FABRICATION OF MICROCHIPS FOR RUNNING LIQUID CHROMATOGRAPHY BY MAGNETOHYDRODYNAMIC FLOW

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Abstract

This paper introduces a new microchip design for liquid chromatography employing magnetohydrodynamic (MHD) flow. An electromagnetic channel (EMC), which has a rectangular cross-section and electrodes that run the full length of the channel maximizes MHD pumping power. EMC fabrication on Si requires a two-mask process. ICP-RIE was employed to open rectangular grooves, the electrodes and leads were then filled by slowly electroplating. A polishing process needed to be developed to achieve the required planarity for subsequent bonding. Finally, the second ICP-RIE step opened a 10 μ m wide channel between the electrodes. Bubble current density threshold tests indicated over 100 mA/mm² could be applied at 960 Hz before electrolysis created gas bubbles. This is high enough to give the required pumping velocities for open tubular liquid chromatography.

Keywords: Fabrication, liquid chromatography, magnetohydrodynamic flow, pumping

1. Introduction

Electroosmotic flow has been successful to date, in part because on a chip it is a challenge to manipulate traditional head pressure driven flow without active valving.

Magnetohydrodynamic (MHD) flow could be very important on-chip, because it creates a pressure driven flow without requiring mechanical valves [1, 2]. Unfortunately, the flow velocity is inversely proportional to the square

Table 1. Relative mean velocities (square channel as reference) generated by channels with the same cross-sectional area and depth but different shapes

Cross-section	θ	Relative mean velocity
trapezoidal	54.7	29.4
trapezoidal	60	41.5
square	90	100

of channel dimensions, so this pump scales in the wrong direction for microfluidics. This report explores designs to resolve drawbacks in using MHD to perform chromato-graphy in narrow channels, using an *electromagnetic channel (EMC)* running the full channel length. This achieves more powerful pumping in narrow channels (Table 1).

2. Design

MHD does not generate high enough pressure for packed bed chromatography, and open tubular chromatography requires narrow channels (10 μ m or less), making the design of an MHD device challenging. Table 1 shows relative mean velocities calculated for channels with different cross-sections. A square EMC gives the highest velocity because of its uniform electric field and low flow resistance. Using electrodes that run the full length of the channels also maximizes the pumping pressure. The use of large cross-section metal deposits to form the wires is required to avoid significant potential differences along the channels, given that currents of 100 mA will be needed. We calculate an average velocity of 0.12 mm/sec could be achieved at 0.45 T with these design elements. Flexible flow control in each channel is required, demanding some complexity in the design. None of these design elements has been used in on-chip MHD previously.

3. Fabrication

The EMC design requires a top plate be bonded to create capillaries from the channels formed in the bottom plate, placing severe constraints on the planarity of the upper surface of the bottom plate. Further, the required feature size of 10 μ m made SU-8 unacceptable as a deep resist, as 15 μ m is a typical lower feature size [3], meaning bulk machining with ICP-RIE was preferred to obtain vertical sidewalls.

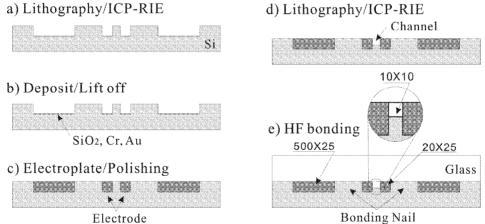


Figure 1. Process steps for the fabrication of EMC for MHD flow. The cross-section of the $10 \times 10 \mu m$ EMC is shown as an inset in (e)

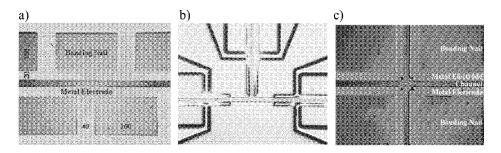


Figure 2. Photomicrographs for MHD channels in different shapes. A) Straight channel; b) T-junction, with three pairs of isolated electrodes; c) Cross junction, with four pairs of electrodes

A two-mask process was employed, as indicated in Figure 1. Oxide was grown to prevent electrical contact to the Si substrate, and a seed layer of Cr/Au was used to initiate electroplating of Ni or Au. Uniform electroplating required current densities less than 3.5 mA/cm^2 . Bonding required less than $0.2 \mu \text{m}$ surface height variation, which was achieved by polishing with 50 nm Al₂O₃ particles. ICP-RIE was used with a second mask to etch a flow channel between the electrodes. Finally, a 0211 glass cover plate was bonded to the silicon wafer by HF bonding. Glass does not bond to the metal, so to ensure bonding near the flow channel an arrangement of *bonding nails* sticking through the metal deposit and polished flush with it was used.

4. Characterization and Discussion

Figure 2 shows photos of three typical realized device elements: a straight channel, a T-junction, and a cross junction. Electrodes in each channel segment can be individually

addressed. Electrode geometry near the intersections, illustrated in Figure 2b, was designed to prevent electrical leakage and cross-talk, by separating the electrodes by at least 75 μ m.

About 80 mA/mm² is required to obtain a flow of 0.12 mm/sec for chromatography. Tests on bubble current density thresholds at a Au surface (Figure 3) were done in 1M KNO₃, giving thresholds more than 20% higher than those reported for 1M NaCl [1]. These initial tests with the EMC design indicate it is capable of the flow rates needed to perform open tubular chromatography.

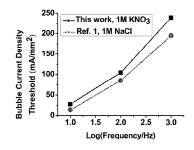


Figure 3. Bubble current density threshold for 1M KNO₃ solution as a function of frequency

A high-field AC magnet with a frequency above 60 Hz is not readily available commercially. Running a commercial 60 Hz magnet will dramatically reduce the field strength at a higher frequency, especially above 400 Hz. In this work, an oscillating magnetic field system has been designed and built that is based upon rotating permanent magnets. The rotor is composed of 32 permanent Nd-Fe-B magnets mounted alternately, face-to-face, as shown in Figure 4. It can generate an AC magnetic field of 0.45 T at frequencies from 0 to 960 Hz. It can be rotated at up to 7200 RPM with a vibration of less than a few thousandths of an inch. The frequency can be adjusted to control the pumping power, but care must be paid to minimizing the eddy

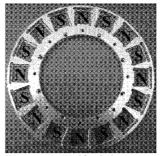


Figure 4. Half of the rotor, composed of 16 magnets, mounted alternately

currents that arise in the conductors due to the rotating field. A microchip was inserted into the gap between the opposing magnets. Figure 5 shows the unit, with a fluorescence detection system designed to probe the chip channels when inserted inside the rotor. A prism, above the chip, within the gap in the rotor plates, was used to reflect both the excitation and emission signals.

5. Conclusions

We have designed and fabricated an EMC which can generate more powerful MHD pumping in a channel with a 10 µm square cross section. The bubble threshold we have achieved will allow flow rates high enough for performing open tubular chromatography. The channel size is small enough for this chromatographic method to be effected. The challenges associated with fabrication have been overcome by optimizing electroplating conditions, developing polishing techniques for planarization of rough electrochemical surfaces, and reducing the ICP-RIE etching rates.

References

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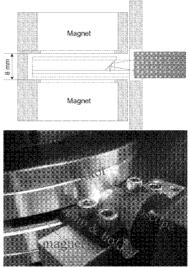


Figure 5. High speed MHD rotor and prism coupled LIF detector, 8 mm total gap width: 1.1 mm air gap, 0.5 mm top cover, 3.3 mm prism and reservoirs, 1 mm chip, 1 mm bottom cover, 1.1 mm air gap