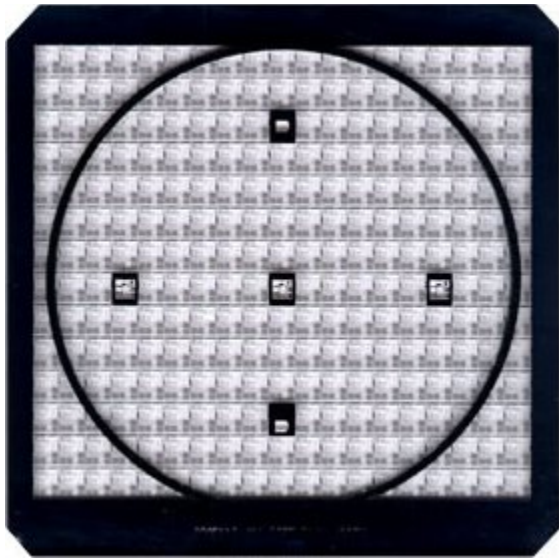
-ve - opposite

+ve - better process controllability for small geometry features. Popular in VLSI fabrication processes.

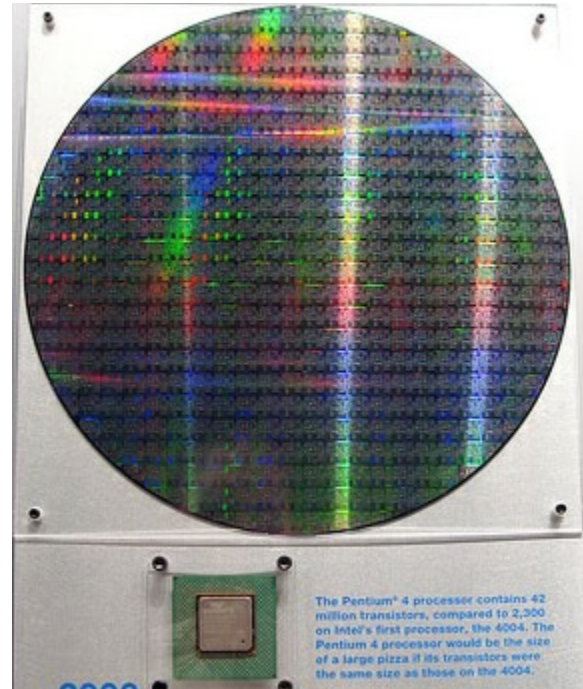
Photolithography, limited by the resolution of lithography



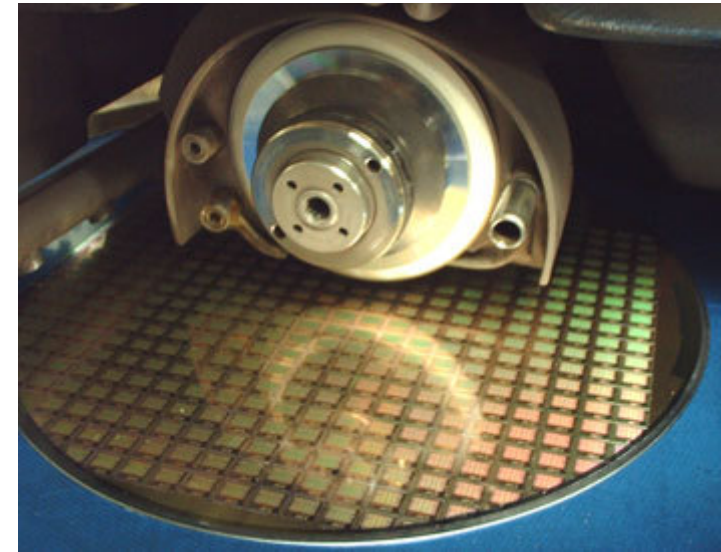
SEM of microgrooves on silicon substrate a - angular view of the microgrooves; b top view of the microgrooves; c-e Cross sectional views at three different locations along the length of microgrooves



Wafer mask for optical projection (one layer)



Wafer at the end of fabrication (many layers)

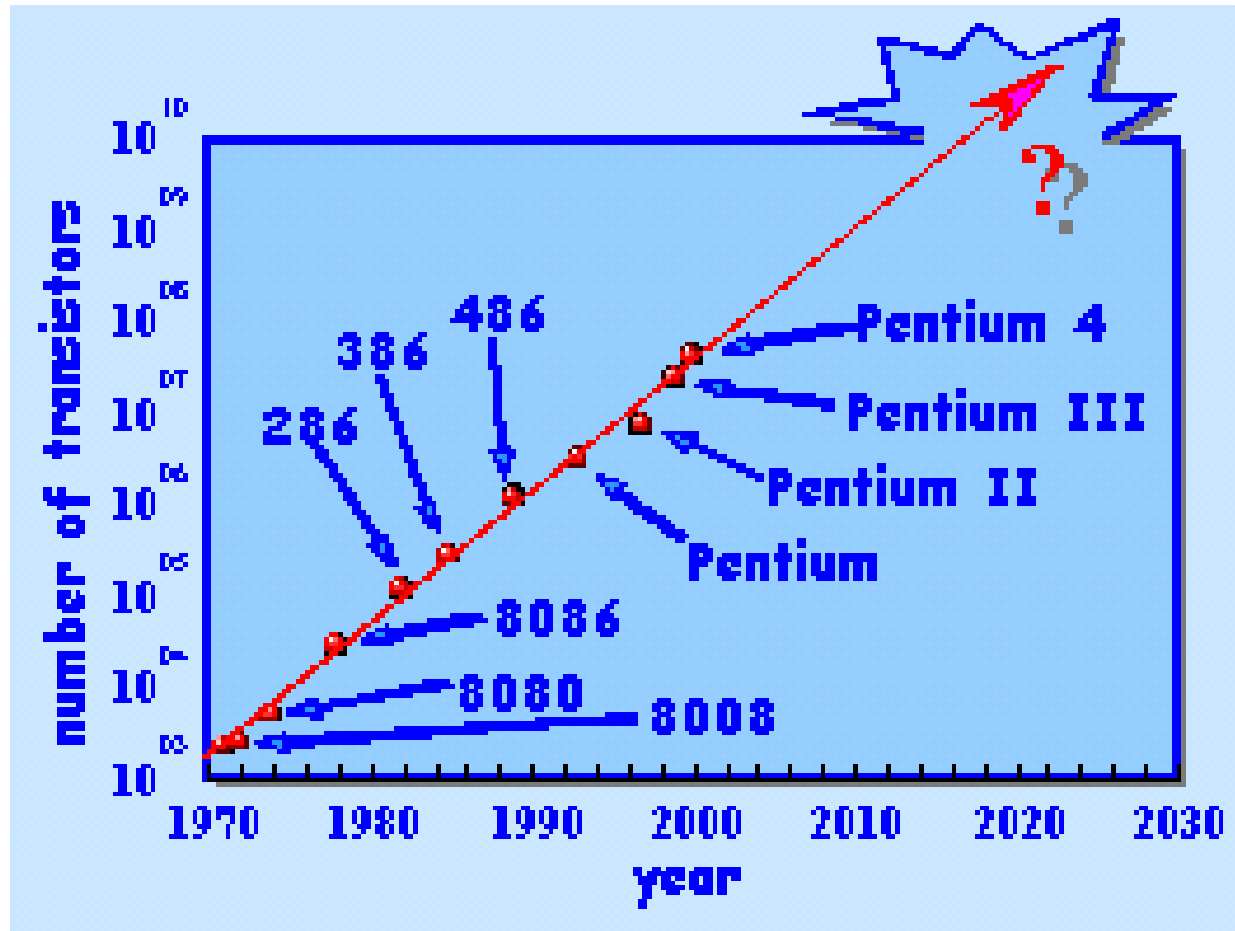


Wafer being cut up into chips after fabrication

**Picture of a wafer after all the photolithography steps (depositing, masking, etching) are done**

# Problems of Top-down Process

- Cost of newer technologies.
- Physical limits of photolithography
- heat dissipation



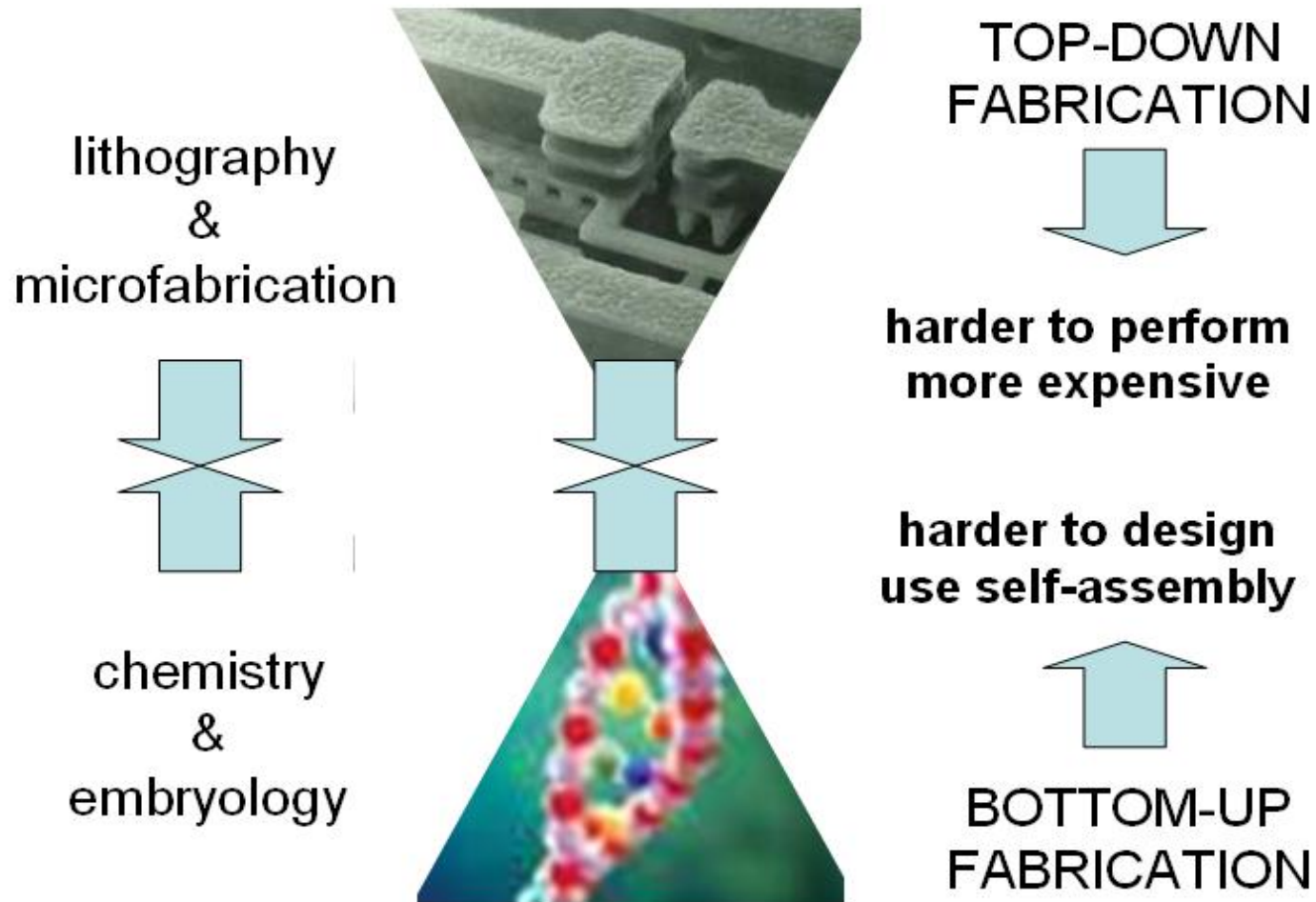


# Lecture 3

## Microscale Manufacturing Practices

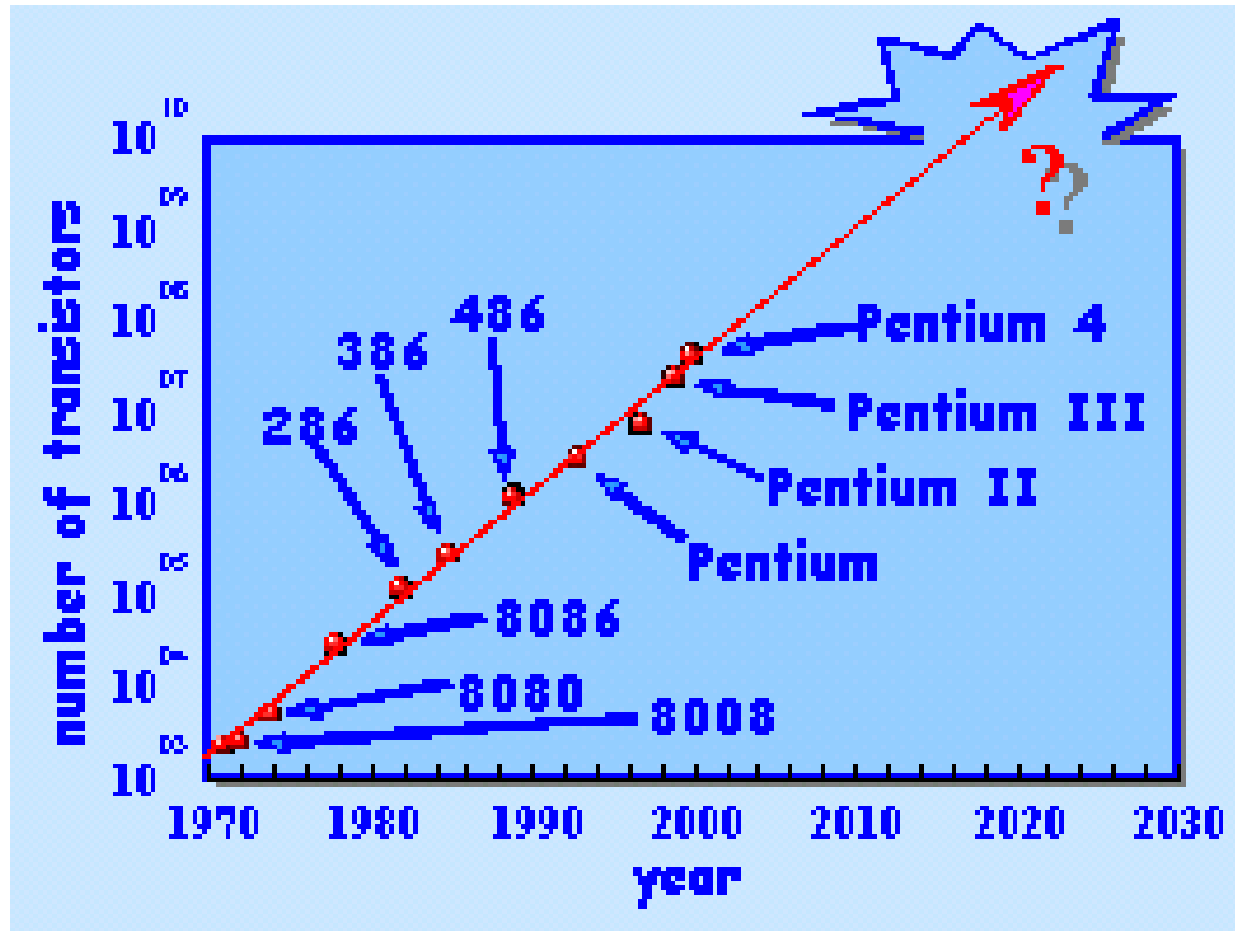
### Bottom up Approach

# Top-down and Bottom-up Processes



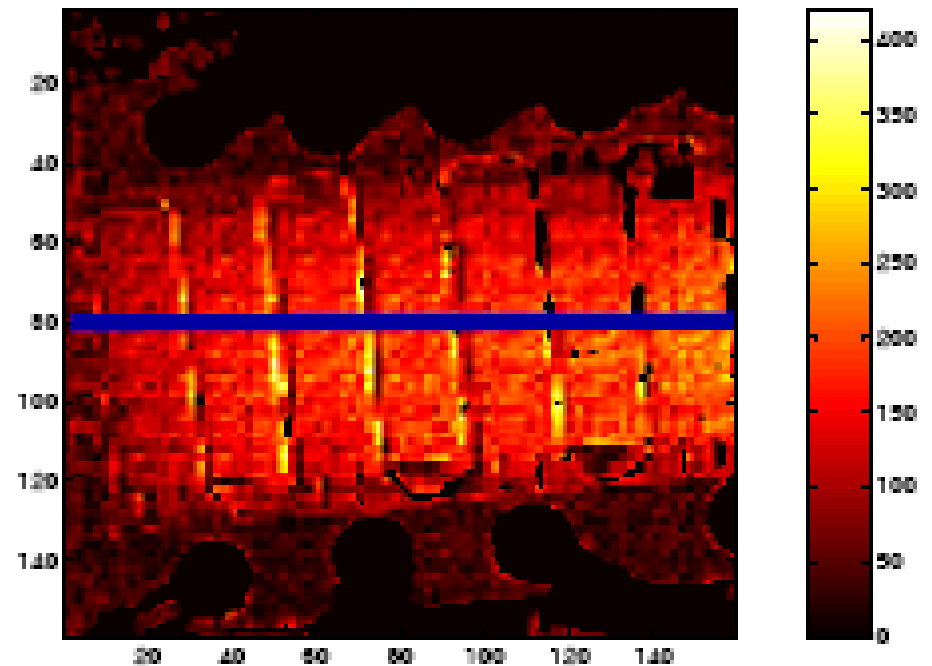
# Problems of Top-down Process

- Cost of newer technologies.
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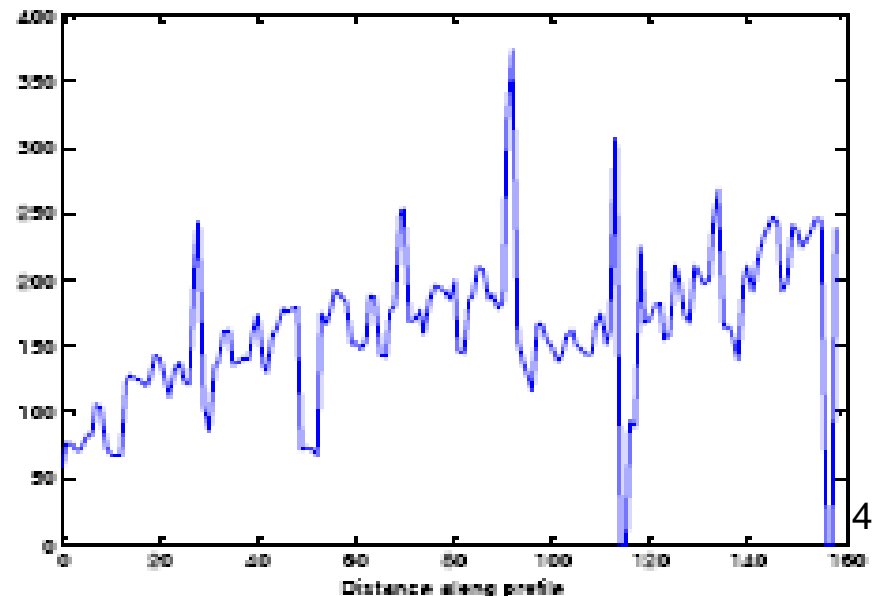


*Heat buildup is becoming one of the major limitations to creating tomorrow's more compact, complex micro devices”.*

David Benson of Sandia National Laboratory's Advanced Packaging Department.



At 450 mA and 100  $\mu$ s on time and 1 KHz frequency



# Top-down Versus Bottom-up

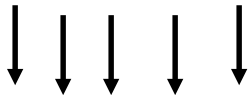
## Top Down Process



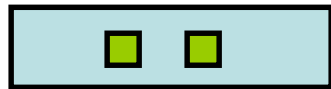
Start with bulk wafer



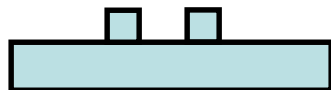
Apply layer of photoresist



Expose wafer with UV light through mask and etch wafer



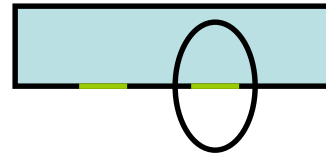
Etched wafer with desired pattern



## Bottom Up Process



Start with bulk wafer



Alter area of wafer where structure is to be created by adding polymer or seed crystals or other techniques.



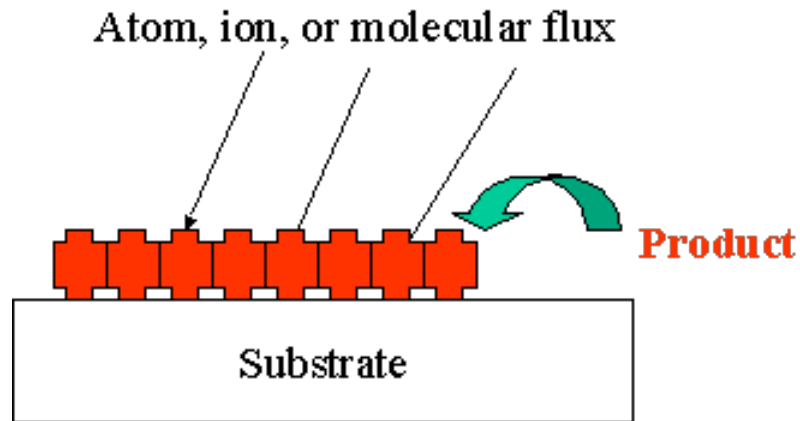
Grow or assemble the structure on the area determined by the seed crystals or polymer. (self assembly)

Similar results can be obtained through bottom-up and top-down processes

# Why is Bottom-Up Processing Needed?

- Allows smaller geometries than photolithography.
- Certain structures such as Carbon Nanotubes and Si nanowires are grown through a bottom-up process.
- New technologies such as organic semiconductors employ bottom-up processes to pattern them.
- Can make formation of films and structures much easier.
- Is more economical than top-down in that it does not waste material to etching.

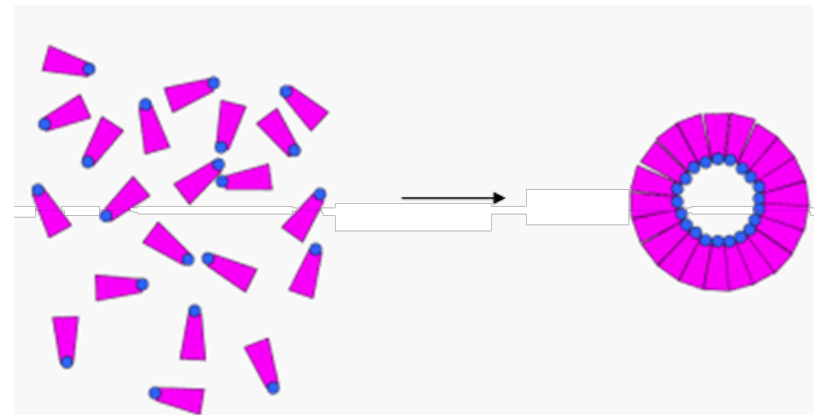
# Bottom-Up Approach



- The opposite of the top-down approach.
- The bottom-up approach selectively adds atoms to create structures.

# The Ideas Behind the Bottom-up Approach

- Nature uses the bottom up approach.
  - Cells
  - Crystals
  - Humans
- Chemistry and biology can help to assemble and control growth.



<http://www.csacs.mcgill.ca/selfassembly.htm>

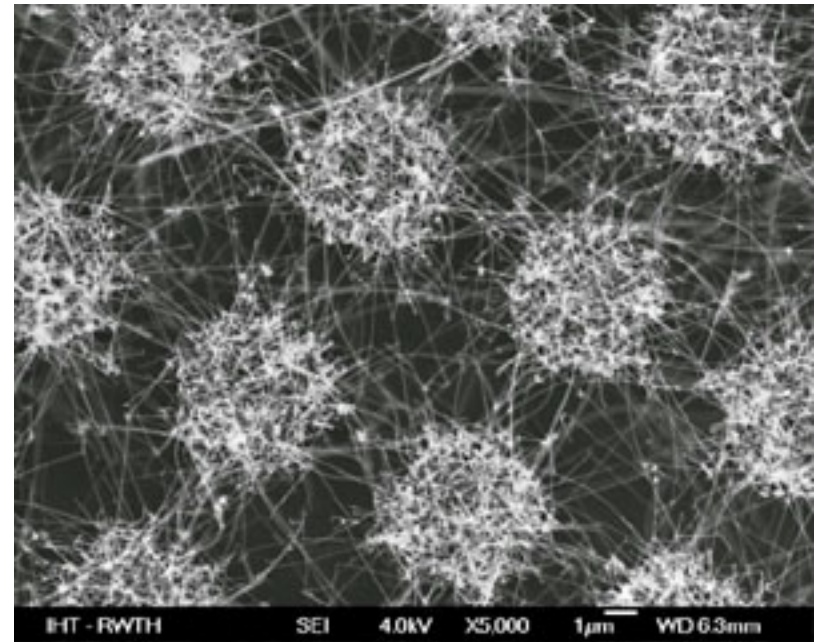


# Self Assembly

- The principle behind bottom-up processing.
- Self assembly is the coordinated action of independent entities to produce larger, ordered structures or achieve a desired shape.
- Found in nature.
- Start on the atomic scale.

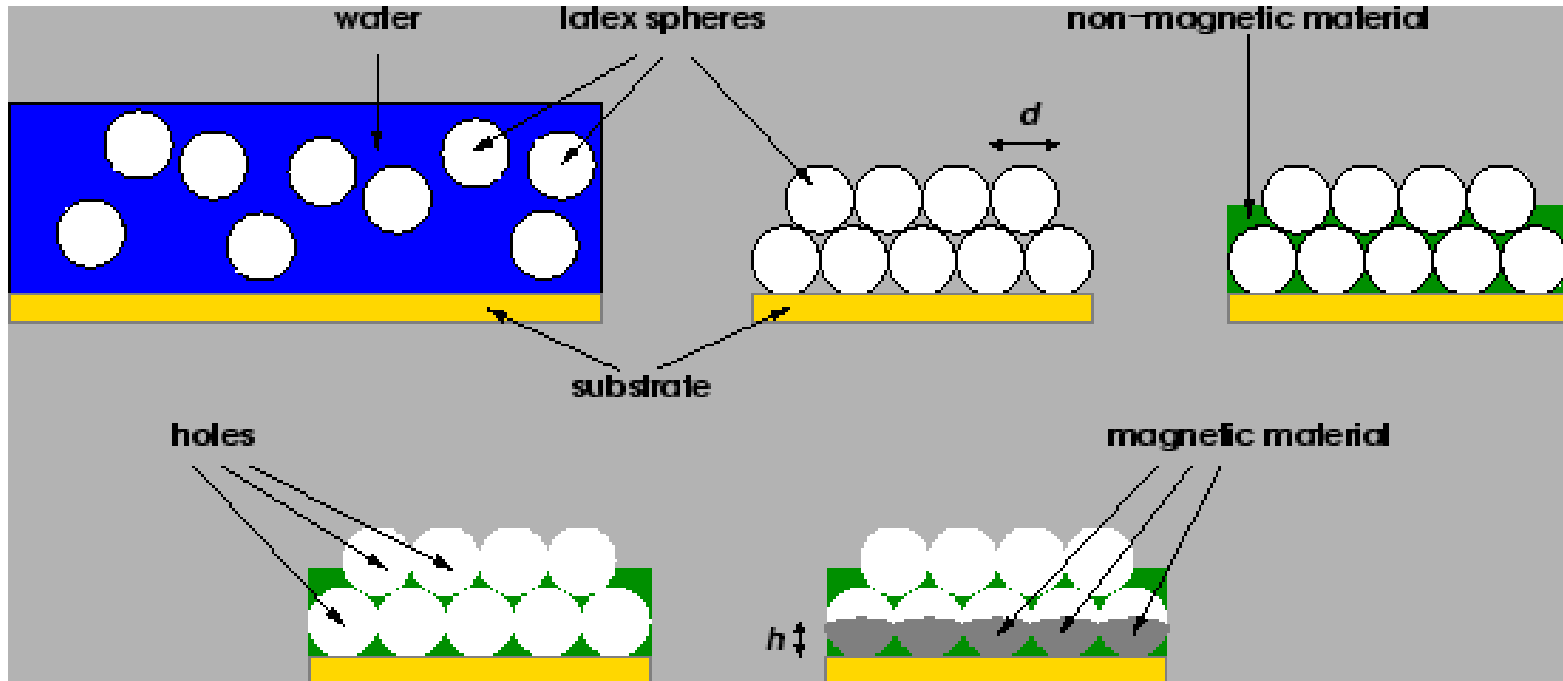
# Applications of Bottom-Up Processing

- Self-organizing deposition of silicon nanodots.
- Formation of Nanowires.
- Nanotube transistor.
- Self-assembled monolayers.
- Carbon nanotube interconnects.



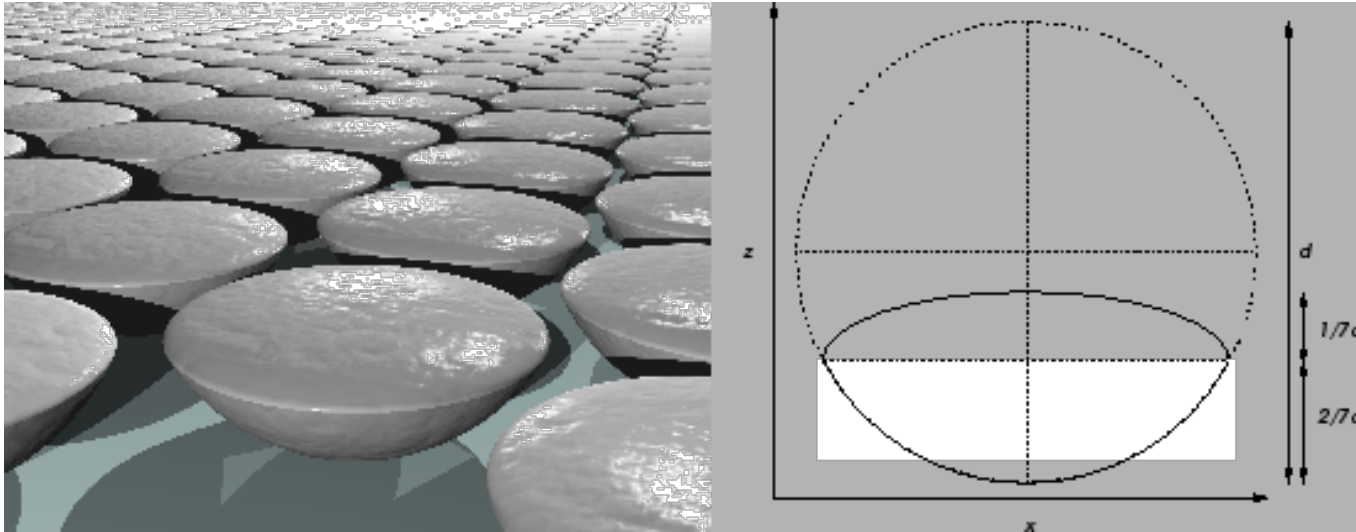
[http://web.ics.purdue.edu/~mmaschma/bias\\_image\\_gallery1.htm](http://web.ics.purdue.edu/~mmaschma/bias_image_gallery1.htm)

# Making of Nanodots



The **double-template self-assembly technique**. First, an aqueous suspension of latex spheres (top left) of diameter  $d$  is poured onto a substrate. As the water evaporates, the latex spheres are attracted to each other (top centre), forming a regular close-packed structure. This template can be filled with a non-magnetic material (top right) and the latex spheres etched away (bottom left). The resulting gaps can be filled with a magnetic material to a varying height  $h$  (bottom right) to form arrays of connected or disconnected part-spherical nanodots.

# Double-template self-assembly technique

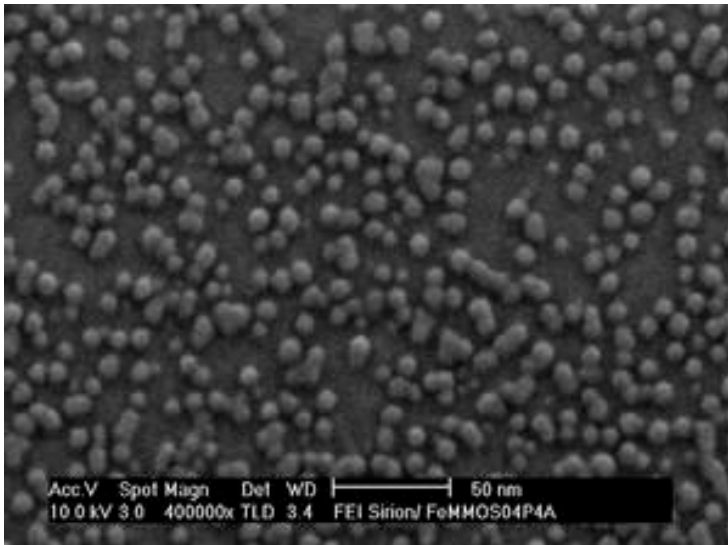


Using these templates, it is possible to create magnetic structures from sizes of 50nm to 1000nm by filling the spaces between the close-packed spheres with some material through electrochemical deposition.

By etching away the polystyrene spheres, another template is formed. This can then be filled with magnetic material, and by varying the fill amount of the resulting spherical holes, connected or disconnected arrays of dots can be formed. This is known as the **double-template self-assembly method**.

# Self-organizing Deposition of Silicon Nanodots.

## High-density information storage



<http://www.iht.rwth-aachen.de/en/Forschung/nano/>

bottomup/deposition.php

- Silicon nanodots are deposited onto silicon dioxide with no need for lithographic patterning.
- The nanodots can be thought of tiny magnets which can switch polarity to represent a binary digit.

Hard drives typically magnetize areas 200-250 nm long to store individual bits, while nanodots can be 50 nm in diameter or smaller

## Recent Developments

Silicon quantum dots (SiQDs) are semiconductor Si nanoparticles ranging from 1 to 10 nm

Potential as optoelectronic devices and fluorescent bio-marking agents due to their ability to fluoresce blue and red light.

The methods for producing nanosized silicon particles include

1. Laser ablation and non-thermal plasma synthesis
2. Electrochemical etching, reduction of silicon halides, thermal destruction of silicon-rich oxides, hydrothermal decomposition of different Si-contained organic precursors, oxidation of sodium silicide, processing porous silicon etc

# Bottom-Up Approach

Developing a simple, efficient method to organize molecules and molecular clusters into precise, pre-determined structure

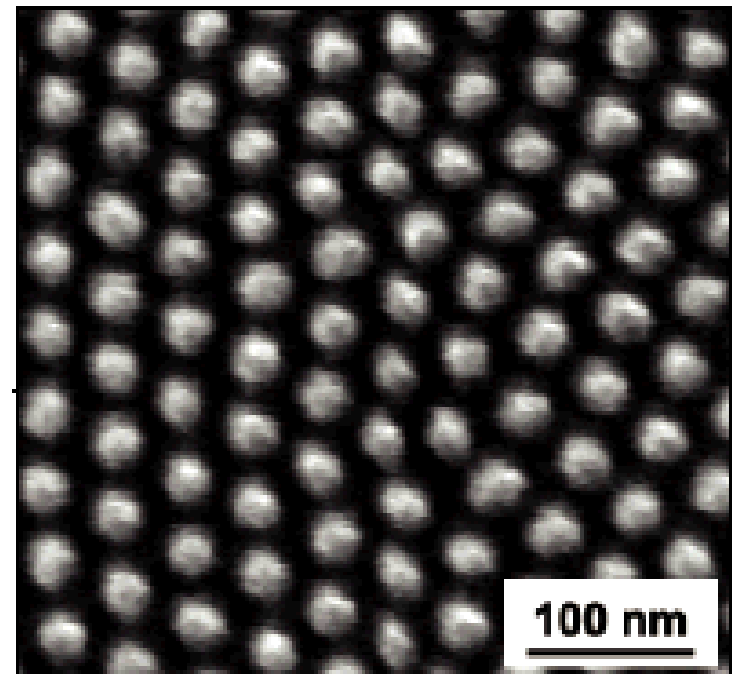
- Selectively adds atoms to create structures
- Nature - Cells, Crystals
- Chemistry and biology can help to assemble and control growth.

## Making Nanodots

Polymer template for nanodot

Self-assembled polymer film

Grow layer of desired material

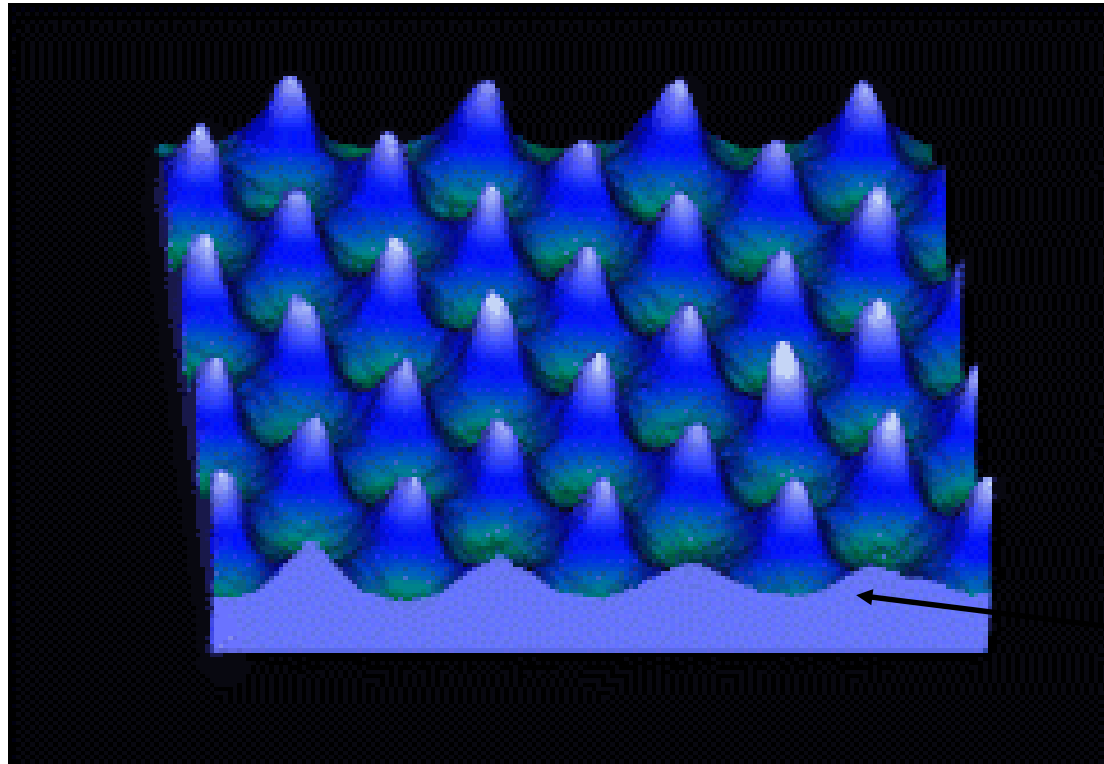


65 billion nanodots per square cm

## Self Assembled Nanodots

Each nanodot can  
hold one bit of  
information.

10 Trillion dots  
per square inch.





## Future: Nanodot-based Smartphone Batteries

**Energy storage** - extremely fast recharge, *nanodots*, chemically synthesized bio-organic peptide molecules that, due to their small size, improve electrode capacitance and electrolyte performance. The end result is batteries that can be fully charged in seconds.

It acts like a **supercapacitor** (with very fast charging), and on the other is like a **lithium electrode** (with slow discharge).

The electrolyte is modified with nanodots in order to make the multifunction electrode more effective.

# Summary

- Top-down processing – the prevalent choice - NOW
- Newer technologies for future products will require a bottom-up approach
- Combination of top-down and bottom-up processes to simplify fabrication?
- Self-assembly

## Process and Plant Design

The process and plant design procedure begins with a product idea or product formula, tested in the laboratory with stirred beakers or standard calorimeters.

The chemical recipes and protocols are developed, which must be transferred into a technical process.

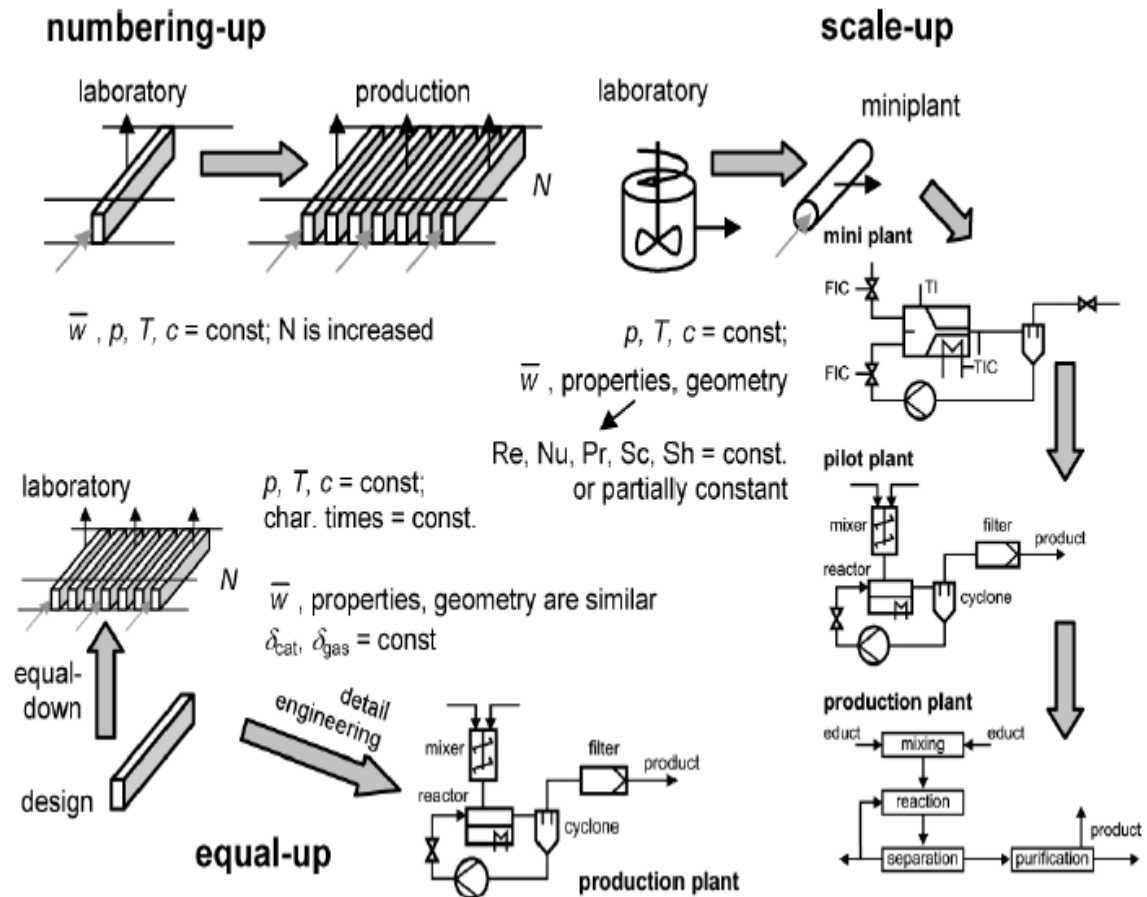
Conventional way,

Miniplant → Pilot Plant → Production facility

Right side of Figure

# Scale-up, Numbering-up

In conventional process engineering, the scale-up process is quite elaborate, Starting with laboratory experiments, the gained data are transferred to mini plants with almost the same length scale, but a more accurate representation of continuous flow processing.



The pilot plant provides feedback on a semi-industrial scale for the correct design of the final production plant. Wherever possible the process conditions of temperature, pressure and concentrations are kept constant.

Channels are the basic element of microstructured equipment, often with rectangular cross section.

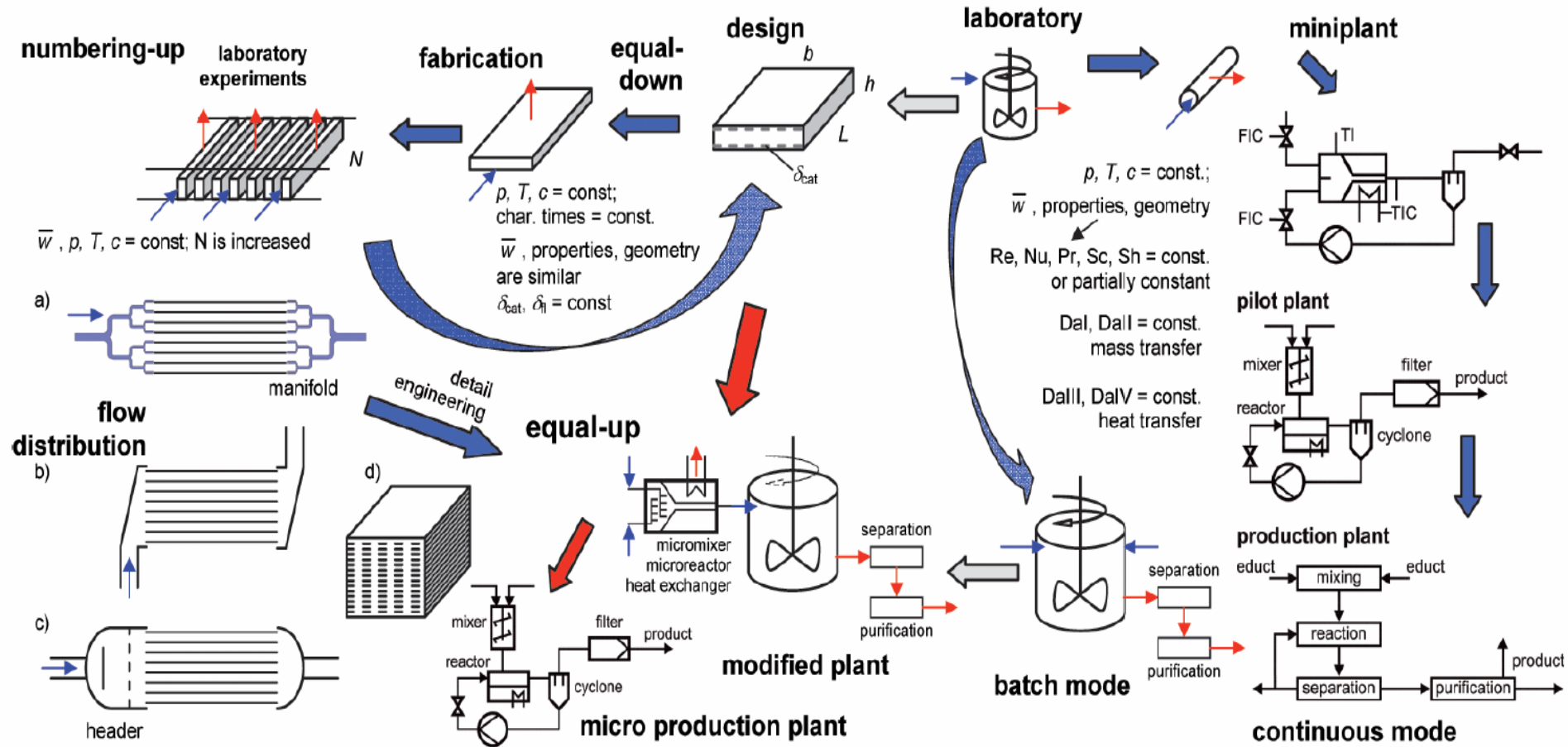
Depending on the process conditions, such as temperature, pressure, and fluid properties, the channel cross section, catalyst layer (if necessary), and minimum numbers are determined on the basis of available fabrication technology and device material.

This process is called equal-down, because the small channel dimensions are determined according to the necessary heat and mass transfer and relevant chemical kinetics.

## equal-up or scale-out

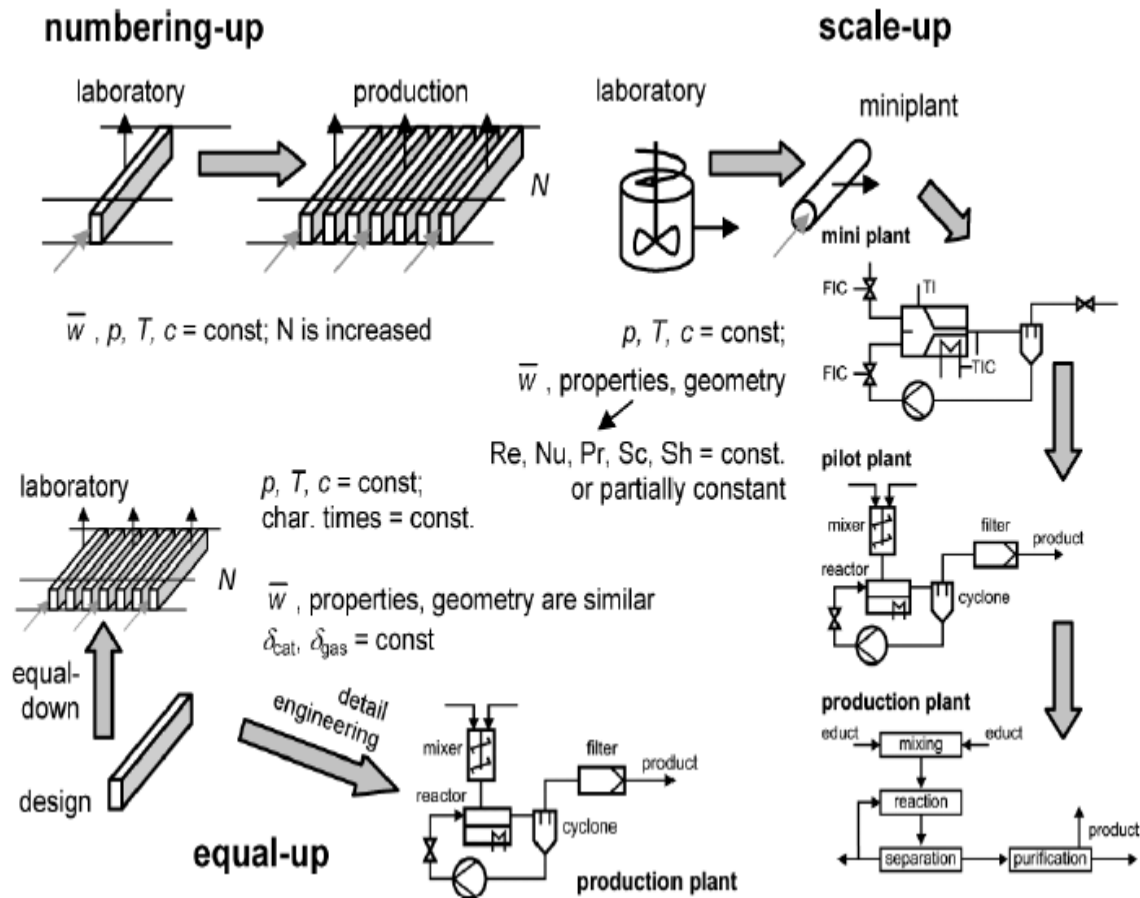
## microstructured devices

## scale-up



Equal-up and scale-up procedures with microstructured devices<sup>22</sup>

The geometry is represented along with suitable process parameters in dimensionless numbers like the Reynolds number  $Re$  or Sherwood number  $Sh$ , which are kept constant or at least partially constant for the process transfer.



The **numbering-up** procedure increases, in the simplest approach, just the number of channels, to enlarge the capacity. The flow distribution and correct integration must be considered and are the major critical points for a successful implementation. With more channels in a microstructured device, well designed manifolds are extremely important for performance.

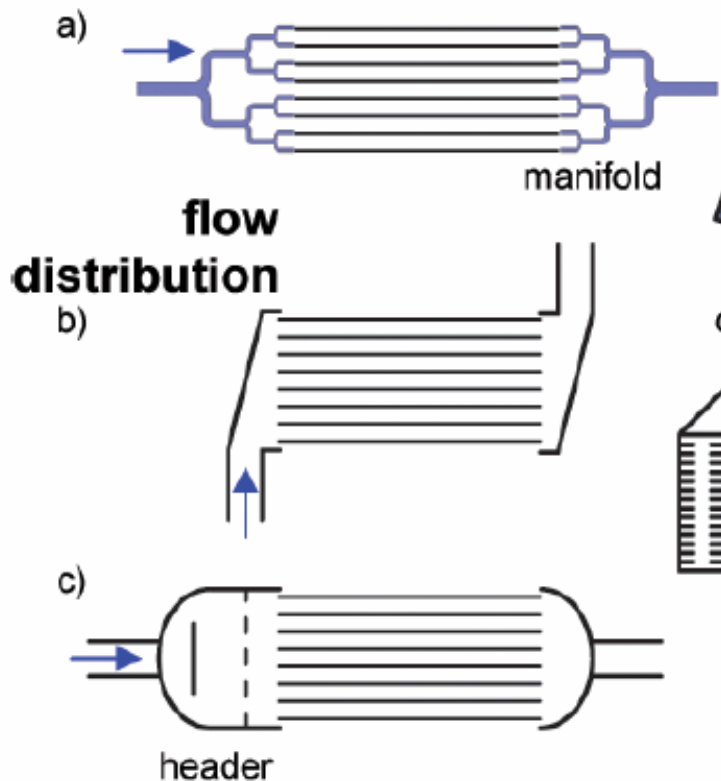
A laboratory device is fabricated and tested with the chemical system to yield experimental data. This data is compared with design assumptions and preliminary simulation results.

In case of successful experimental tests, the next design step is to layout a device or a number of devices handling the desired product capacity - **internal numbering-up** of these elements increases the entire flow rate.



The flow distribution and correct integration is the most critical point for successful implementation.

Depending on the size of the entire device, various fluidic manifolds or flow headers can be applied.



The velocity of the inlet flow is directed to the side walls by a central plate. With this arrangement, all channels are facing the same fluid velocity and are supplied with uniform flow rate.

The appropriate flow manifold depends on the number of channels as well as the shape of the entire device.

Microstructured devices for high flow rates often consist of a stack of microstructured plates.

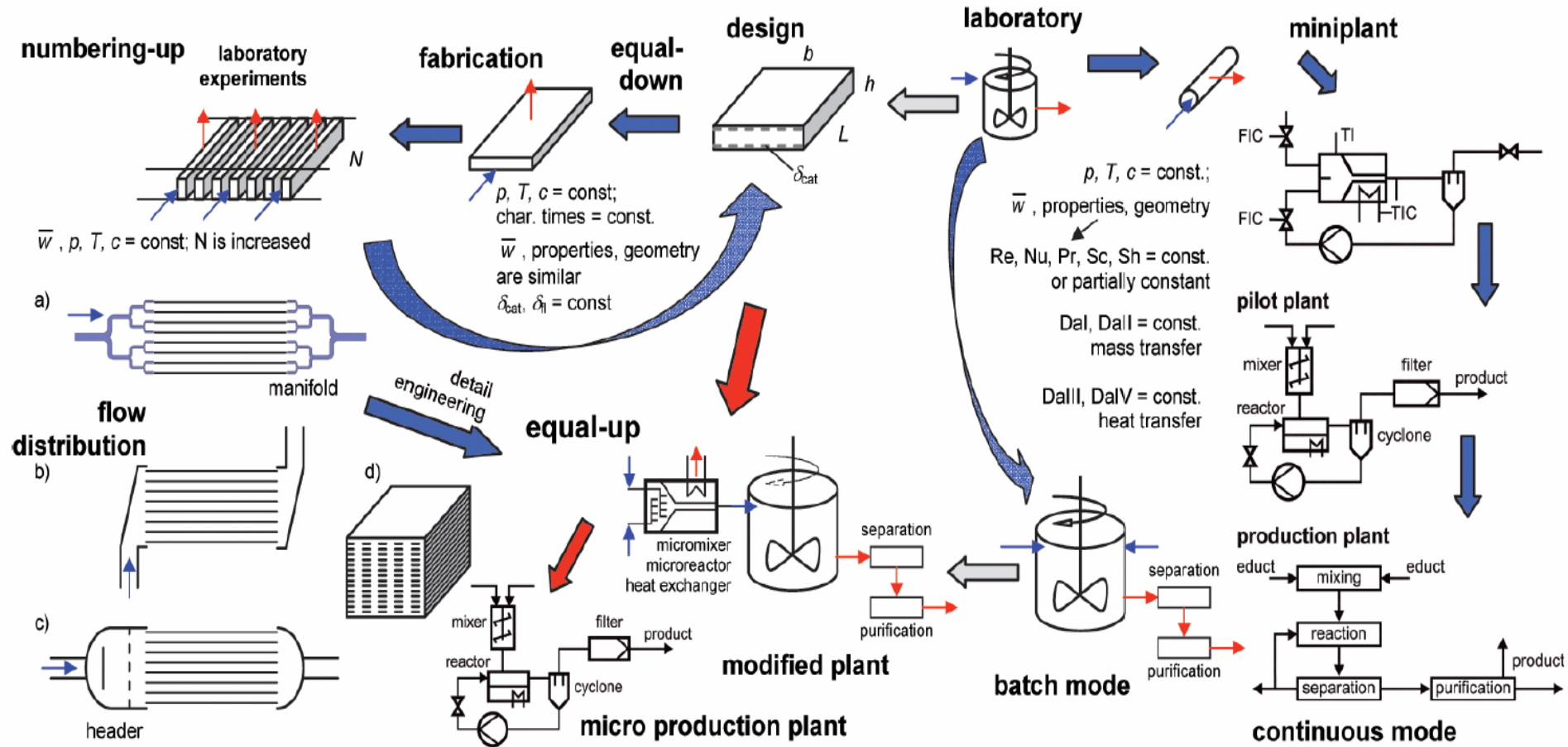
Experimental experience and proper integration of microstructured elements in a conventional apparatus are essential in order to design and fabricate this plate stack.

The equal-up process starts from the process or product to be realized, and the main effects and parameters are identified for miniaturization.

equal-up or scale-out

microstructured devices

scale-up



Equal-up and scale-up procedures with microstructured devices<sup>27</sup>

These key parameters to the production design are with respect to key geometries, fluid dynamics, mixing, reaction kinetics, and heat management.

The experimental results indicate, which dimensions cause the benefits of the small length scale (e.g. the boundary layer  $\delta_{fl}$ ).

Other design parameters from fabrication have to be transferred to the production device, such as shape and structure of the active surface , e.g. the catalyst layer thickness  $\delta_{cat}$ .

# Main Issues of Successful Microstructures

## Example: Heat Exchangers

Microstructure devices are excellent tools for laboratory research in many application fields with specific advantages.

### MANUFACTURING

For lab-scale-type devices, single microchannel systems, either manufactured from silicon by semiconductor technologies or made from metals by mechanical micromachining, or wet chemical etching are mainly used for flow characterization, heat transfer, and experimental investigation of chemical reactions.

This led to a variety of microstructure devices suitable for several applications, namely heat exchange, evaporation, mixing, generation of emulsions, and running chemical reactions in the lab-scale range.

To achieve higher mass fluxes, parallelization of single microchannel devices, scaling up of the microchannel dimensions, or generation of internally parallelized multichannel systems, (equaling-up) are commonly used.

## PROCESS PARAMETER RESTRICTION

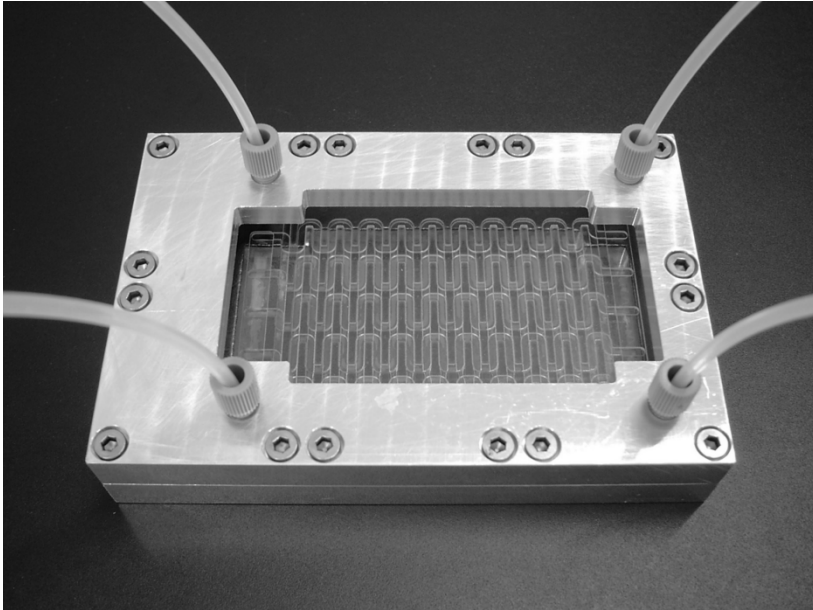
### Material Choice –

Temperature resistance, corrosion resistance, and thermal properties. **Thermal Stress / Corrosion?**

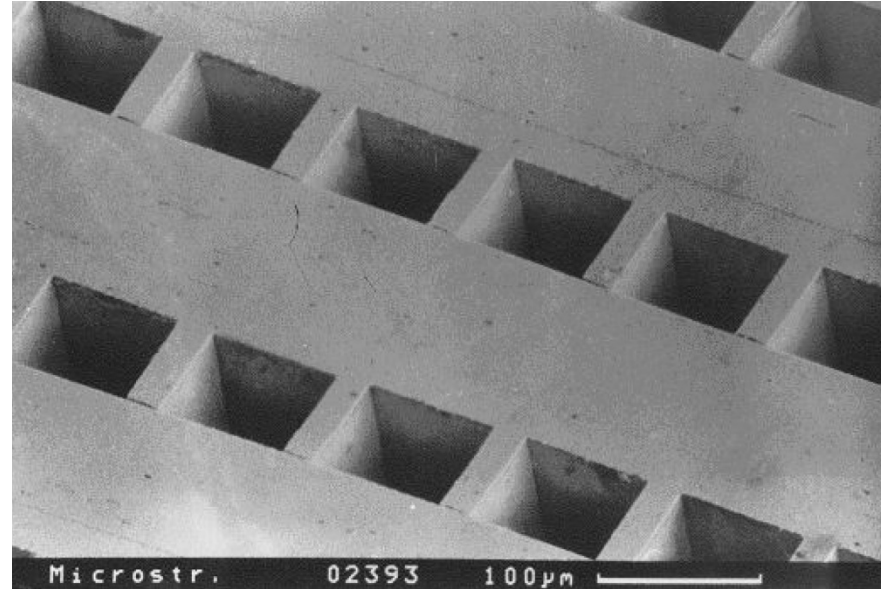
### Fluid Dynamics Restrictions

Laminar/turbulent, fouling, cleaning - use surface coatings, ultrasound?

# Microstructured Devices



Temperature-controlled residence time module. This glass microstructure device is used to adjust the residence time of a chemical reaction mixture to obtain good performance

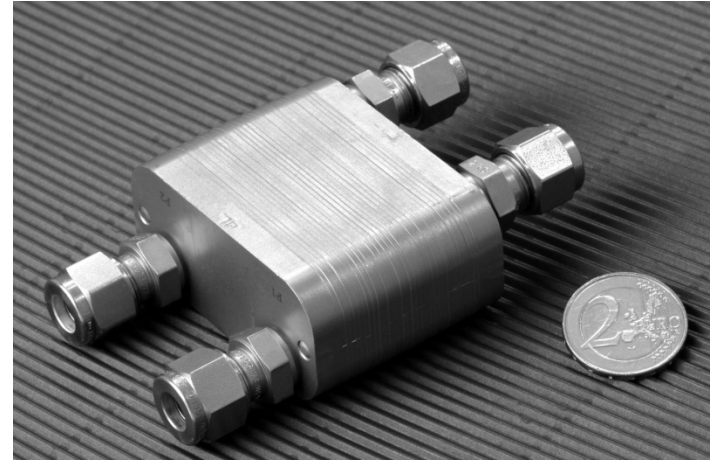


Crossflow-arrangement of mechanically machined microchannel foils. The foils are made of stainless steel and connected by diffusion bonding to form a stable stack.

# Microstructured Heat Exchangers

Microstructured heat exchangers deliver high transfer rates, but they also have drawbacks, application limits, during design and operation.

1. Wall heat conductivity
2. Pressure loss, manifold design
3. Corrosion, fouling, and catalyst deactivation



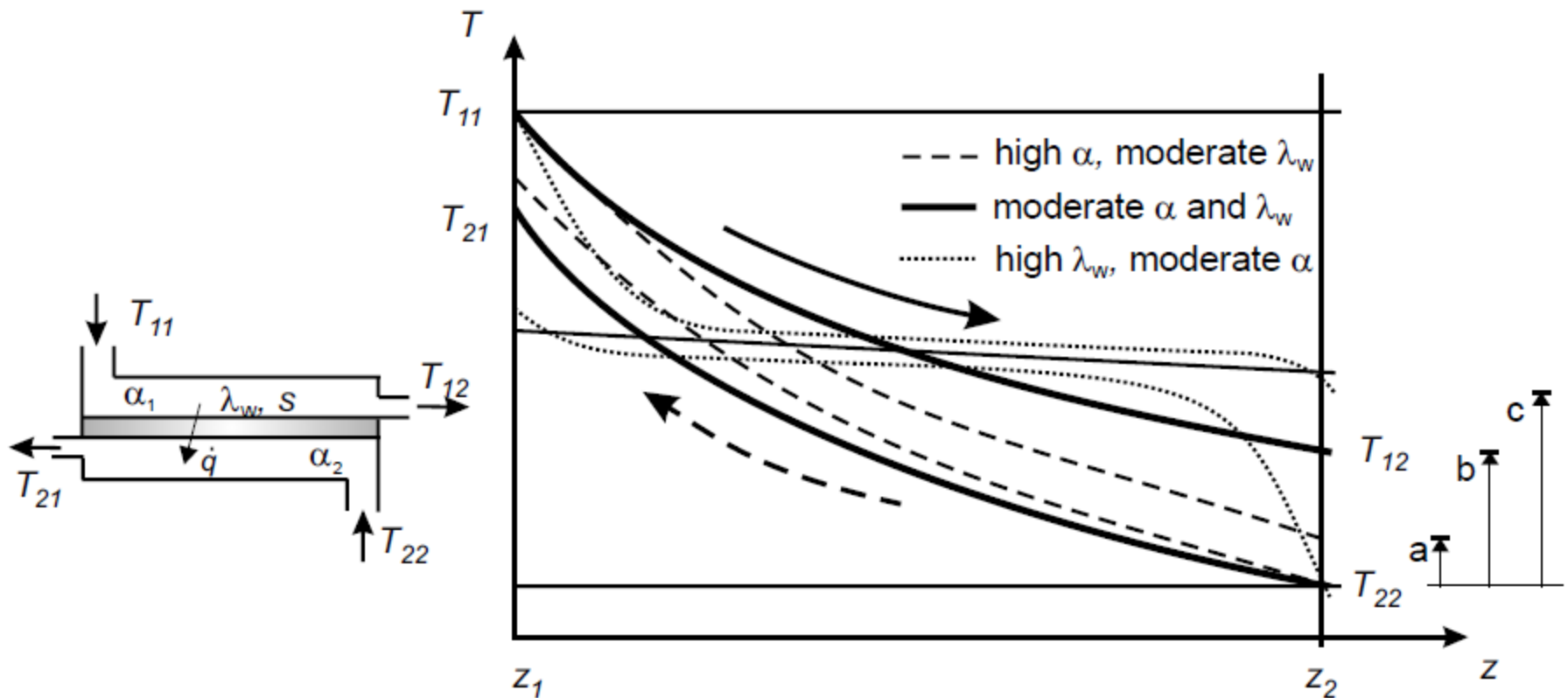
Stainless steel counterflow  
microstructured heat exchanger



## 1. Wall heat conductivity

The ratio of wall thickness to channel diameter is relatively high in microchannel devices, hence, a considerable amount of heat is transferred through the wall parallel to the flow direction, **which lowers the driving temperature difference and decreases the amount of the transferred heat.**

The amount of **parasitic heat flux** has to be considered for highly conductive wall materials like copper, alumina or silicon, and for a low heat capacity flow of the transfer media, such as gases or low flow velocities.



Heat transfer coefficient -  $\alpha$ , wall heat conductivity -  $\lambda_w$ .

Temperature profile along a counter-flow heat exchanger with different heat transfer coefficients  $\alpha$  and wall heat conductivity  $\lambda_w$ . **The fine-dotted line displays the temperature profile in a microstructured heat exchanger with high heat conductivity in the wall and low overall heat transfer coefficient.** The temperature difference at the end of the heat exchanger corresponds to the heat exchanger efficiency

# Three cases

- moderate axial wall heat conductivity with high convective heat transfer (case a),
- moderate axial wall heat conductivity with moderate convective heat transfer (case b), and
- high axial wall heat conductivity with moderate or even high convective heat transfer (case c).

The first case a) exhibits the best heat exchanger efficiency displayed as the temperature difference at the entrance or outlet of the heat exchanger.

The last case, with high axial heat transfer, also exhibits high heat transfer rates at both ends of the heat exchanger, however, in the middle of the device, only a marginal amount of heat is transferred. The temperatures of both fluids are almost identical in the middle of the device with little temperature change.

The area in the middle of the heat exchanger provides inefficient heat transfer and diminishes the device performance.

### 3. Corrosion, fouling, and catalyst deactivation

#### Surface roughness may play a dominant role in microchannels.

If the surface elements reach far into the channel, the hydraulic diameter is constricted, which increases the pressure loss and leads to an earlier transition to turbulent flow.

To include the surface roughness  $\varepsilon$ , a constricted hydraulic diameter  $d_{cf}$  is defined with the narrowest gap width instead of the mean inner distance  $d_t$  of the channel walls.

$$d_{cf} = d_t - 2\varepsilon$$

The corrected hydraulic diameter explains quite well the observed phenomena in rough microstructured channels

Corrosion becomes relevant in microchannels where the surface roughness influences the flow behavior and the transport characteristics. A corrosion layer of 100  $\mu\text{m}$  is tolerable in conventional systems, but may be fatal for microchannels.

Particle generation and processing are major steps in many applications. Particles may attach to the wall, decrease the cross section, and influence the pressure loss and flow velocity. The particles sticking to the wall may attract more particles and lead to fouling and blocking of the channels.

Heterogeneous catalysis with high exothermic or endothermic character are suitable, but catalyst deactivation or poisoning can be critical for long-term operation

## 2. Pressure loss

For high flow rates, small channels induce a high pressure loss due to the high surface-to-volume ratio.

Hence, low viscosity fluids are preferred for application in microchannels due to a tolerable pressure loss.

The pressure loss in a channel – from Bernoulli equation:

$$\Delta p_{12} = p_1 - p_2 + \frac{\rho}{2} (w_1^2 - w_2^2) + g (y_1 - y_2)$$

In microchannels, the gravitation force is negligible compared to friction forces

$$\Delta p_{12} = p_{tot,1} - p_{tot,2} \quad \text{with} \quad p_{tot} = p + \frac{\rho}{2} w^2$$

The pressure loss is calculated for a complex channel arrangement as:

$$\Delta p = \left( \lambda \frac{l}{d_h} + \zeta \right) \frac{\rho}{2} \overline{w}_{ref}^2$$

$\overline{w}_{ref}$  is the constant reference velocity,  $\lambda$  is the channel friction factor.

The pressure loss consists of portions from straight channels described by  $\lambda$  ( $l/d_h$ ) and portions from bends, curves, connections, and other internals described by  $\zeta$ .

The pressure loss coefficient  $\zeta$  is primarily defined for turbulent flow in devices and can be found in textbooks

In general, the flow below  $Re = 10$  can be regarded as straight laminar flow where no vortices appear and the pressure loss coefficient  $\zeta$  can be neglected.

For high  $Re$  numbers, especially for  $Re > Re_{crit}$ , the laminar contribution can be neglected.

In the transition regimes,  $10 < Re < Re_{crit}$ , a square fit of laminar and turbulent values can serve as a first estimation for the pressure loss.



hydraulic diameter  $d_h = 4A/lp$

For laminar flow in long straight channels the channel friction factor  $\lambda$  is inversely proportional to the Re number in the channel

$$\lambda = \frac{C_f}{\text{Re}} = \frac{C_f \eta}{\rho d_h w}$$

The channel friction factor  $\lambda$  is inversely proportional to the channel diameter  $d_h$ ; smaller channel increases the friction factor.

The pressure loss in a channel can be calculated from

$$\Delta p = \left( C_f \nu \frac{l}{d_h} + \zeta d_h \overline{w} \right) \frac{\rho}{2} \frac{\overline{w}}{d_h} = C_f \frac{\eta l}{2 d_h^2} \overline{w} + \zeta \frac{\rho}{2} \overline{w}^2$$

In long straight channels, the pressure loss  $\Delta p$  depends mainly on the first term, hence

the pressure loss varies

- almost linearly with the velocity, channel length, and viscosity,
- inversely proportional to the square of the hydraulic diameter  $d_h$ .

In curved channels with internals, the hydraulic diameter influences the pressure loss only marginally, but convective effects determine the pressure loss coefficient  $\zeta$ .

The pressure loss can also be described as

$$\Delta p = \left( C_f \frac{l}{d_h} + \zeta \text{Re} \right) \frac{\rho v^2}{2} \frac{\text{Re}}{d_h^2}$$

The pressure loss depends on Re number with a linear and a quadratic part.

With decreasing device length dimensions, the pressure loss is nearly proportional to the square of the Re number.

The volume flow rate  $V$  through  $N$  nearly rectangular, parallel channels -

$$V \propto N \left( d_h^2 \overline{w} \right)$$

The final form of pressure drop for a system of  $N$  channels:

$$\Delta p = \left( C_f \eta l + \zeta \frac{V}{N} \right) \frac{\rho}{2} \frac{V}{N d_h^4}$$

This equation only accounts for **parallel channels with a single flow manifold.**

$$\Delta p = \left( C_f \eta l + \zeta \frac{V}{N} \right) \frac{\rho}{2} \frac{V}{N d_h^4}$$

The pressure loss consists of two parts,

1. the laminar part ( $C_f$ ) varies linearly on the volume flow rate.
2. The convective part of internals ( $\zeta$ ) depends on the square of the volume flow rate.

The hydraulic diameter plays a crucial role with the inverse dependency of the fourth power.

Decreasing the channel diameter and the resulting higher pressure loss can be balanced by either increasing the channel number or decreasing the channel length or volume flow rate.

$$\Delta p = \left( C_f \eta l + \zeta \frac{V}{N} \right) \frac{\rho}{2} \frac{V}{N d_h^4}$$

The pressure drop increases linearly with velocity and viscosity is less important in short, curved channels with many internals.

Within short microchannels, not only is the pressure loss low, but also precipitation or polymerization of various chemicals are possible due to proper mixing of the immiscible reactants.

However,

For gas-flow microreactors with long, straight channels, the pressure loss is approx. 2.5 times lower than in fixed bed reactors for the same mass transfer conditions

The key parameters of the production device, including channel height  $h$  or wall thickness  $s$ , are equal to the laboratory equipment,

however,

the geometry of the active surface or the fabrication techniques may differ, for example, the microchannels have the same cross section or only the same height, but differ in length.

## Application Examples - Recent Experiments on heat Sinks

Requirement of extremely high heat removal rates

- phase change heat transfer
- minimum temperature difference

This is particularly true in flow boiling applications, where only modest increases in device temperature will allow the removal of very large heat fluxes.

In the design of applications, such as microchannel two-phase flow heat sinks and micro thermal pumps, **the liquid/vapor two-phase flow pattern is critical.**

<http://www.rpi.edu/tphtl/research/lvfp/lvfp.html>



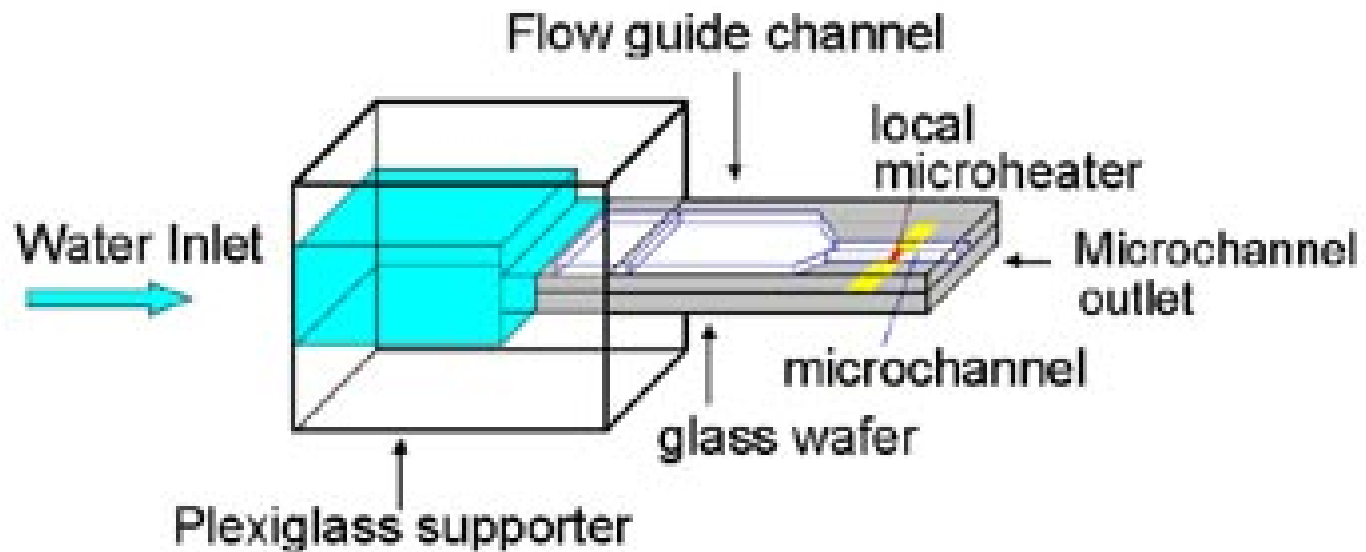
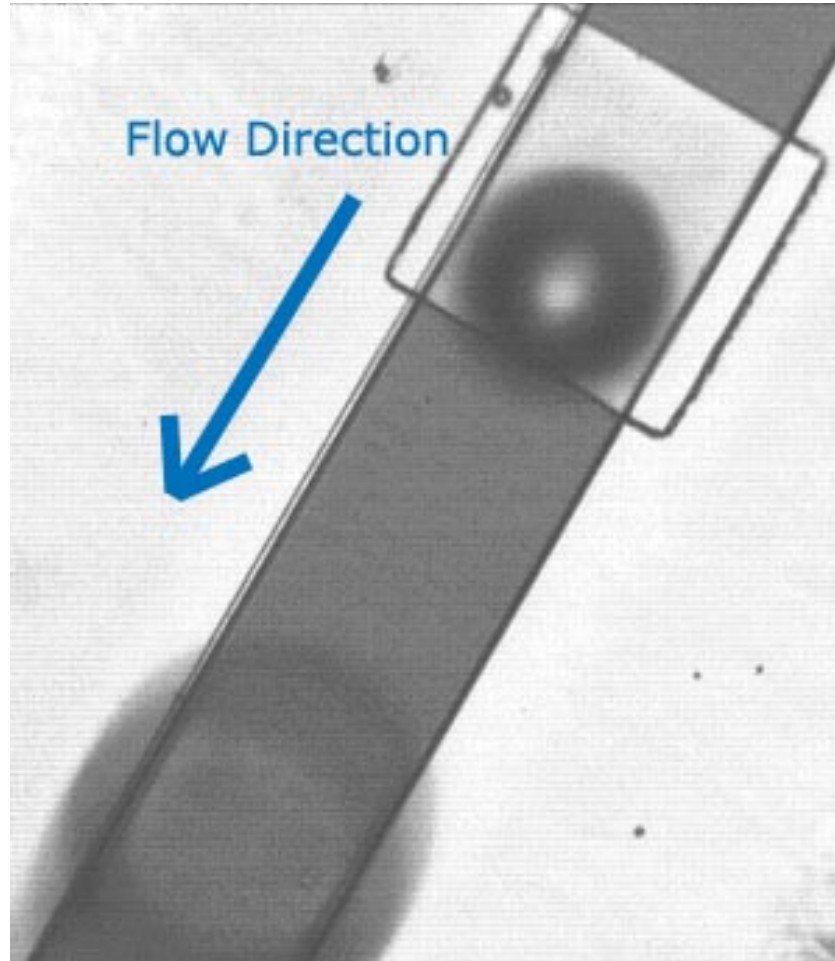


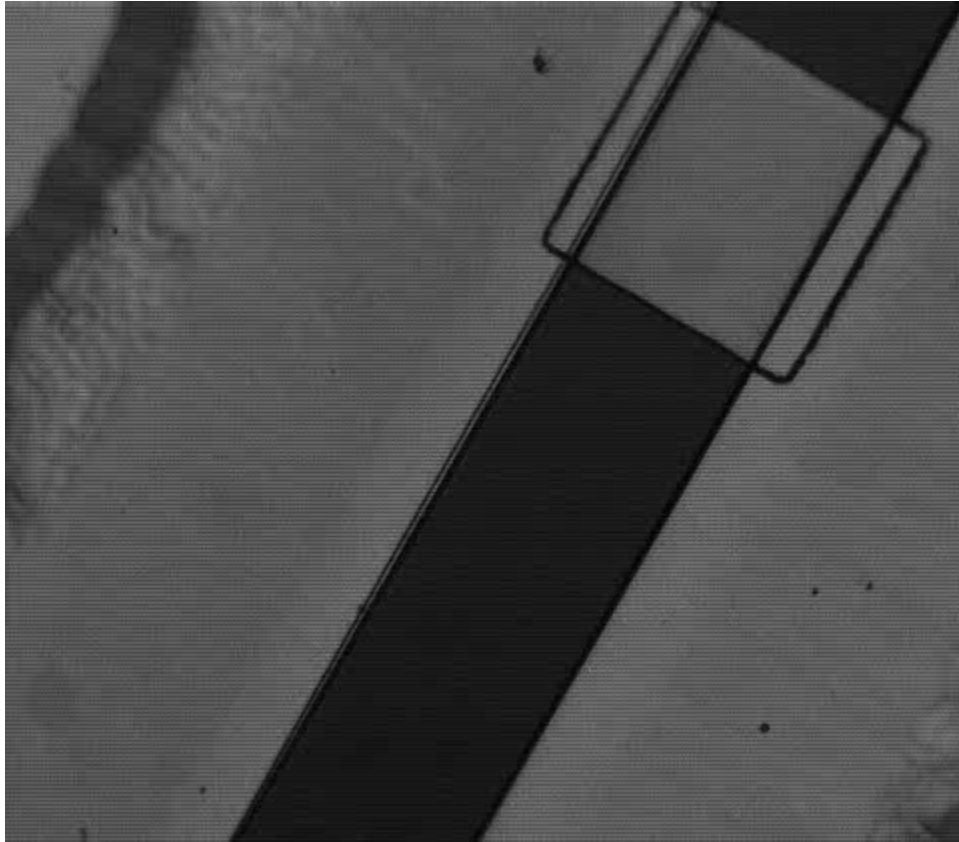
Fig.1 - Configuration of the experimental specimen and test section

- Microscale platinum heater fabricated on a Pyrex glass wafer
- Shallow trapezoidal microchannel with hydraulic diameter  $D_h = 56\mu\text{m}$
- Use high-speed digital CCD video camera and microscope,
- Study the two-phase flow patterns,

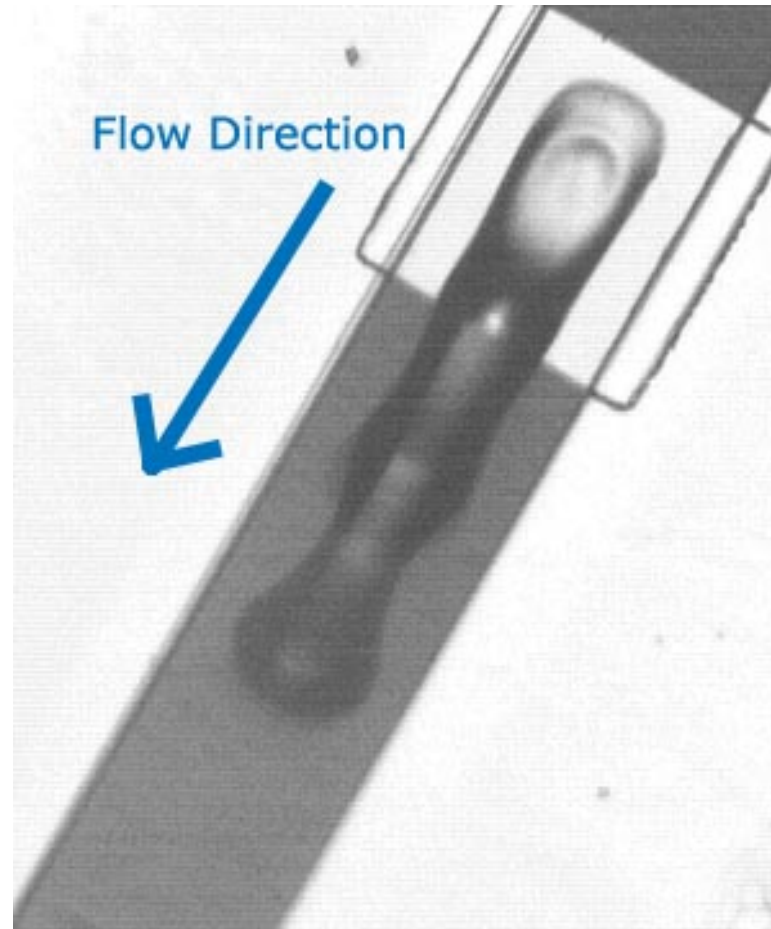
Impact of the size of the microchannel and the mass flow rate on the boiling incipience and two-phase flow patterns are analyzed.



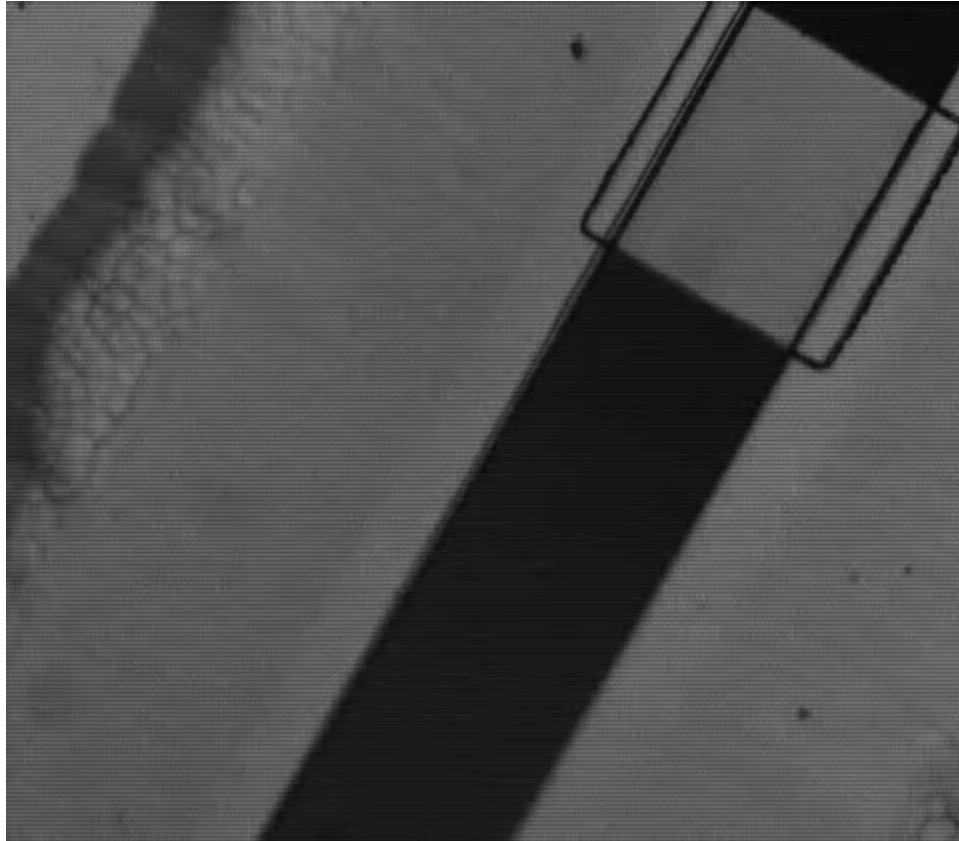
Bubbly flow in the microchannel under small flow rates and low power



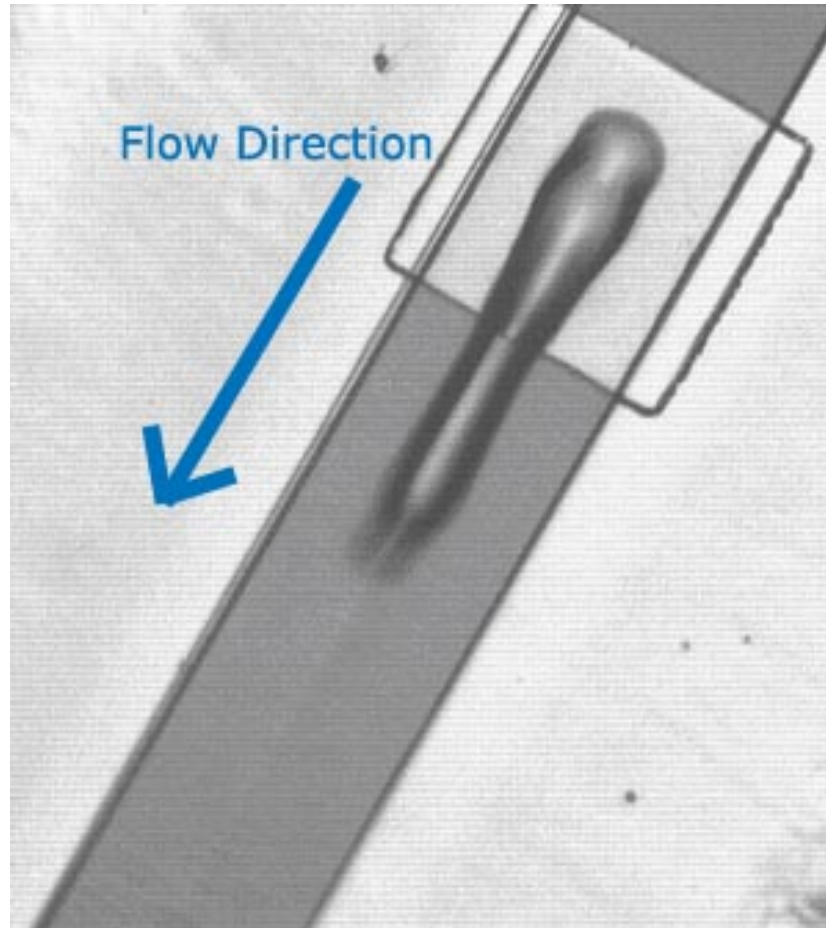
**Bubbly Flow**



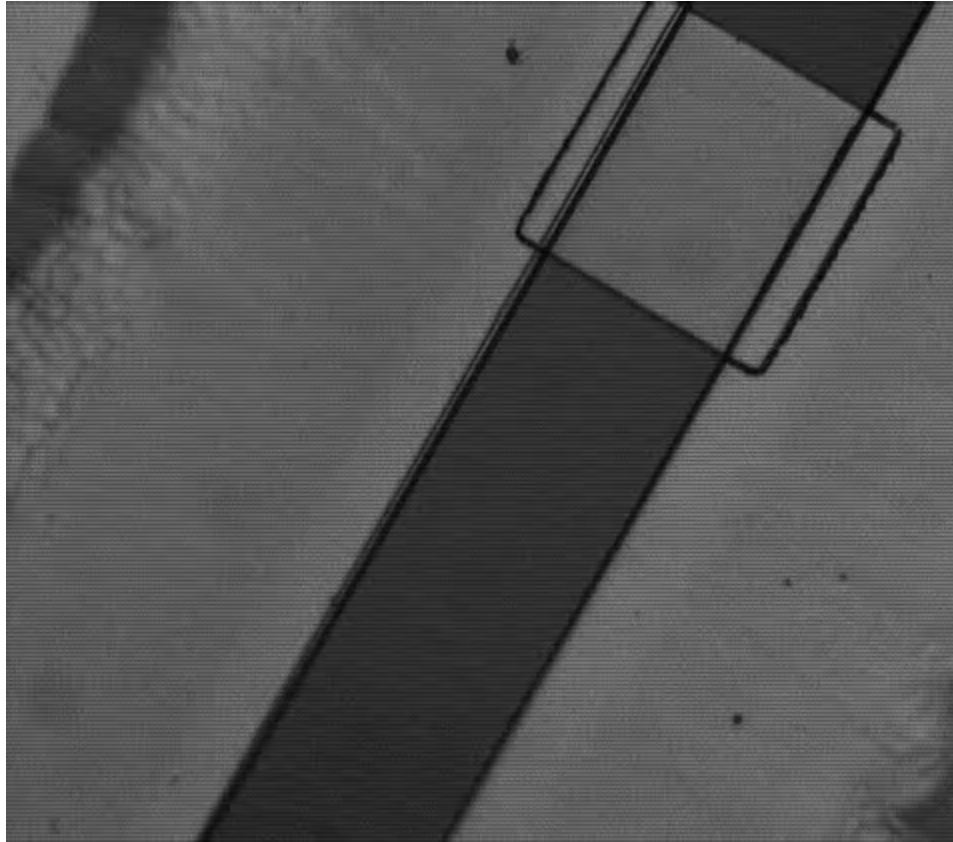
Wavy flow in the microchannel under moderate mass flow rate and moderate power



**Wavy Flow**



Annular Flow in the microchannel under large flow rates and high power



**Annular Flow**

**A better understanding of the fundamental phenomena occurring in these types of flows**

**Effects of surface geometry/modifications/properties/heat flux**

Microchannel devices present many *opportunities*

however,

their *disadvantages and special requirements* must be taken into consideration for successful application.