Milli-fluidic Reactor for Catalyst Research

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Summary

Miniaturization of laboratory processes to form micro total analysis systems (µTAS) has gained a great deal of attention in academic and industrial research laboratories. Advantages including increased speed of analysis, parallel screening, well-defined and repeatable experimental conditions, and significantly lower cost of research make microreactors attractive to synthesize nanomaterials. Due to the well-defined space and reaction conditions within the reactor channels problems associated with heat and mass transfer are minimized and proper chancel design allow separation of nucleation and growth phases critical for obtaining monodisperse particles.

Researchers at the Center for Advanced Microstructures and Devices (CAMD) jointly with groups on LSU campus have established a modular microfluidic design concept and a variety of fabrication methods to quickly transfer a reactor chip design into a working device readily available for studies [1]. Together with partners at LSU's Energy Frontier Research Center for Atomic-Level Catalyst Design (http://www.efrc.lsu.edu/index.html) these capabilities are utilized in synthesizing new catalytic materials and analyzing their properties.

Exploiting the unique properties of nanoclusters consisting of about 100 atoms requires atomically precise fabrication techniques. Polymer millifluidic chips combine simple, reproducible, and cost-effective fabrication with controlled experimental setups ensuring repeatable synthesis of these clusters and the opportunity for *in situ* characterization [4,5,6,2]. In first experiments the millifluidic platform has been demonstrated to be a simple, inexpensive tool for controlled, large scale and automated synthesis of nanoclusters as well as a tool for probing reactions using any number of spectroscopies [3,4,5,6].

Fabrication of microfluidic chips

PMMA microfluidic chips were made using the hot embossing technique [7]. Completing the AutoCAD drawing (Fig. 1) with the desired, simulated design [8]is followed by micromachining of a Brass mold insert using Kern MMP micro Milling Machine available at Center for Bio-Modular Microsystems-CBM[2], LSU (Fig. 2). Brass (353 brass alloy) is used as insert substrate due to its good machining capability and low hardness which increases the tool life. Note that, the brass insert (Fig. 3) contains the inverse fluidic chip design where the smallest cutting tools used was 50µm in diameter [9]. Next, the brass mold insert is mounted into a HEX 2 hot embossing machine installed at CAMD (Jenoptik, Jena, Germany) (Fig. 4) and replicated into 3mm thick rectangular PMMA sheets. Pattern transfer is done under vacuum with moderate force and at temperatures (150-160°C) above the glass transition temperature Tg of PMMA material [7]. After demolding of the PMMA chips from the brass insert at temperatures below Tg (Fig. 5) they are separated using fly-cutting. This process also opens inlet and outlet holes. After separation (Fig. 6) the chips undergo ultrasonic cleaning in microsoap, IPA, and Di-water followed by dehydrating treatment for 5hrs at 60°C. It should be noted that these millifluidic chips have low to moderate aspect ratios and therefore can be replicated by injection molding in high numbers and at low costs.

Sealing of the open channels is done by thermally welding the cover sheet onto the open chip is done in a convection oven at ~110˚C using the spring clamp fixture shown in Fig. 7 [10,11]. A total force of ~100KN for the 3"x1" chip area along with 1.5hrs dwell time followed by a slow cool down ramp of several hours to room temperature ensure proper sealing without any deformation of the delicate channels. This complete the fabrication process (Fig.8).

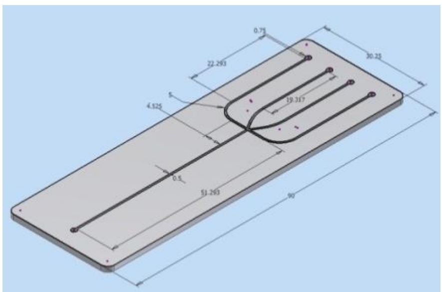

Fig.1: 3D CAD design of the 4-inlet port Hydrodynamic focused mixer

Fig. 2: Kern MMP micro Milling Machine at CBM2

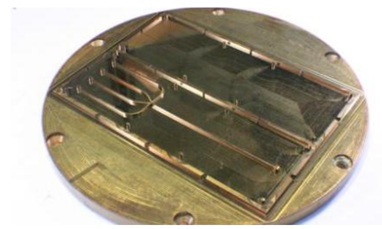

Fig. 3: Mold insert made from brass with inverted pattern of the fluidic chip.

Fig. 4: HEX 02 hot embossing machine installed at CAMD

Fig. 5: Fluidic chip and cover slide hot embossed into PMMA.

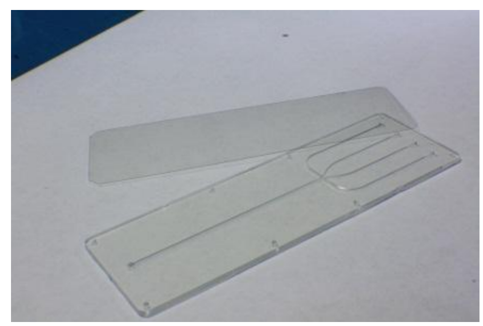

Fig. 6: Fluidic chip and cover slide after separation and cleaning.

Fig. 7: Spring-loaded clamping fixture for thermal welding of cover welding.

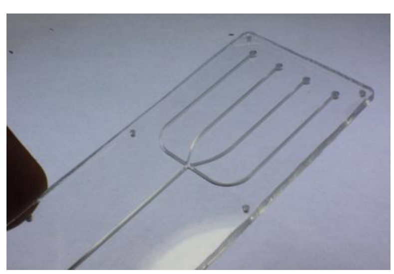

Fig. 8: Sealed chip ready for testing.

Fluidic interconnect and testing

In order to conveniently use micro- and millifluidic devices standardized macro-micro interfaces along with design flexibility are crucial criteria for success [12]. CAMD's modular fluidic stack concept [1] combined with standard interconnects for example offered by microplumbers (http://microplumbers.com/) is an attractive and cost-effective solution to this request. The chip design is shown in Fig. 9 where the inlet arrangement of a hydrodynamic focusing design circled in blue meets microplumbers manifold layout allowing for an easy place & clamp interconnect solution (Fig. 10). Key features of this connector include manual thumb screw tightening and O-ring sealing for leak-proof connection between the microfluidic device and the manifold, up to 6 fluid ports can be connected via PEEK tubing to various chemicals and easy viewing of fluid movement due to transparent polymer materials.

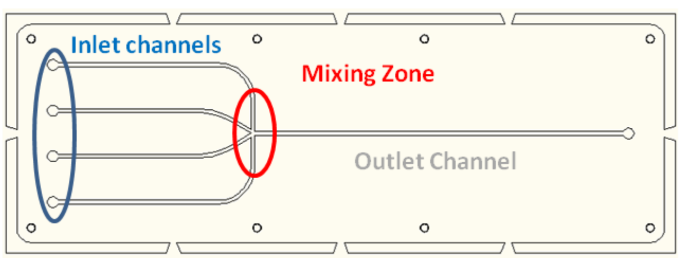

Fig. 9: Concept and layout of millifluidic chip matching the inlet channel arrangements with microplumbes manifold while offering customized solutions (mixing zone) for user applications.

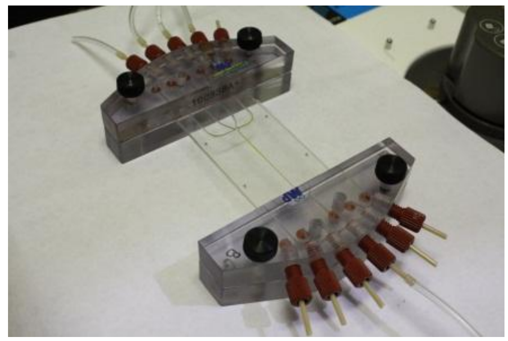

Fig. 10: Microplumber manifolds with clamped fluidic chip ready for experiments.

In conclusion combining in-house micro-milli-fabrication expertise with customer designs and commercial interconnect solutions is offering a powerful technology for microreactor research and applications and has successfully been employed in the EFRC catalyst research efforts with results published elsewhere [2-5].

References

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