

The New Multiple Stream Mixer Reactor from Microfluidics

Microfluidics has developed a revolutionary approach to fast, controlled reaction chemistry utilizing multiple reactant fluid streams. Advantages of the process include:

- Mixing energy density an order of magnitude, or greater than any other technology
- Product qualities not available from conventional batch reaction technology
- Enhanced intimacy of reactants, particularly for reactants in immiscible streams.
- Size and size-distribution control for precipitated products
- Manufacture of nanomaterials in commercial quantities
- Low capital cost at manufacturing level
- Predictable scaleup from laboratory to high throughput manufacturing

The high-intensity Multiple Stream Mixer Reactor (MMR) is a patented continuous mixer/reactor that was designed to optimize the fast chemical reactions required in many of today's chemical processes. It allows development and manufacture of nanomaterials in a process controlled to the molecular level of mixing. In most conventional chemical reactors, inadequate mixing and mass-transfer rates limit the value and performance of a fast chemical reaction. As a result, product yields are low, and unwanted by-products are produced.

The MMR utilizes basic Microfluidizer® technology in pressurizing liquids and converting the pressurized energy to intense mixing in a proprietary mixing chamber. Multiple streams of pressurized reactants are brought together in the MMR chamber with residence times of a few hundred microseconds to a few hundred milliseconds. The reactant streams are subjected to dispersion at nanometer size by passage through high-shear microchannels and collision in an intense energy field. Alternate mixing devices, such as a single stream mixer, do not provide the conditions required for fast reactions, because feed components are pre-mixed, and some precipitation occurs prior to the reactants reaching the micro-mixing zone. The purpose of this new technology development is to:

- 1) provide materials with uniquely small crystallite size;
- 2) synthesize materials having improved nano-scale homogeneity;
- 3) improve phase and chemical purity of products in production systems where multiple reactions can occur.

In producing uniform nanometer-scale dispersions or suspensions, the history of the starting material limits the properties of the end product. Dispersion/mixing systems cannot create particles smaller than the existing primary crystallites, since mixing energy densities do not approach levels required to disrupt covalent or ionic bonds. Only by controlling the history of the starting material can truly predictable nanoparticle preparations be manufactured.

The MMR system allows total control of material history and produces nano-scale structures, which can then be separated, purified and redispersed in a desired medium at the primary structure sizes created. Reactions of two or more streams of pure starting materials are conducted in an ultraturbulent collision zone, continuously and under total control of reaction conditions and reactant stoichiometry. Precipitation products can then be recovered and further processed into desired formulations. This process is best accomplished in a standard Microfluidizer processor. Alternately, this primary structure size can be maintained in a solid material for further processing.

In its simplest form, the MMR is a device which accelerates independently two streams of highly pressurized reactant solutions by pumping them into narrow channels and causing collision of the streams in a microliter-size reaction zone. Computer control of inlet valves, using feedback from flow sensors, ensures proper stoichiometry in the reaction zone. Each stream is pressurized in the range of 5,000 to 40,000 psi, led into an inlet channel a few mm long with a cross-sectional area in the range of 0.01 - 0.05 cm² where fluid velocities of 3-30 msec are achieved. Jets are then formed in a second channel about 0.01 mm long and with a cross-sectional area of 0.0001 - 0.001 cm², with each jet reaching velocities in the 80-300 m/sec range. The channels are formed in diamond or ceramic constructs.



The jets are caused to collide in a reaction zone, which has a volume of a few micrometers after which the reacted mixture is released, at lower pressure, to a relatively large exit channel. Residence time in the reaction zone can be varied, and can be in the low millisecond range. All of the kinetic energy in the colliding streams is utilized in the ultraturbulent mixing region in the reaction zone. Here, the liquid structures are near molecular size and there is little diffusion resistance, so that rapid reactions are favored and slower alternate reactions leading to impurities are minimized. Also, there are no significant temperature, pH or concentration gradients which could cause phase inhomogeneities in the product crystals.

The inlet streams can be mixed in a macromixing zone before entry into the inlet channels. Liquid mixing occurs for a few milliseconds in these channels creating a small amount of microcrystalline product nuclei to seed the reaction in the reaction zone, if necessary. Details of the pressures, channel dimensions, reaction zone dimensions and exit channel dimensions are experimentally optimized to accommodate the specific kinetics of the reaction being conducted. Bulk fluid temperature rise in the system is 5 – 20 degrees centigrade, depending on process pressure, and heating or cooling pre-and post reaction can be provided.

Post-reaction processing may be necessary for some reactions to prevent crystal growth or to alter crystal morphology (length/diameter ratios of needle-shaped crystals, for example).

Agglomeration is a natural post-reaction event. It can be minimized by dilution of the product stream. Alternately, the product can be redispersed at a later time.

Ultraturbulent collision reaction chemistry, as practiced in MMR applications, may someday be accepted as a unique unit operation in chemical engineering.

Figure 1

Macro-Meso-Micro Chamber

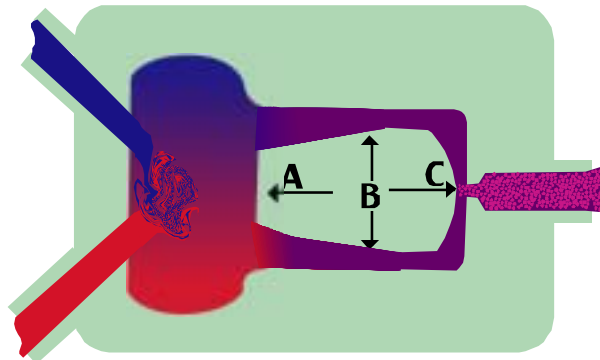
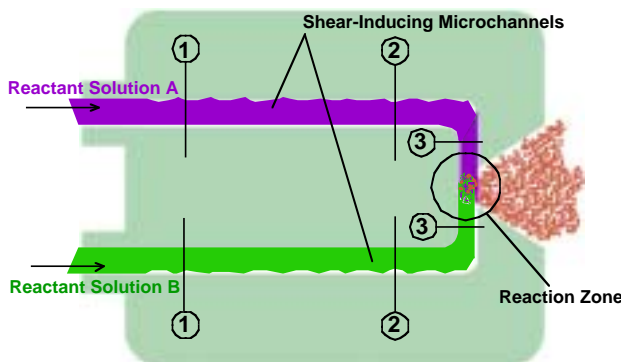
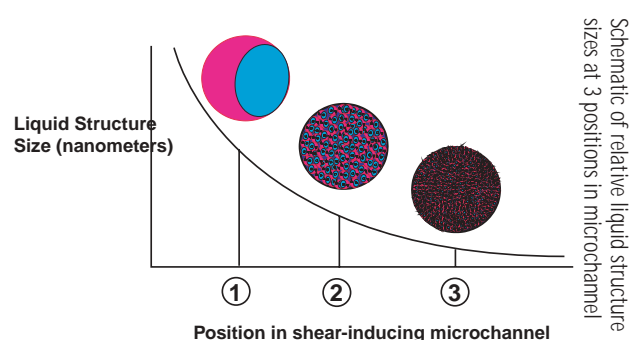


Figure 2

Direct Impingement Chamber

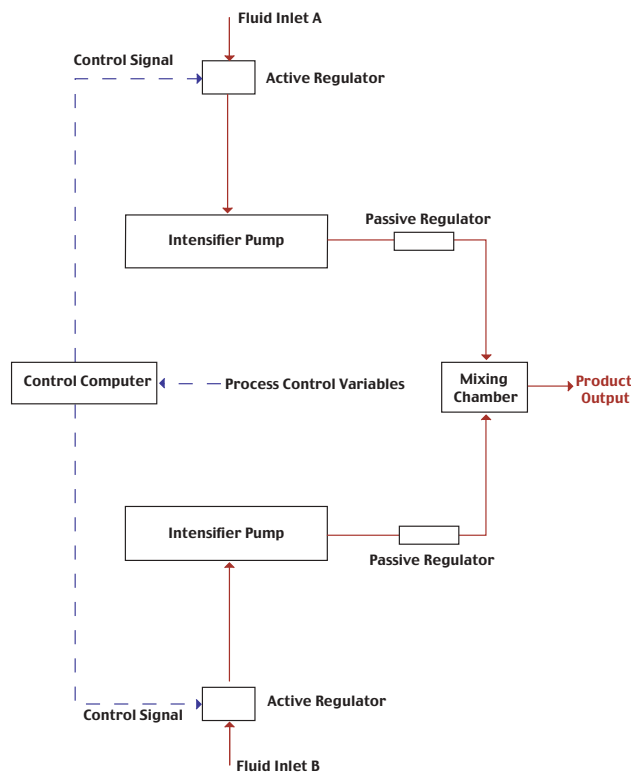


Liquid Structure Sizing Reduction



In collaboration with others, MMR prototypes were successfully built and tested by Microfluidics (see Figure 3). The first trials were for the precipitation of metal oxide catalyst precursors with reduced crystallite size and improved nanometer-scale uniformity and phase purity. The laboratory MMR uses computer controls to adjust the flows from two pumps so that they operate in phase, i.e., they start and stop at the same times, but may pump at different rates. Especially important is that the machine is designed to ensure that not only is the average stoichiometry delivered correctly, but also ensures, as much as possible, that the correct stoichiometry is used throughout all phases of the pump stroke.

Figure 3
MMR Schematic Diagram



For precipitations, homogeneous nucleation can occur when the local concentration exceeds the supersaturation limit. Increasing the level of supersaturation through high intensity mixing can result in a rapid rise in nucleation rate, and a decrease in crystallite size of the precipitated product. In addition, intense mixing minimizes local inhomogeneities in solution, e.g. inhomogeneities in pH or concentration. As a result, the properties of the product in terms of phase purity and uniformity of composition and uniformity of crystal size, are also improved. The resultant catalyst crystals produced during the program measured less than a micrometer in diameter, had a narrow size distribution and had a micro-composition described as previously unachievable phase purity.

The laboratory MMR used for catalyst precursor preparation has a capacity of 10 gallons/hour. Scaleup to 400 to 600 gallons/hour, and higher, is readily achievable.

The degree of control of microstructure size and uniformity achieved by this technology is unprecedented, and thus enables new product development and existing product enhancements. Some product classes which may utilize the MMR technology include:

- **water-insoluble drugs** - can be recrystallized, purified and then dispersed as nano-suspensions in an injectable medium, or formulated for topical, transdermal, inhalation or oral delivery
- **superconductors** - uniformity of phases at the nanometer scale is essential for high performance
- **abrasives** - control of size and uniformity leads to optimal control of abrasion and polishing
- **planarization media** - in the manufacture of semiconductors or semiconductor wafers, planarization of successive deposition layers must be defect free. **Chemical Mechanical Planarization (CMP)** processing relies on uniformity and size control of abrasive crystals.
- **pigment synthesis** - paint performance (appearance, wear, application, etc.) depends on controlled physical properties of pigments
- **ceramics** - for low thermal expansion and isotropic strength applications
- **ultrapure chemicals** - recrystallized by ultraturbulent processing

Microfluidics seeks to form working relationships with potential users of its breakthrough technology. In return for sponsorship, collaborators will gain access to the technology on a favorable basis, and achieve an early competitive advantage. Microfluidics is prepared to manufacture equipment and provide technology assistance for process development, optimization and application.

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