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28. međunarodni sajam i kongres za hemijsko inženjerstvo, zaštitu okoline i biotehnologiju

U Frankfurtu je u periodu od 15. do 19. maja 2006. godine, održan 28. međunarodni sajam i kongres za hemijsko inženjerstvo, zaštitu okoline i biotehnologiju, ACHEMA 2006. Nemačko društvo za hemijsko inženjerstvo i biotehnologiju, DECHEMA (www.dechema.de) prati ovaj sajam i kongres, objavljivanjem izvešaja o pravcima razvoja novih proizvoda i tehnologija. Kao i ranijih godina, počev od prošlog broja (Hemijska industrija, vol. 60, no. 5–6) ovi izveštaji, u nešto skraćem obliku, objavljuju se i u našem časopisu.

PROCESS INTENSIFICATION: PROBING AND MINIMIZING THE LIMITATIONS

Today, the absolute size of a system or piece of equipment is not the only issue. The real goal of process intensification is to increase the space/time yield, improve selectivity and reduce overall production costs.

A reduction in size undoubtedly remains an important strategy in the process intensification toolkit (micro process technology). However, the hardware, apparatus or any other component is not the primary consideration. The real attention is focused on the functions, for example heat exchange as a unit of operation rather than on the heat exchanger per se. Essentially, the goal is to identify the limitations of conventional material and heat transport systems and then circumvent these limitations.

Better, faster, safer

A number of different strategies are combined under the heading "process intensification", making a precise definition elusive. The term can be used to describe a preference for multi–purpose systems or small dedicated–lines which can be duplicated so that capacity can be ramped up as needed (numbering up).

Process intensification can also refer to completely new types of reactors and techniques which can substantially improve space/time yield and reduce the costs associated with reactors and pipes. Typical examples from the field of polymerization engineering include micro reactors, rotating–disc reactors, kneader reactors and similar screw–based machines as well as spray polymerization. There are also methods which are based on ionic or catalytic polymerization mechanisms, where polymerization speed is inherently higher. Process integration based on reactive distillation or reactive chromatography also appear to offer significant potential.

It is, however, difficult to distinguish between "process intensification", "process optimization" and "process integration." How do you differentiate between "micro reactor technology", "micro process technology", etc.? Actually, there is a clear distinction. Process intensification is a strategy, and the other two terms belong more in the tool category (hardware). The important thing is to get educts and products to the right place at the right time and to efficiently control the heat and material transport.

"Intensification means revolution rather than evolution," explained Henrik Hahn who works at Degussa. He and his team are not trying to alleviate the familiar bottlenecks. Instead, they are aiming for a quantum leap which will significantly shift process efficiency in the direction of the theoretical maximum or that will take the process in an entirely new direction.

Otto Machhammer from BASF sees differences in approach. Process intensification strategies vary depending on how aggressive the goals are. If a reactor has to be as small as possible, then a micro reactor can be the answer. If the goal is business driven, then the strategy must address the total process or situation including raw materials, energy flows, staffing requirements and logistics issues. The problem will be defined differently, and an interdisciplinary effort is the key to success.

IMM (Institute for Microtechnology in Mainz) uses a more concrete definition. According to IMM, process intensification refers to a process which is

- better (higher yield and/or selectivity)
- faster (enhanced space/time yield)
- safer and better for the environment (green chemistry with no high–risk process systems)
- more cost effective (lower investment costs and/or total operational costs).

A new technological approach will be required which encompasses everything from equipment and a deeper understanding of the process to process automation. Possible strategies include micro process technology, intensification of heat and material transfer, non–traditional methods of energy input, new approaches to process control and a reduction in the number of process steps by integrating reaction and product preparation.

"We need new, unconventional strategies for process intensification – revolution instead of evolution," declared Martin Strohrmann from BASF. "The engineers cannot solve these problems on their own. We have to delve deeper into the basics and strengthen our networks with universities, professional bodies and partners in the EU."

Numbering–up better than scaling–up

What actually happens? Dimensions shrink down to the millimeter or micrometer range. Size minimization is accompanied by a very significant intensification of heat and material transfer. The surface to volume ratio, the specific phase boundary, rises in micro–structured equipment to several thousand m^2/m^3. For example, micro heat exchangers which are no larger than a cube of sugar can handle the energy of an entire one–family home.

The new numbering up approach eliminates the risks which are associated with scaling up, and this gives process intensification a significant advantage. Micro systems are often run in parallel using the optimal parameters which were identified in the lab. Multi–scale systems are an extension of this approach. In the field of reactor technology, the traditional approach was to adapt the chemical process to the equipment. For example, the walls were used for temperature control, but this becomes increasingly difficult as equipment dimensions continue to increase. Now the equipment can be adapted to the chemical process, so that the full potential of a chemical reaction can be exploited. Process intensification will be the solution of choice for reactions which involve intensive mixing or which are very endothermic or exothermic. The chemical reactions can take place without any limitations on heat and material transport.

Last, but not least, safety aspects are not insignificant, because smaller volumes of reactants are easier to handle and control.

No alternative to an interdisciplinary approach

The "Multi–Phase Flow, Material Transport Process and Reactor Development" working group at the University of Bremen's Environmental Engineering Institute shows very clearly that "process intensification" is an interdisciplinary effort which encompasses a whole range of technology and research fields. The working group is currently working on the development of complete, fully functional micro reactor systems which are designed to replace entire processes.

The working group's partners use micro metallic powder injection molding (Fraunhofer IFAM, Bremen) to produce components. The also supply silicon etch processes (IMTEK Microsystems Technology Institute, University of Freiburg; IMSAS, University of Bremen), develop μ–MSR technology (BIAS Bremen Institute for Applied Radiation Technology) and perform system integration (Schulz–Systemtechnik, Visbek).

In its micro powder molding injection process (μ–MIM), Fraunhofer IFAM uses very fine particles (< 5 μm) to mold complex shapes. The powder is mixed with a special binder system and then injected into a tool or mold insert. Techniques such as silicon etching, micro machining, micro erosion, laser machining and LIGA are used to make the reusable mold inserts.

IFAM has used μ–MIM on a number of different materials including stainless steel, iron, hard alloy, copper and tungsten–copper. Small structures (10 μm) with an aspect ratio of 16 (height to width) can be produced. Additional development goals include:

- large–surface forming of micro structures,
- volume production of micro components,
- achievement of closer tolerances in component dimensions,
- reduced surface roughness,
- development of further materials for use in micro system technology.

Micro–scale mixers, heat exchangers, pumps and reactors are already available at IMTEK. The micro devices, which have flow channels in the low μm to mm range, are examples of a significant scale down which influences material characteristics and the transport processes. Miniaturization has a number of advantages including:

- high gradients for pulse, heat and material exchange,
- good control of process parameters due to smaller volumes and shorter paths,
- high integration of process units with each other and with instrumentation components.

Process Intensification "Project House"

Degussa will invest 15 million euro over the course of three years in its process intensification project to conduct research into new process strategies and reactor designs. The team will be looking to develop process strategies in three areas: "highly active catalysts", "functional materials" and "disperse systems".

The fourth area, "Chemical ExplorENG" (Exploring Chemical Engineering), acts at the glue which holds the project together. The focus here is on development of modular systems which are used to produce special chemicals. Various modules can be built simultaneously and "plugged together" on site. This reduces the time is takes to get a line up and running, and it reduces time to market. Modular design can also save money when capacity needs to be ramped up. This is a big advantage when demand is low at the time of product introduction.

The "highly active catalysts" group plans to use micro process technology and a new reactor design to significantly improve gas synthesis processes which make use of heterogeneous catalysts. The project team will benefit from the results of the "Demonstration Project to Evaluate Microreaction Technology in Industrial Systems" (DEMiS) which was subsidized by the German government and which Degussa has now brought to a successful conclusion together with Uhde GmbH and participating universities. More active catalysts and new catalyst preparation techniques will be needed to exploit the full potential of microprocess technology.

The goal of "functional materials" research is to find new ways of encapsulating solids, polymerizing water–insoluble monomers and producing ultra–fine organic particles. The team is looking at products like adhesives that can be activated and impact modifiers. The team intends to use mini emulsions, which have super fine droplets with a narrow size distribution and diameters in the 110–100 nm range, as the vehicle. This type of nano droplet reactor can only be produced by using high specific energy input during the emulsification process. A number of current scientific articles contain an impressive description of how mini emulsions can be produced in a laboratory, but no one has succeeded in implementing the process in a production–scale system.

The "disperse systems" team is exploring ways to reduce process times and thereby increase process efficiency. Research is focused on alternative process paths for the production of color paste and on new reactor designs for intensive fermentation. This could expand the operating range of the traditional continuous stirred tank reactor. The production cultures which are used in fermentation have been continuously improved over the years. The Project House can help capitalize on the progress which the company has made in its culture development activities by improving our reactor technology. One of the major priorities is to develop reactors which improve oxygen input, because this is a major limiting factor during fermentation.

Current practical examples

Example I: On September 27th 2005, six months after the cooperation agreement was signed with IMM Institut für Mikrotechnik Mainz GmbH, the pilot production phase was successfully completed on a micro reactor system which is used to produce nitroglycerin at the Xi'an Chemical Industrial Group (HAC) in China. The line is designed for continuous production of nitroglycerin, and it has a throughput of about 15 kg/h. The nitroglycerin is used exclusively as medication to treat acute angina pectoris attacks. The product has to meet very stringent quality requirements, and it is produced under GMP conditions. The micro reactor has three main parts: a unit to produce nitrating acid from fuming nitric acid and sulphuric acid, the actual micro reactor and the subsystems which are used for phase separation, purification and drying of the synthesized nitroglycerin. The nitrating acid is produced continuously just prior to being fed into the micro reactor, and it reacts directly with the glycerin. The two reactants are mixed continuously, and mixing only takes milliseconds to complete. A large surface–to–volume ratio ensures that reaction heat is dissipated immediately. The low reaction volumes also reduce the potential risks.

The project demonstrates the "classic" advantages of process intensification: higher yield, improved product quality, enhanced safety and lower environmental risk.

Example II: A high–performance reactor, which was developed at the Karlsruhe Research Center, has proven its suitability for chemical production in an industrial environment. DSM Fine Chemicals GmbH in Linz, Austria used the new system to produce more than 300 tons of a high–grade product for the plastics industry in 10 weeks. The yield increased significantly compared to traditional methods. Raw material consumption and waste volumes were reduced, and the micro reactor also enhances process reliability.

The core element in the new production system is a "micro reactor" made of special nickel alloy. The reactor, which is 65 cm long and weighs 290 kg, has a throughput capacity of 1700 kg of liquid chemical per hour. Klaus Schubert, Director of the Institute for Micro Process Technology at the Karlsruhe Research Center, explained that "the term micro refers to what goes on inside the reactor. Chemical substances are brought together in micro blenders and then react in tens of thousands of micro channels. The heat created by the reactions is dissipated via micro channels in a matter of seconds. The micro reactor can handle several hundred kW of heat energy."

The micro reactor at DSM replaces a core reaction step which previously had taken place in a large continuous stirred tank reactor where several thousand tons of toxic, corrosive chemicals were mixed. "The Karlsruhe micro reactor has substantially increased yield compared to our previous process which was purely based on continuous stirred tank reactor production", commented Peter Pöchlauer, Project Manager at DSM Fine Chemicals GmbH in Linz, Austria. "We have been able to reduce raw material consumption and the volume of waste, and this improves our efficiency and reduces the impact on the environment. The micro reactor has also increased process reliability."

Example III: The IMRET series of conferences (International Conference on Microreaction Technology) reflects the rapid pace of development in the field. The conferences, which alternate between Europe and the US, were initiated in 1997 by the micro reaction working group at DECHEMA. Some interesting applications which show progress towards industrial–scale production were described at IMRET 8 which took place on April 11^{th}–14^{th} 2005 in Atlanta and in a report on micro process technology in the US which was published by VDI/VDE Innovation + Technik GmbH. Examples include hydrogenation for the production of pharmaceutical intermediates by Bristol Myers Squibb (USA), free radical polymerization at the University of Kyoto (Japan) and the use of hydrogen peroxide to epoxidize propylene, which is a sample application of a gas–phase reaction in an Uhde DEMiS reactor. These are examples of systems for industrial production which go beyond component development and characterization. The system at the University of Kyoto is 3.5 m x 0.9 m and has an annual production capacity of 5–10 tons.

MICRO REACTION TECHNOLOGY – TINY DEVICES WITH TREMENDOUS POSSIBILITIES

By demonstrating radical reductions in size and weight, and better heat transfer, mass transfer and other performance advantages compared to conventional systems, microreactors, microfluidic devices and micromachined components have the potential to revolutionize chemical analysis, chemical synthesis, industrial automation, plant safety, gas detection, medical and biomedical applications.

Under normal circumstances, process developers routinely concentrate their engineering efforts on scaling up promising technical concepts from R&D scale, to demonstration and pilot plants, and ultimately to commercial–scale facilities. But in one corner of the chemical engineering universe, researchers are working in the opposite direction – developing a variety of devices, which, at full scale, can comfortably fit on their inventors' fingertips.

Microtechnology involves the fabrication of so–called micro–electro–mechanical systems (MEMS) – such as microreactors, microfluidic devices and microscaled machines – whose outer dimensions are measured in millimeters or centimeters, and whose inner surfaces are configured with pores or channels that have dimensions measured in micrometers (one micrometer equals one–millionth of a meter).

Because of their inherently high surface–area–to–volume ratios, such tiny mechanical components demonstrate order–of–magnitude improvements in heat– and mass–transfer rates, allowing highly efficient, compact and cost–effective devices to be created to carry out chemical and thermal reactions more safely, and with greater selectivity and conversion rates, higher yields, and improved product quality.

The possibilities are endless

In the decade or so since microreactor technology first emerged as a scientific discipline, there has been a steady increase in the number of chemical reactions and physical changes that have been successfully performed in such miniature devices.

Over the past few years, promising developments have emerged related to the design, development, fabrication and commercialization of microscaled mechanical devices as varied as heat exchangers, pumps, mixers, gas absorbers or adsorbers, liquid–liquid extractors, chemical reactors, bioreactors, combustors, catalytic reactors, enzymatic reactors, fuel processors. In addition, microsensors and microactuators for pumps, valves, compressors and other components have also been successfully developed, and microscaled components have been demonstrated for drug–development (so–called "lab–on–a–chip" devices), and as diagnostic tools for medical purposes.

Considerable work is also under way to develop microfluidic devices. This subset of microtechnology involves the design, modeling, manufacture and mass production of tiny systems that handle fluids (gases, vapors or liquids) in volumes that can be as small as nano– or pico–liters.

Microfluidic devices rely on an array of active and passive microstructures that control the flow and mixing of the fluids, in order to produce the desired physical, chemical and microbiological reactions in a rapid, cost–effective manner. As with other micromachined devices, the enormous surface–area–to–volume ratio within a microfluidic device brings attractive mass transfer and heat flow advantages.

Through their design and fabrication efforts – often with the help of computational simulation and modeling of fluid flow – today's microtechnology pioneers continue to decipher how fluids flow through smooth and textured microchannels and capillaries, and determine how modifications of the channel surfaces and configurations will affect flow or mixing characteristics. Researchers across a wide range of engineering disciplines are also continuing to explore effective ways to overcome the effects of viscous drag and friction, to obtain sufficient clearance space between dynamic members and the walls of the pump cavities, and sealing the pump cavities from the exterior.

Today's lightweight, compact, high–performance microscaled devices have important implications for chemical process, biotechnology, medical, automotive, consumer, manufacturing, environmental, and space–related applications, among others. And interest in this new frontier is global, with promising developments routinely being announced by industrial, government and academic researchers in the U.S., Europe, Japan and Australia.

Scale–up and microfabrication techniques

There are three general options for using microreactors and microfluidic devices: As miniaturized analytical laboratories, as production units for chemical substances or for a better understanding of chemical processes. To achieve commercial–scale production capabilities, numerous microscaled units are linked in parallel. Process developers are still working to design proper manifolding strategies to do this.

Since most early systems are essentially expensive, handmade prototypes, the ability to make the equipment affordable for commercial production will ultimately rely on the ability to scale up and perfect the mass production techniques needed for these microscaled devices. The precise fabrication requirements needed to manufacture complex devices that measure no larger than a finger nail or shirt button cannot be overstated.

The tiny three–dimensional structures within microreactors and microfluidic devices are typically fabricated by etching or micromachining channels and patterns on a wide range of substrates. The materials that are used range from stainless steel, high–temperature alloys, and nonferrous metals (such as copper and aluminum) to borosilicate glass, ceramics, engineered polymers and silicon.

Multiple, patterned layers are assembled to form more complex three–dimensional structures, whose buried channels and reservoirs either enable fluid mixing, or maintain separate fluid flows through the use of laminar flow that prohibits mixing.

Today, a host of state–of–the–art fabrication techniques – many of the same techniques that have allowed the electronic industry to pack more and more memory into smaller semiconductor chips – are used to lay down designs and selectively etch away parts to create the desired channels and patterns. These techniques include photolithography, non–reactive ion etching, chemical etching, electrochemical and laser machining, photo–ablation by excimer laser, chemical–vapor deposition, thin–film deposition and selective plating.

The fabrication of such tiny microstructures requires extreme precision and resolution, as the etched channels often have relatively large depth but narrow widths (i.e., very high aspect ratios). Developers agree that high resolution, repeatability and reliability remain ongoing challenges as low–cost mass–production techniques are being developed.

Researchers at Lawrence Livermore National Laboratory have developed techniques that allow them to precisely align and fuse together glass and/or silicon substrates that have been patterned and etched to create various types of microreactors and microfluidic devices. First, the group precisely aligns the substrates (claiming an alignment accuracy of within 1–2 micrometers), then the glass–to–glass, silicon–to–glass, and silicon–to–silicon bonds are created using a variety of state–of–the–art annealing processes.

Mikroglas chemtech GmbH (Germany) is specialized in the production of microfluidic products out of glass. They are using a special fotostructurable glass where very small structures with high aspect ratio can be produced. By diffusion bonding of up to 20 glass plates with different functions, it is possible to manufacture various customized microreactors, especially for handling of aggressive chemicals.

Meanwhile, using a modular assembly approach, a team of investigators at the University of Illinois (Urbana–Champaign) and Northwestern University has developed a novel method for simplifying the construction of microfluidic devices. First, the opposing aspects of a given design – such as the passive microfluidic components (such as channels and reaction reservoirs) versus the active electromechanical control structures (such as tiny sensors, actuators, pumps and valves) – are separated, and then they are manufactured separately. The components within each category are then built on separate layers, or even as two separate devices. The distinct layers, or separate devices, are then connected through common nanoscale channels. This approach is said to simplify both the design and the construction of the finished integrated device, and allows for customization merely by using a different microfluidic component and reprogramming the overlying sensor and actuator circuitry.

Developing sophisticated packaging strategies is another ongoing challenge as mass production techniques are developed for microreactors, micromachined parts and microfluidic devices. Effective packaging must not only encase the microfabricated component (to protect it from its operating environment), but it must also be designed to allow for all of the needed electrical and microfluidic interconnects, and must include features that allow for precise alignment with other microscaled components, optical viewports, and enable proper thermal management.

Ongoing developments

Despite the challenges, promising new applications based on microtechnology are being announced every day. Some representative developments, spanning a range of diverse applications, are profiled below.

Microscaled machines

The scale of today's microtechnology–based inventions boggles the mind. For instance, engineers at the Massachusetts Institute of Technology (MIT) are developing a gas–turbine engine that can easily fit on a dime. The entire device – complete with an integrated electric generator – is expected to have dimensions of just 2 centimeters diameter by 3 millimeters thickness, and weigh just 1 gram. MIT researchers have already manufactured a 4–millimeter–diameter, radial–inflow turbine wheel from silicon using deep reactive ion etching. Calculations suggest that the later versions of the complete gas–turbine generator system made from heat resistant silicon carbide – with a volume less than 1 cm^3 – could deliver as much as 100 Watts of electric power using hydrocarbon fuels. Researchers envision using these tiny engines being used for portable power production. General Electric is also working to develop high–efficiency microturbine technology.

Meanwhile, Ehrfeld Mikrotechnik BTS is offering the LH 1000 micromixer, which is designed to handle flowrates of several 1000 L/h, with a pressure loss of less than 1 bar at 1000 L/h. The mixing inside the 100–by–100–by–80 mm^3 unit is performed by multi–lamination. The unit contains only two microstructured foils (for easy maintenance) with more than 12000 microslits, each of 50 μm width, to cut the two inlet streams into a large number of alternating microlayers for extremely fast mixing of two components.

Also the Institute of Microtechnology Mainz GmbH (IMM) has developed a micromixer, called the Star–Laminator, that is capable of handling flowrates of up to 300 L/h at a pressure loss of 12 bars. The complete unit, with dimensions of just 45–by–25–by–30 mm^3, consists of a stack of thin foils (numbering between 320 and 1,600 of them), each bearing microchannels with different patterns.

Chemical synthesis

In the area of chemical syntheses, microreactor research is proceeding along two parallel courses: Efforts are underway to both improve the selectivity, conversion rates and yield of well–known reactions, and to find ways to carry out problematic reactions (such as those that are highly exothermic) more safely and cost–effectively.

Microtechnology's initial promise in this arena is for the production of low–volume, high–value applications, such as costly specialty chemical and pharmaceuticals, but other developers have set their sights on larger–scale applications, as well.

The improved safety associated with using microreactors to carry out highly exothermic reactions results from several factors. First, when carried out in conventionally sized reactors, highly exothermic chemical reactions run the risk of runaway reactions that could lead to fires or explosions. The large ratio of material volume inside the reactor to the surface area of the chamber walls this makes it hard to extract the heat generated during the reaction.

By comparison, when exothermic chemical reactions are carried out in extremely small volumes within the microchannels of the reactor, the extraordinary ratio of surface area to material volume promotes very efficient heat dissipation, which greatly reduces the risk of a thermal runaway, and inhibits undesired gas–phase chemistry by thermal or chemical quenching of free radicals.

Similarly, conventional tank reactors often experience temperature gradients within the materials being reacted, so materials near the walls might experience different temperatures compared to materials in the center of the vessel; this not only impairs the reaction, but may result in the formation of undesirable (and dangerous) byproducts and waste materials, as well. However, when the relative surface area inside the reactor is increased dramatically in relation to the volume of material in the reactor (through significant reactor downsizing), temperature can be more precisely controlled, thereby eliminating temperature gradients, and drastically improving the success and safety of the process.

For this reason, many researchers are investigating the use of microreactors for potentially explosive reactions. The ability to carry out the direct, controlled (rather than explosive) reaction of hydrogen and oxygen to form hydrogen peroxide is one opportunity where microreactors can offer a safer, more effective and less costly route compared to the conventional approach. One of the first examples was performed by the German Fraunhofer Institute for Chemical Technology (ICT): they investigated highly exothermic nitration reactions by using glass microreactors from mikroglas. They could show that for example the nitration of naphtalin was possible with very aggressive chemicals, under conditions which normally would lead to explosions and with a very high selectivity and yield.

Similarly, researchers at the Institute of Microtechnology Mainz GmbH (IMM) and Massachusetts Institute of Technology are developing a microreactor that can perform the direct fluorination of aromatic compounds. In early trials, the fluorination of toluene with elemental fluorine was carried out in a microreactor setup that included reaction channels and heat exchanger structures in close proximity. Due to its potentially explosive nature, this reaction can only be carried out in conventional equipment at –70 °C, very carefully, under lab–scale conditions. However, by using a falling–film reactor designed by the Institute of Microtechnology Mainz GmbH (IMM) – a reactor whose microstructured reaction plate maximizes internal surface area to improve liquid distribution – the reaction mechanism can be changed from a radical–chain type reaction (which is uncontrollable and unselective) to an electrophilic substitution one (which is safe and more selective), even at –10 °C.

The Leibniz–Institute for Catalysis (former ACA, Institut für Angewandte Chemie Berlin–Adlershof / Germany) combines longtime expertise in the field of catalytic processes with extensive experiences in the application of the microreactor technology. It uses a broad variety of microstructured components like falling film microreactors, micro bubble columns, micro jet reactors, microstructured reactors with catalytically active walls and interdigital micromixers for reactions in the liquid phase. The former ACA has applied the microreactor technique to photochemical reactions e.g. side chain chlorination of alkyl aromatic compounds, fluorination with elemental fluorine, photooxygenation using in situ generated singulett oxygen and sulfonation of aromatic compounds using gaseous SO_3. The Leibniz–Institut for Catalysis at University Rostock was formed by merging of ACA and IfOK on 1st January 2006.

IMM also recently established a long–term cooperative agreement with China's Xi'an Huian Chemical Industrial Group to develop applications of microreactor technology for the production of fine, speciality and commodity chemicals. Hi'an Huian Chemical recently started producing nitroglycerin – a poisonous, explosive compound that requires the use of extremely acidic reactants during manufacturing – at about 15 kg/h, using IMM's microreactor technology.

The Karlsruhe Research Center and DSM Fine Chemicals GmbH in Linz, Austria recently demonstrated the production of more than 300 tons of a polymer product in a 10 weeks production campaign. The employed high–performance microreactor developed at Karlsruhe Research Center enables the manufacturing of 1700 kg/h liquid chemical products.

In addition to electrochemical reactions, thermal and photochemical reactions can also be improved using microtechnology, too. With this in mind, a new microreactor for rapid temperature cycling has been developed by the Karlsruhe (Germany) Research Center and Germany's University of Erlangen. The device is able to change the temperature of a gas flow by 100 K within 2 s, versus several minute for a fluidized bed. The group plans to integrate a catalyst into the device and perform heterogeneously catalyzed gas–phase reactions.

Researchers at Eindhoven University of Technology have also found that a microreactor that promotes non–uniform flow distribution for improved fluid cooling can improve the platinum–catalyzed oxidation of ammonia to produce nitrous oxide. For this highly exothermic reaction, proper cooling is essential to avoid hot spots and to reduce temperature gradients across the catalyst, both of which decrease the selectivity for nitrous oxide.

In other chemical synthesis applications, Velocys and Dow Chemical are working to develop a microchannel process to more cost effectively produce ethylene. Velocys is also working witk Total S.A. on another microchannel application to convert "stranded" natural gas into pure hydrogen or synthetic liquid diesel fuel.

DuPont Co. has developed a versatile microreactor, the size of a hockey puck, that is fabricated from layers of wafer–like disks made from ceramics, glass, polymers, composites and metals, whose precise interior channels – measuring a mere 10 to 5,000 micrometers across and connected to the inlet and outlet ports – contain the reactions.

Also microreaction toolboxes for chemical synthesis, like the one from Ehrfeld Mikrotechnik BTS, mikroglas or German Fraunhofer Institute for Chemical Technology (ICT) plays an important role in the development of new products and processes. The toolboxes based on a large variety of modules, where each module is designed to perform a process operation, including mixing, heat exchange, catalysis, separations, and analysis and control of reactions involving gases, liquids, and multiphase fluids. Even precipitation reaction can be carried out within these toolboxes. In combination with process automation system it is an powerful tool for development and small production.

Meanwhile, to improve chemical reactions, microreactor technology is also showing promise for improving certain physical reactions. For instance, in Japan, a microscaled alternative to energy–intensive mixers for making emulsions has been developed by researchers at the National Food Research Institute, the University of Tsukuba, and the University of Tokyo. The researchers have been able to form monodispersed oil–in–water dispersions simply by forcing soybean oil through a silicon device embedded with microchannels. The array is configured as a 4 inch–diameter wafer with 10,000 channels – each 200 micrometers long and 20 micrometers in diameter – into a continuously flowing aqueous phase. Microspheres of oil emerge from these channels at pressure greater than 1.8 kPa to form the emulsion. A single device can produce 6.5 mL/h of stable emulsion.

Fuel cells

Fuel cell developments are being hotly pursued, as they have great application potential as stationary power plants, as power sources for vehicles, and as portable power generators for electronic devices. So it is no surprise that fuel cells represent another area in which microtechnology is bringing some noteworthy performance advantages.

In a project funded by the U.S. Dept. of Energy (DOE, Washington D.C.), researchers at the Pacific Northwest National Laboratory (PNNL) are using microfabrication techniques to develop compact microreactors that will use partial oxidation and steam reforming to convert liquid hydrocarbons such as methane into hydrogen (which is not easily transported or stored) at the point–of–use, to power fuel cells. The fuel cells will then convert the hydrogen into electricity for use in automotive applications. PNNL researchers say that their prototype miniature fuel cell/fuel processor system could weigh as little as one kilogram, and continuously provide 5 Watts of baseload electric power, with 10 Watts of peak power, for one week.

Similarly, researchers at the Netherlands' Eindhoven University of Technology are developing tiny, microchannel fuel processors that function as hydrogen–producing plants for use with fuel cells. In early modeling and testing, this microreactor design outperformed conventional fixed–bed designs.

Meanwhile, researchers at the Pacific Northwest National Laboratory (PNNL) have built what they believe to be the world's smallest fuel processor, about the size of a blueberry, that is being developed for use in handheld wireless equipment and sensors for the U.S. military.

In another fuel–cell–related development, researchers at the University of Illinois (Urbana–Champaign), working with INI Power Systems, Inc., have designed a microfluidic fuel cell that operates without the need for a solid membrane to separate the fuel and oxidant. Eliminating the membrane is expected to reduce fuel–cell costs (because the membrane typically accounts for about 20–30% of a fuel cell's cost), and this development will make it possible for the fuel cell to operate with either alkaline or acidic chemistry, the researchers say.

The microfluidic cell consists of a Y–shaped channel (about 1 mm in height and width), in which two liquid streams containing fuel and oxidant are merged without mixing (thanks to laminar conditions at these small dimensions) into the 3–cm–long stem. Work is ongoing to increase the power output by connecting multiple cells in a stack.

Industrial sensors and analytical devices

Many devices based on this technology, including pressure sensors and airbag accelerometers in automobiles, are already in large–scale commercial use. Sensirion AG recently commercialized an integrated circuit that contains a miniature thermal mass–flow sensor with all related signal–processing electronics (amplification, analog–to–digital conversion, integration, linearization and termperature compensation) on a single microchip. The combination of smaller dimensions and integrated signal conditioning enables the chip to control mass flow ten times faster (150 microseconds) and more accurately (0.8% of measured value) than conventional devices that use coils around a steel capillary, according to the company.

Important for sensor elements is that the internal volume should not be larger than the internal volume of the used microreactor. They will be used in very aggressive chemicals, so that the right choice of material is extremely important. The German company Bürkert developed together with mikroglas a commercially available "sensorblock" for microreaction systems which includes temperature and pressure sensors as well as a pressure relief valve and a non–return valve. Only chemically inert materials like glass, teflon or cermics are used.

For chemical reactions in an continuous operating plant and in micro reaction production plant inline analytic is important. For example Bayer Technology Services together with Ehrfeld Mikrotechnik BTS demonstrated the use of NIR–spectroscopy in a micro reaction plant by using a optical flow through cell.

Meanwhile, researchers at Australia's CRC for Microtechology are working to develop and commercialize cost–effective diagnostic assays, based on microfluidic devices using polymerase chain reaction (PCR) technology, that will enable the rapid detection of common food–poisoning pathogens (such as *Salmonella*, *Campylobacter* and *E. coli*) directly from food samples.

Biocatalysis

During biocatalysis, certain enzymes require co–enzymes in order to function properly as catalysts. Because co–enzymes are expensive, they often need to be regenerated in situ in order to make biotransformations cost–effective for industrial production of fine chemicals. Last year, researchers at the University of Illinois at Urbana–Champaign and Université Paul Sabatier pioneered a microfluidic electrochemical device that regenerates co–enzymes while avoiding unwanted side reactions that have plagued alternative methods. The microreactor is made from a polymer plate in which a Y–shaped channel made by micromachining or replica molding. Opposing walls of the 3–cm–long stem each have a gold coating that serves as the electrodes. Two liquid streams – one containing a phosphate buffer and another with all the reactants (substrate, enzyme, co–enzyme, mediator – are pumped through the Y channels and merged, without mixing, into the stem. Because of laminar–flow conditions, the stream containing all the required reactants can be focused near the cathode, preventing the reverse reaction – which occurs in bulk–phase reactors – from taking place, thus driving the equilibrium in the direction desired. At a total flowrate of 0.01 cm^3/min, the microreactor demonstrated a regeneration efficiency of about 31%. The group is now developing arrays of multiple–stream laminar–flow–based microreactors (with recirculation) to increase the conversion efficiency and boost the throughput.

In summary

Microreactor technology promises to revolutionize the chemical process industries in much the same way that microelectronics has transformed information processing over the past few decades. Miniaturized devices and reactors offer technical advantages for a large number of applications.

Among the many benefits derived from microreactors, safety is a major one. Particularly where chemicals that are dangerous to manufacture, handle, ship or store are concerned, these substances must often be produced in small quantities onsite, as needed. Microscaled reactors open the door for small–scale, point–of–use, and on–demand production of extremely harmful and toxic substances, at lower cost and with reduced risk compared to conventional approaches.