New Directions in Reactor Design Through Miniaturization

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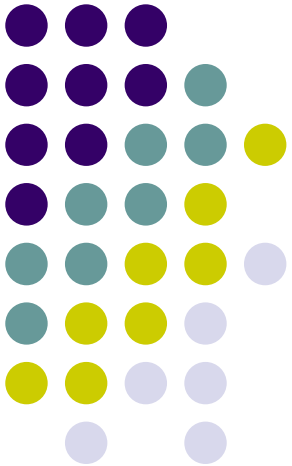
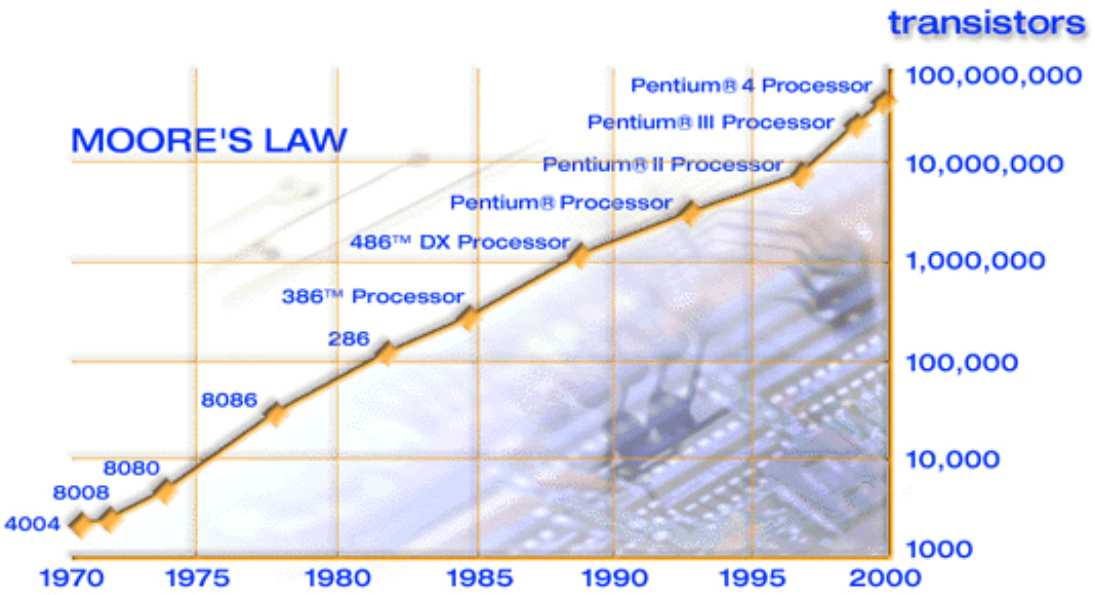
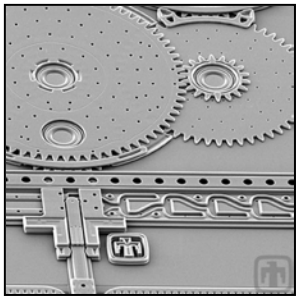
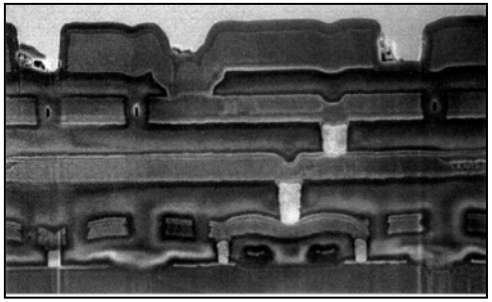


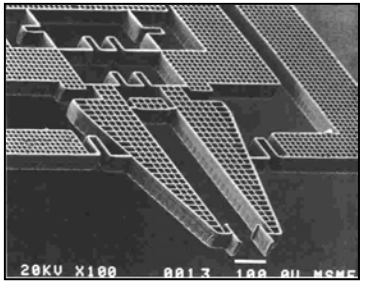
Miniaturization Progress

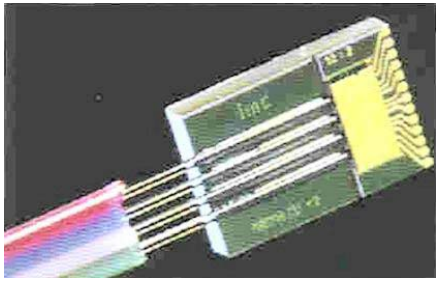
Microelectronics to MEMS



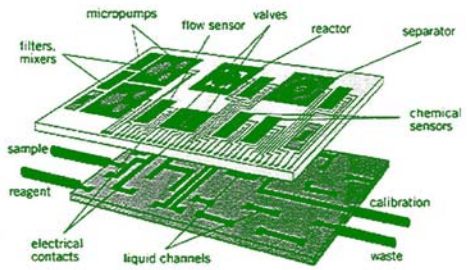








Microchemical Systems



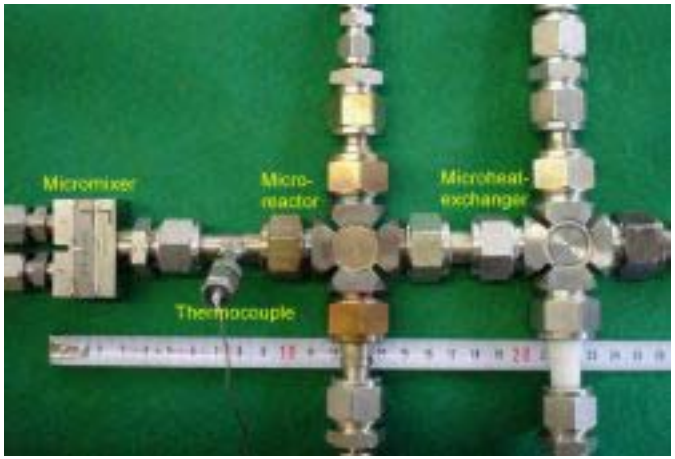
Miniature reaction and other unit operations, possessing ***specific advantages*** over conventional chemical systems

Outline

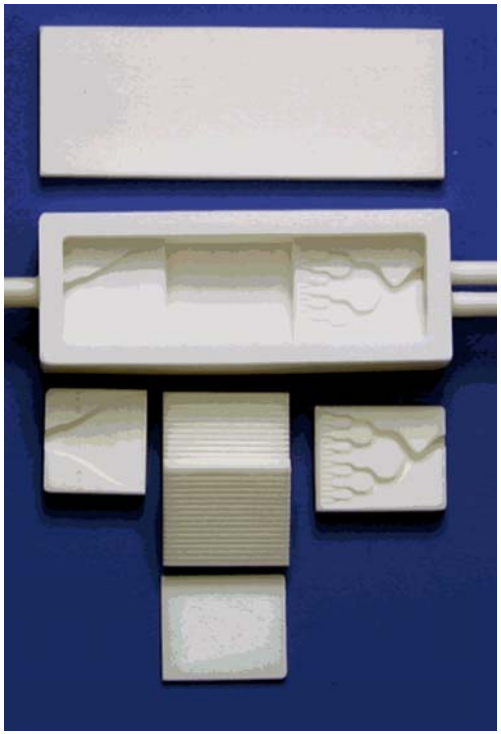
- Microreactors—What?
- Benefits of Miniaturization—Why?
- Examples of Microreactors: Fabrication, Characterization, and Reaction—How?
- Conclusions

Microreactors—What Are They?

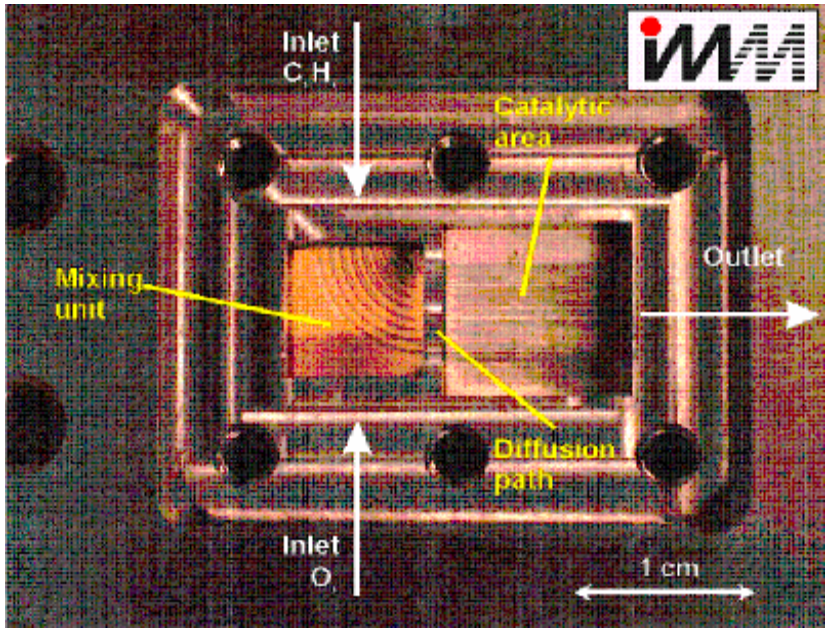
- Not your mother's microreactor
- At least 10X smaller than benchtop "microreactor" of the past



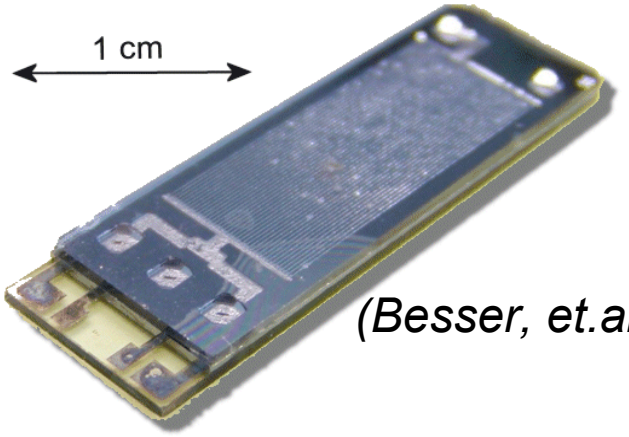
(Forschungszentrum **Karlsruhe** GmbH)



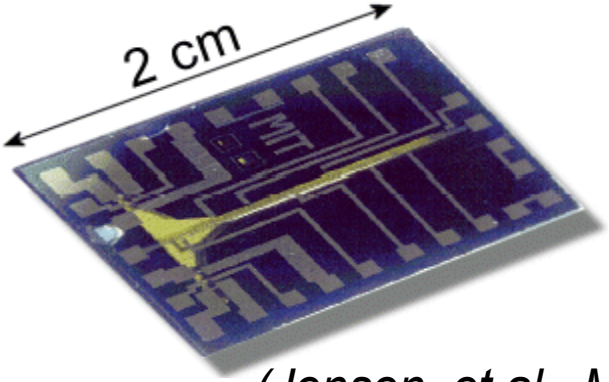
Microreactors—What Are They?



(Ehrfeld, et.al., IMM)

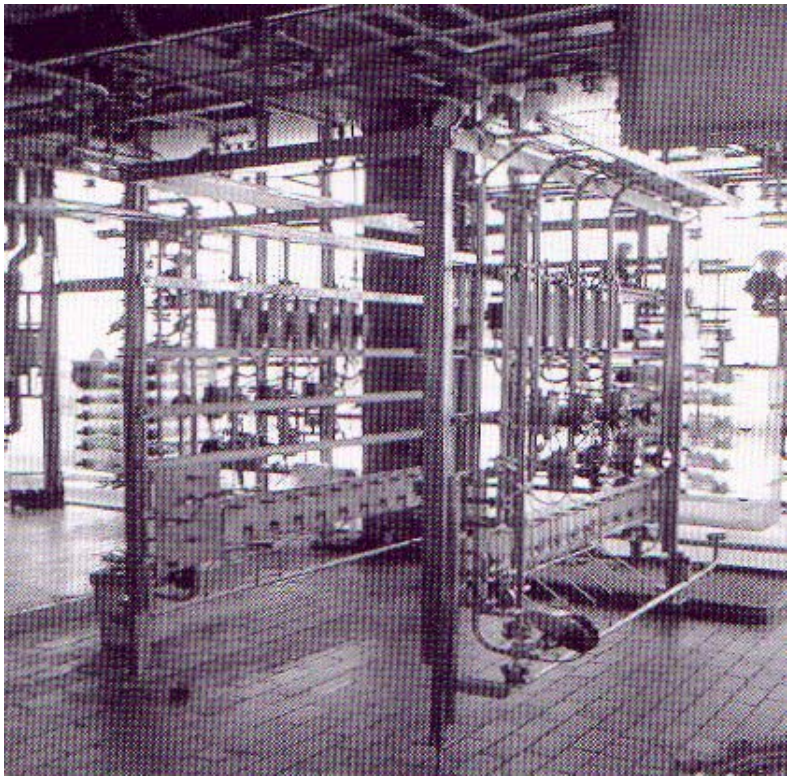


(Besser, et.al., IfM)



(Jensen, et.al., MIT)

Integration-Industrial Processes

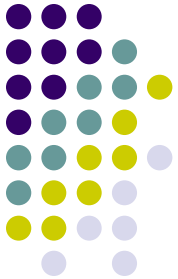


Input flows must be divided and reduced

Output flows must be combined

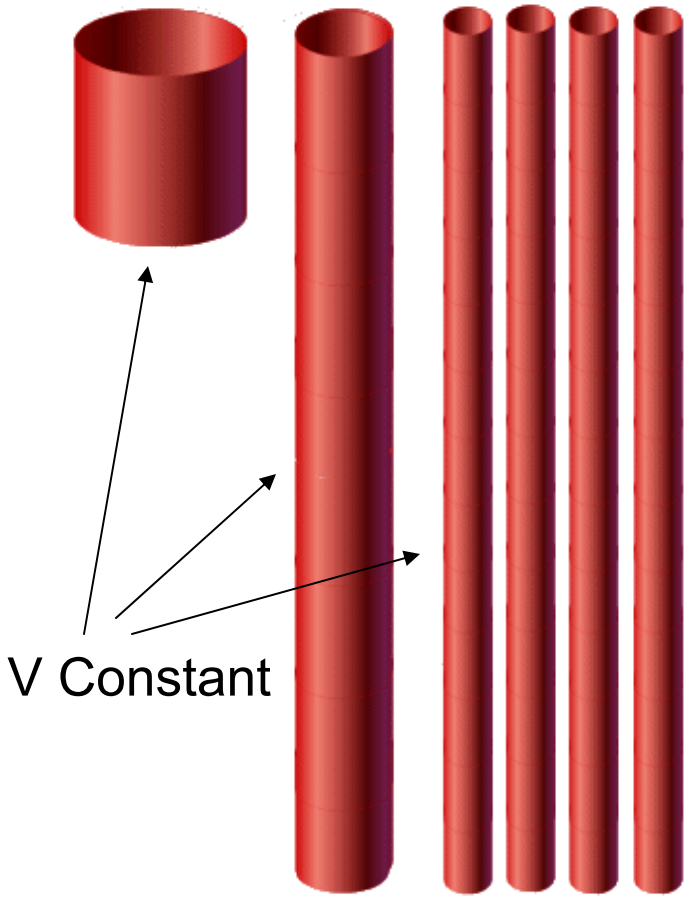
(Merck production plant using micromixers)

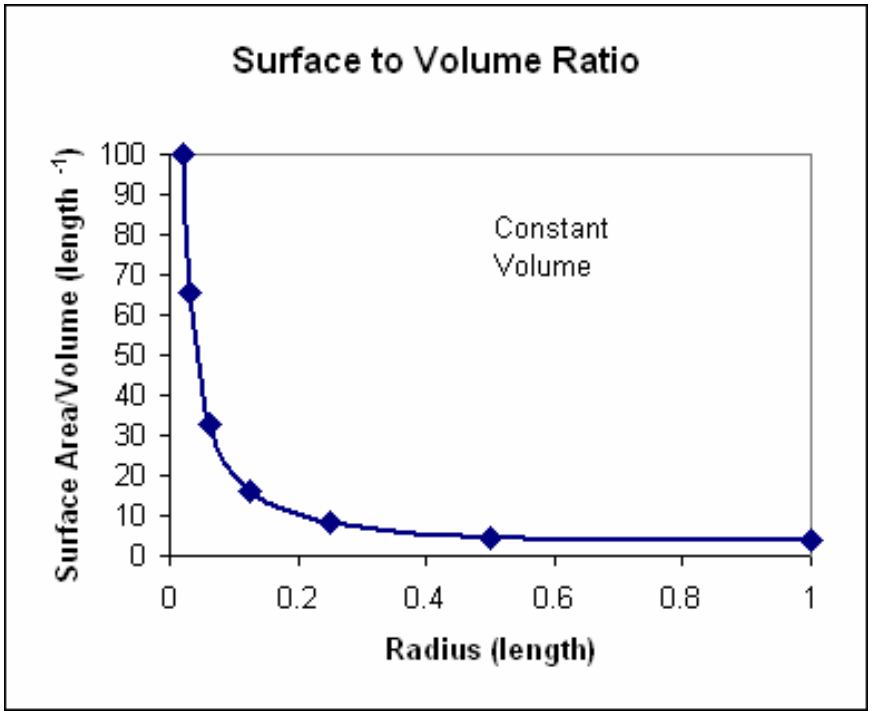
Benefits of Miniaturization—Why?



- Surface to Volume Ratio
- Low Inventory (“Hold Up”)
- Residence Time Distribution
- Low Transport Resistances
- Robust Materials
- Cost

Benefits: Surface to Volume



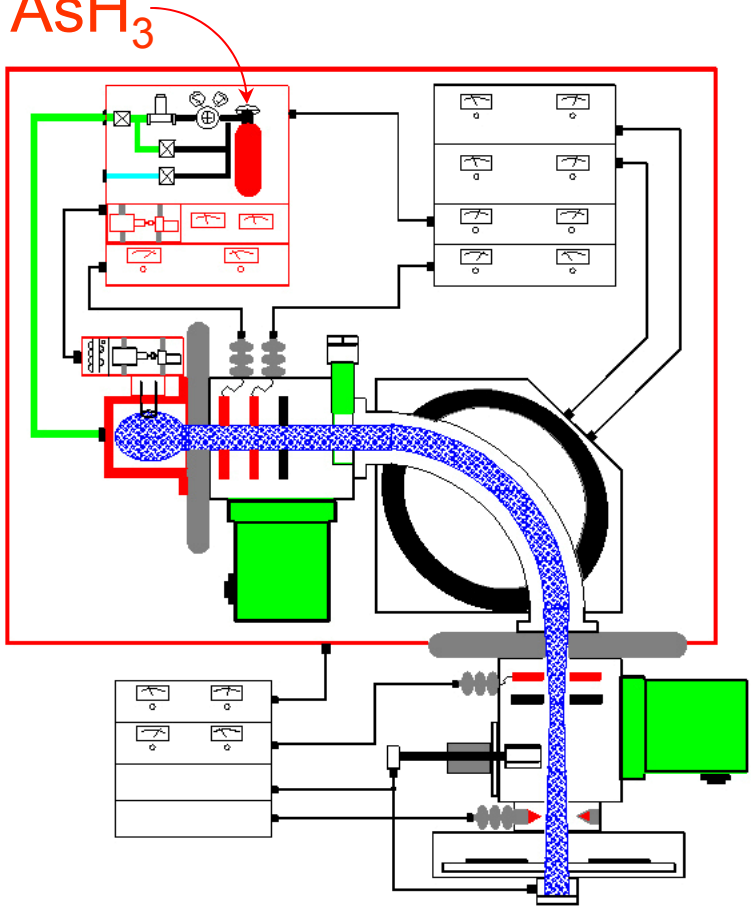


Heat Management

Surface Reaction

Explosion-Safe

Benefits: Low-Inventory (Hold-Up)



Schematic of As^+ Ion Implanter

Phosgene Reactor, Geismar, LA

- Safety
- Environment

Benefits: Residence Time Distribution

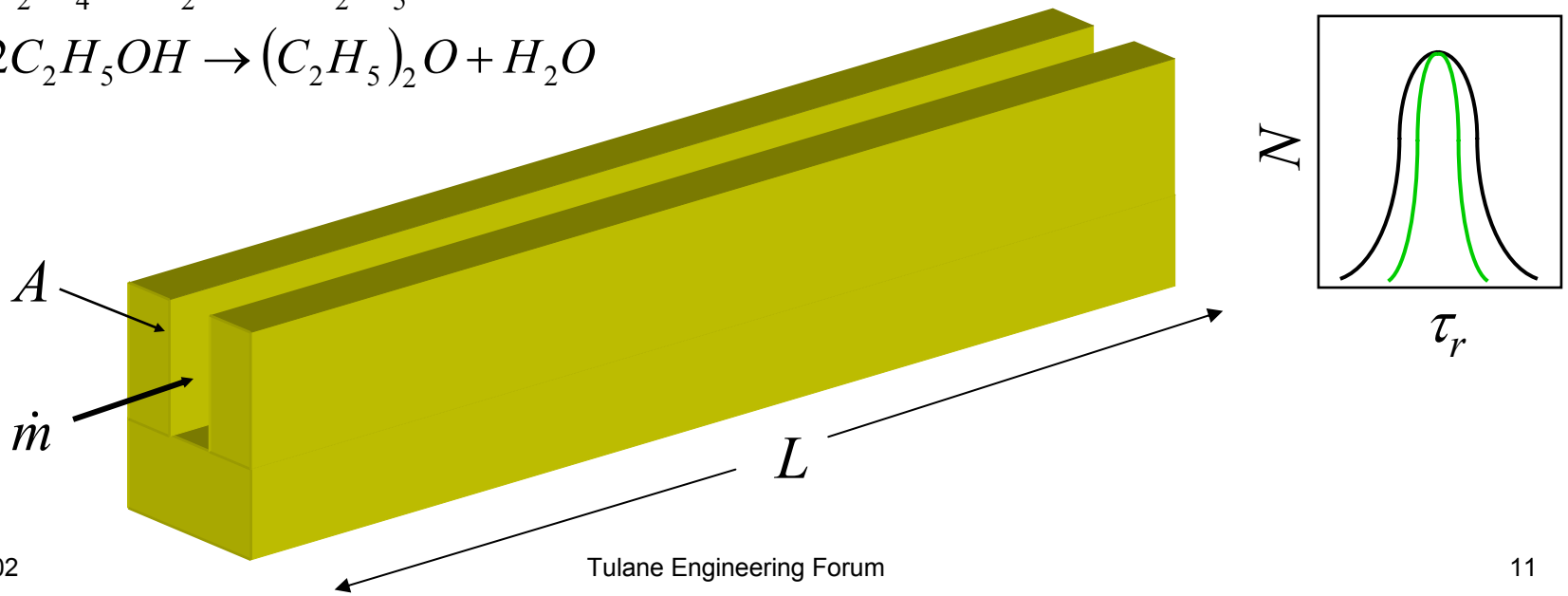
<u>Precise Control Over Geometry</u>

Tuning of residence time

Improved selectivity

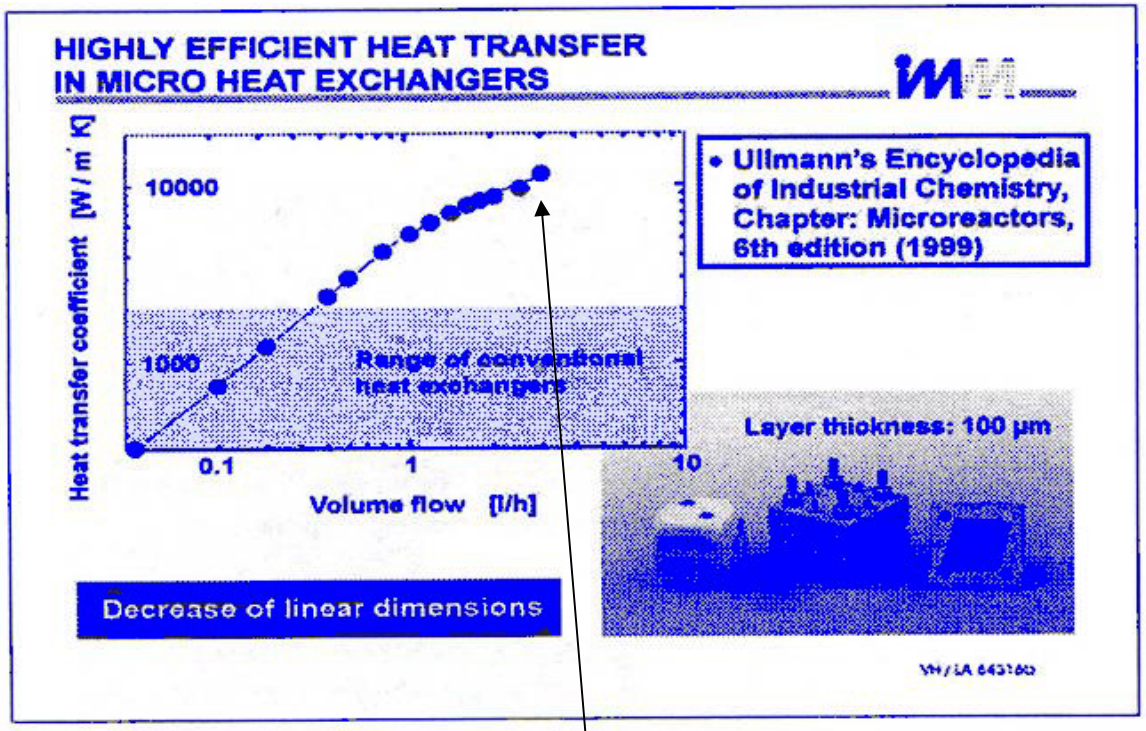
$$C_2H_4 + H_2O \rightarrow C_2H_5OH$$

$$2C_2H_5OH \rightarrow (C_2H_5)_2O + H_2O$$



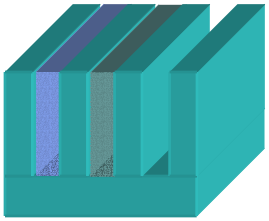
Benefits: Low Transport Resistances

Overall Heat Transfer Coefficient



(AIChE)

U = 25,000 W/m^{2}K

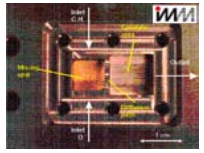


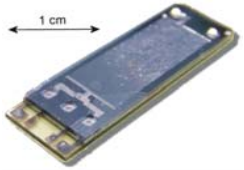
$$q''_x = -k\frac{dT}{dx}$$

(conduction)

Benefits: Robust Materials

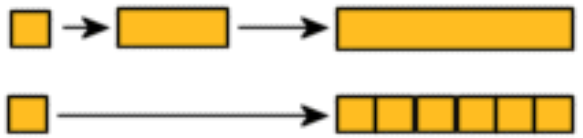
- High strength, high melting point materials:
  - Metals
  - Ceramics
  - Silicon
- Array of fabrication processes (MEMS technology)
- Non-traditional reactor materials
  - Polymers



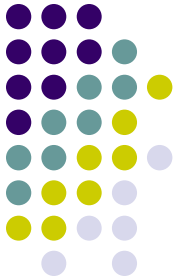


Benefits: Cost

- Reactor Fabrication
  - High volume batch
    - Si integrated circuit fabrication model
    - Metal/ceramic micromachining techniques
    - Interface of reactor to plant (?)
- Scale-Up Process
  - Linear process
  - Characterize unit module; scale up throughput by addition of modules

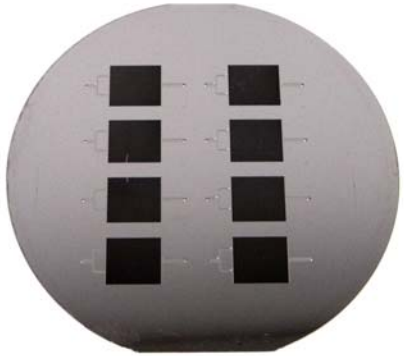


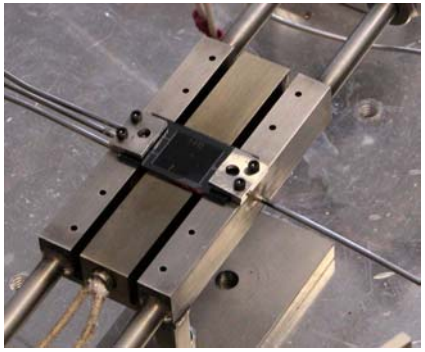
Microreactor Example—How?

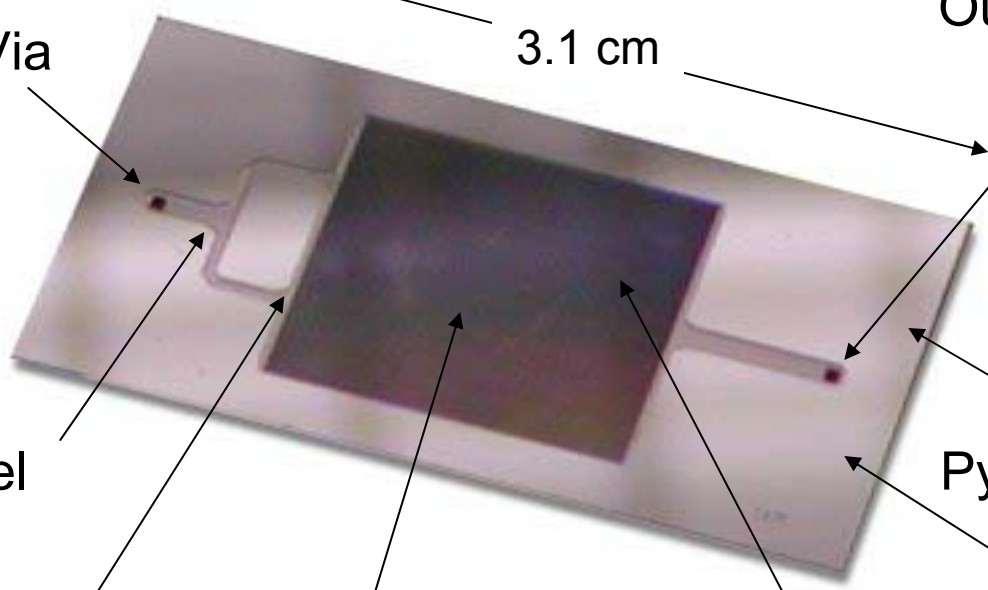


- Reactor Fabrication
- Reactor Characterization
- Reaction Results

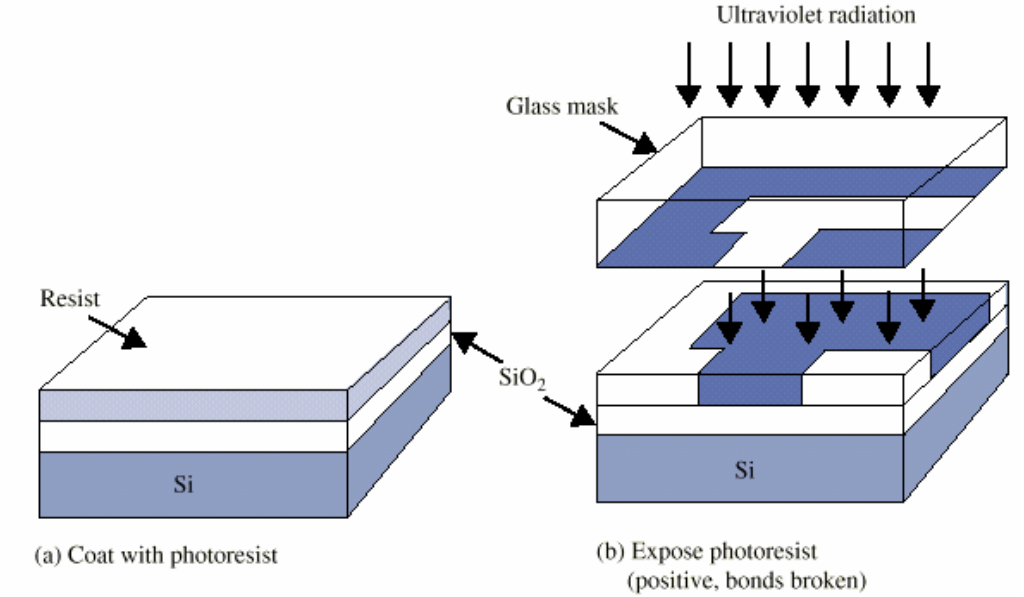
Microreactor Device





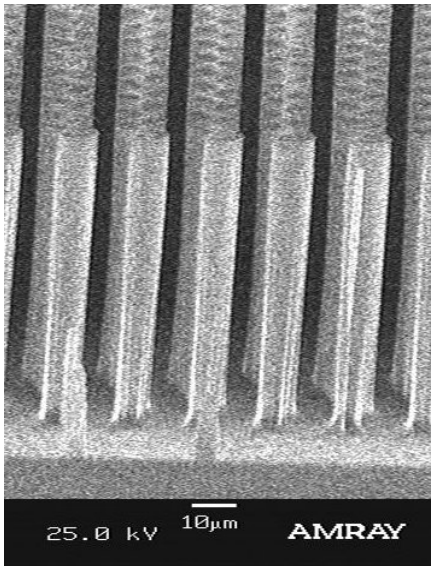


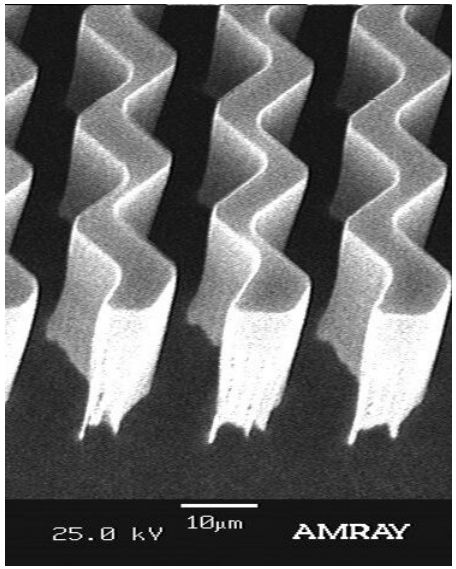
Fabrication: Photolithography



(Shackelford)

Fabrication: Silicon Etching

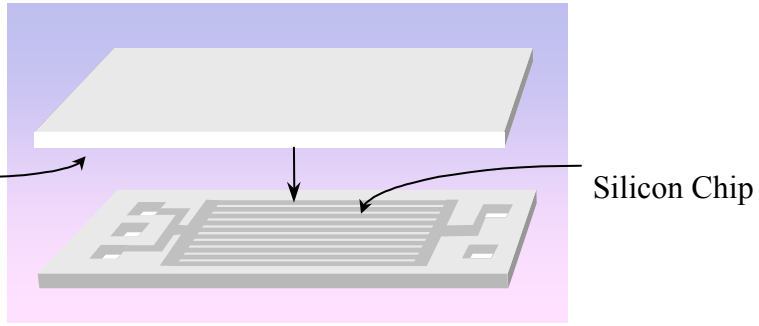


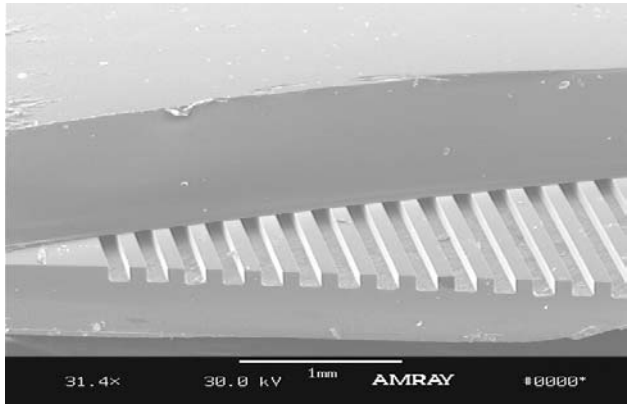


Structured Catalyst Support
in Reaction Zone

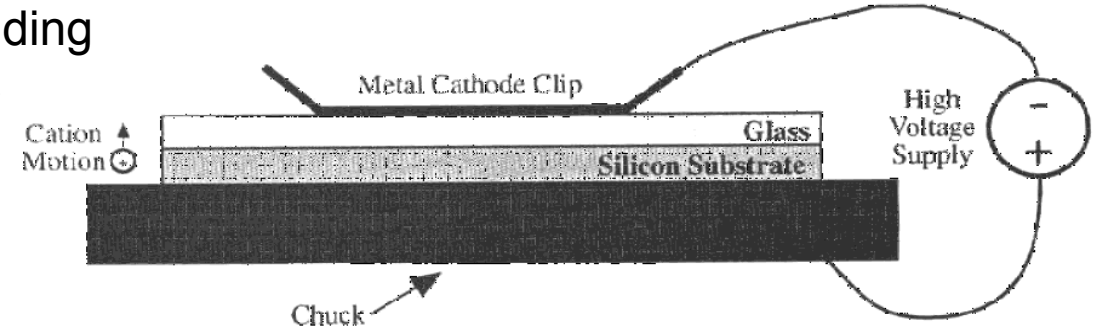
Alcatel Deep Reactive
Ion Etch System

Pyrex-to-Silicon Bonding





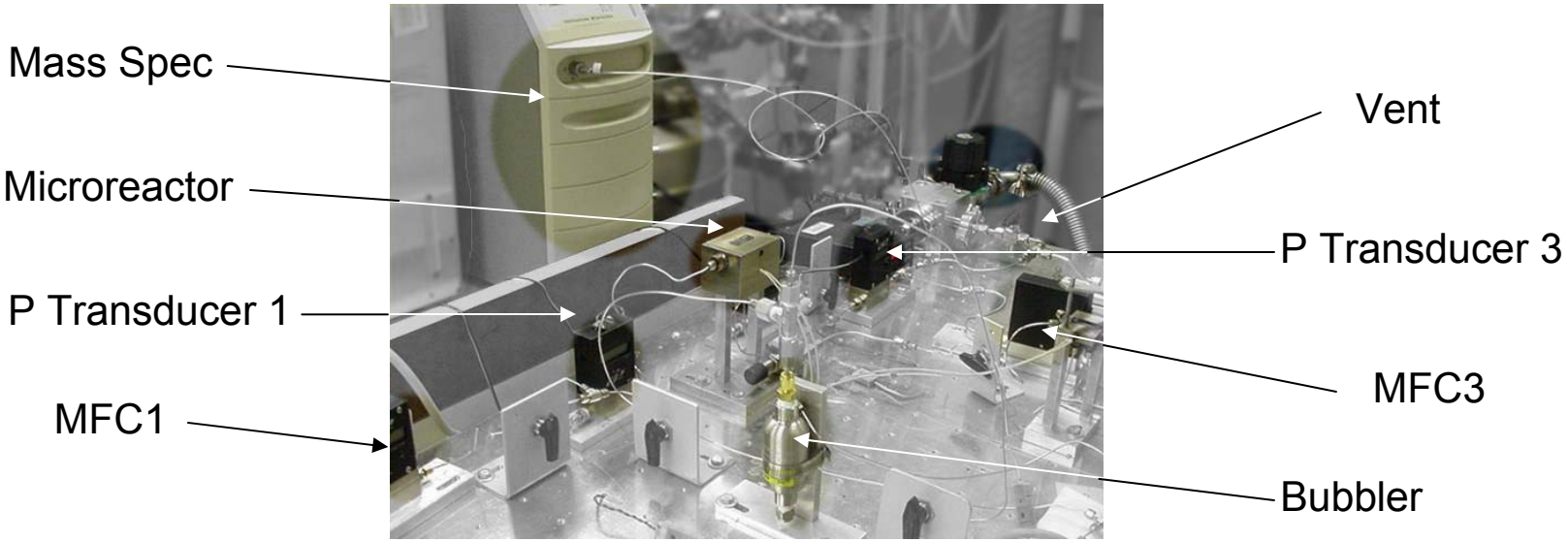
Anodic Bonding

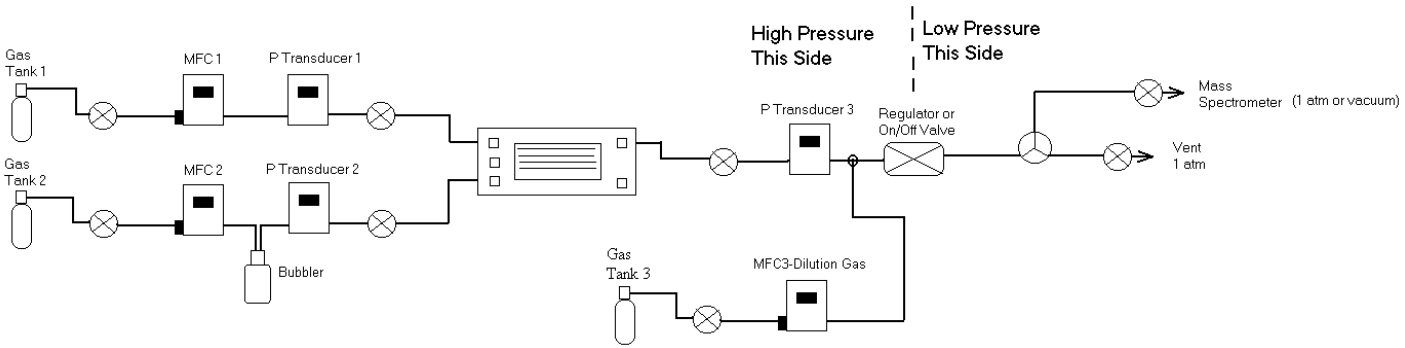


(750 VDC, 450°C)

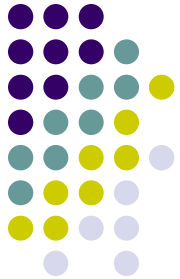
(Kovacs)

Characterization Experiment Setup



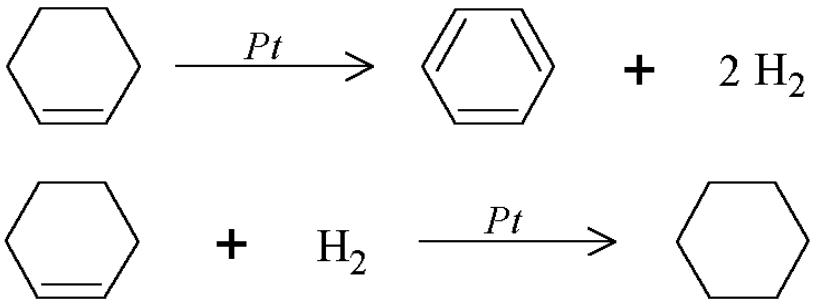


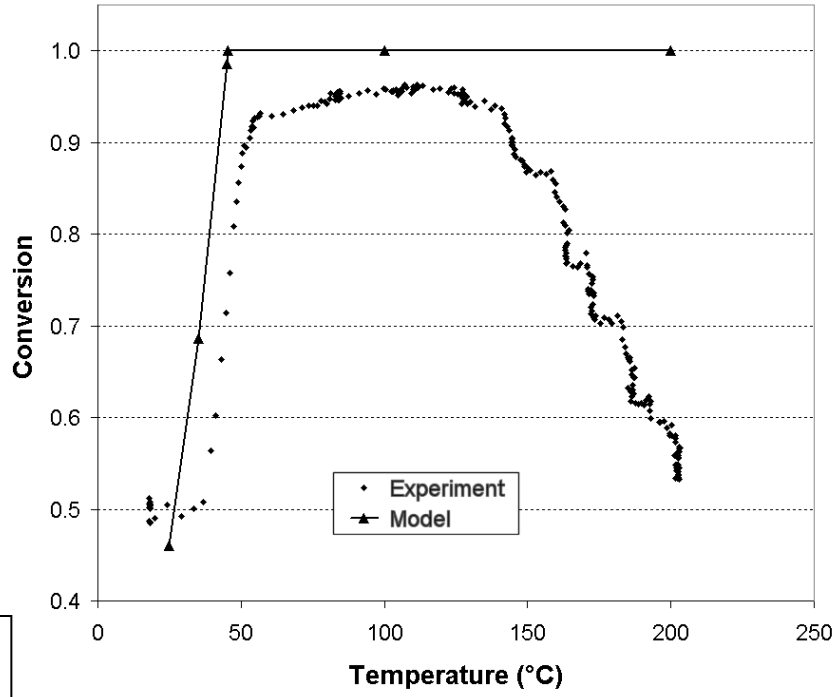
Examples: Reactions



- Hydrocarbon hydrogenation/dehydrogenation
  - Cyclohexene hydrogenation/dehydrogenation
  - Benzene hydrogenation
- Hydrogen + oxygen in explosive regime
- Syngas conversion
- Catalytic combustion

Cyclohexene Hydrogenation/Dehydrogenation

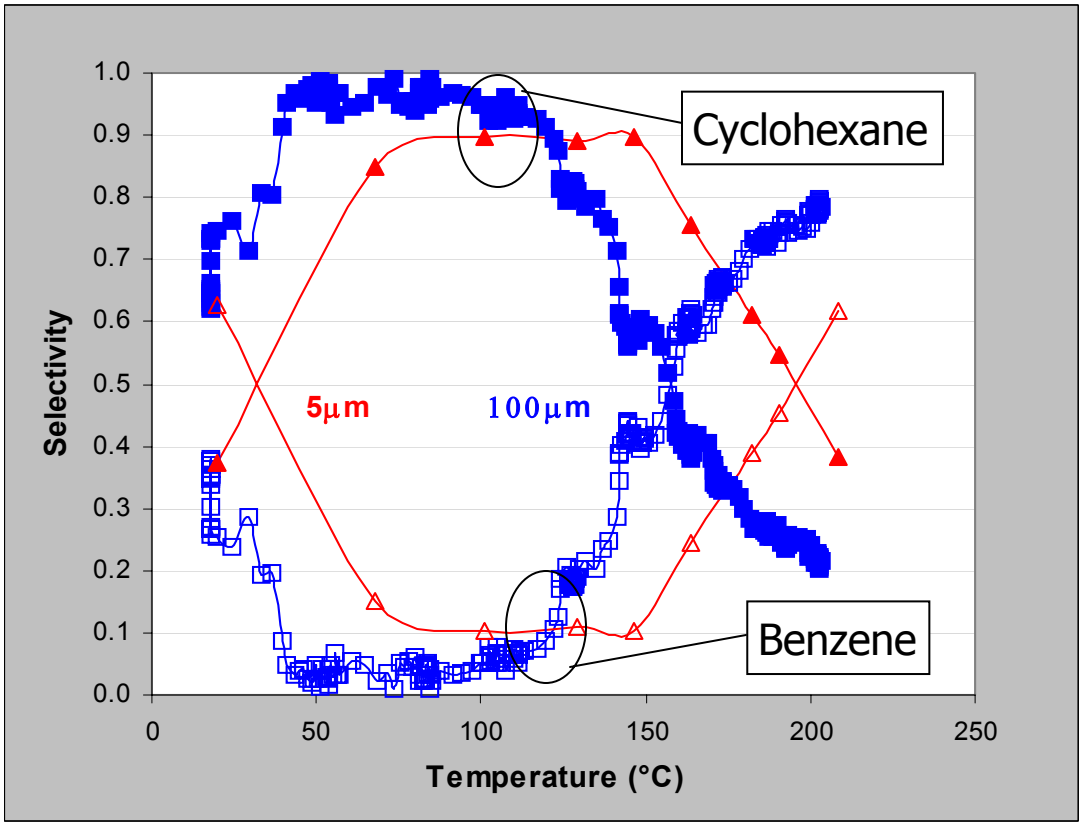




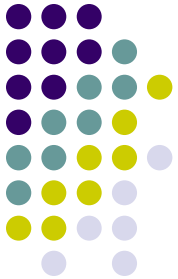
Models for hydrotreating and reforming reactions

Effect of Temperature on Selectivity

- Room temperature activity for both products
- Hydrogenation favored T_{room} to 150°C
- Dehydrogenation favored above 150°C
- Time and temperature dependent deactivation
- 5 μm reactor more tolerant of T and t

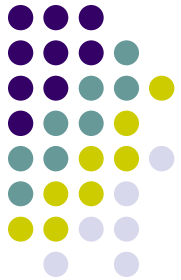


Implementation of Microchemical Systems



Technology	Application
• Arrays for parallel characterization (R&D)	• Catalyst discovery; process development
• On-site, on-demand production	• Toxics
	• Fuel processing (fuel cells)
• Special environments	• Space; offshore platforms
• New factory paradigm	• Highly selective synthesis (pharma, fine chemicals)
	• DOE Vision 2020-30% reduction in waste, pollution

Conclusions



- Microreactors possess special properties due to their **small dimensions** (< 500 μm).
- Various choices of **robust materials** are available suitable for a variety of applications (metal, ceramic, silicon, polymer).
- **Silicon** microreactor example illustrates reactor fabrication, operation, and characterization.
- ***Model hydrocarbon catalytic hydrogenation and dehydrogenation*** reactions illustrate ability to take relevant reaction engineering data safely and with low consumption.
- Microreaction technology will find a number of **niches** in analytical and process chemistry in the new millenium.

Acknowledgements

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