A POLYMERIC PARAFFIN MICROPUMP WITH ACTIVE VALVES FOR HIGH-PRESSURE MICROFLUIDICS

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ABSTRACT

We present the potentially strongest micropump in sub-cm^3 size yet for microfluidics, using simple processes and materials such as epoxy, paraffin, and polyimide. Utilizing the large volume expansion associated with the melting of paraffin for actuation, a pump consisting of two active valves and one pumping chamber operated by three identical paraffin actuators has been realized. UV-curable epoxy, which encloses the paraffin, forms the channel structure and joins the glass cover, actuator membrane and resistive heaters for melting the paraffin, is the main construction material. With water as a pumping fluid and a 2 V drive voltage, the valves were subjected to pressures up to about 1 MPa without showing any leakage. A flow rate of 74 nl/min was obtained in normal operation.

Keywords: Active valves, High pressure, Micropump, Paraffin, UV-curing epoxy

INTRODUCTION

For micropumps, piezoceramics and silicon are the most commonly used materials. Also, micropumps often use two passive check-valves and a single pumping chamber. Valve-less pumps where nozzle and diffuser valves determine the flow direction have also been presented.[1] If low flow rates and high pressures are needed, other solutions with valves sustaining high pressure with little or no leakage are necessary. Furthermore, today many fluidic chips are small whereas the pumps used for controlling the fluidics on the chip are external (off-chip) and big. Using an on-chip pump decreases the channel length of the fluidic system enormously, rendering the system a much smaller dead volume. This also makes possible a totally integrated microfluidic system with a very small overall size.

There is also a need for simpler processes and economical materials, especially for rapid prototyping. New methods for simpler and faster processes have been presented, using liquid phase photopolymerization [2]. In this process a UV-curable liquid prepolymer, e.g. an epoxy, is selectively exposed to UV-light through a contact mask, which after rinsing uncured prepolymer results in a completed structure. This is a simpler and faster low temperature process than other relatively fast processes like for example SU-8 processing, which need more steps including spinning, prebaking, exposure, developing and postbaking.

Paraffin delivers a large volume expansion even at high counter pressures, has a melting temperature that can be tailored from -100 to +150 °C, is non-toxic and is readily processed by replication techniques such as casting, making it a highly interesting actuator material for microvalves and micropumps [3]. Previous work has been done with paraffin in valves using other designs and processes. For instance, materials like glass and metal have been used for further size reduction [4]. The actuator presented in this work is based on an actuator presented earlier by the authors [5].

In this work, we present a micropump with active valves that sustain high pressures without leakage. The pump is small, 2.5 x 1 x 0.3 cm3, giving smaller dead volumes and enabling on-chip integration. The pump is made of epoxy and utilizes the large and powerful expansion of paraffin - when melted - for actuation. In addition, the epoxy process used is a type of liquid phase photopolymerisation with simultaneous moulding, which gives very fast prototyping.

PUMP OPERATION, MATERIALS AND DESIGN

A cross section of the pump is shown in Fig. 1. Identical membrane actuators activate the pump chamber and the active valves. When the paraffin is melted by the heaters the normally open membranes seal the inlet and outlet holes and make a stroke in the pump chamber. The membranes return to their original form when the paraffin solidifies. By sequencing the actuation, a pumping action is achieved.

The design is based on effective heating by integration of the heaters inside the paraffin and allowing UV exposure from both sides of the non-transparent heater film in the fabrication process. The design also utilizes that the membranes in the actuators are made concave, also seen in Fig. 1, enabling a single layer channel structure.

An exploded view of the pump is illustrated in Fig. 2. The bulk structure is made of UV curing epoxy and the channel structure is sealed with a thin glass lid with drilled inlet and outlet holes. The resistive heaters are made by photolithography and wet etching of copper on a polyimide film i.e. a flexible printed circuit (FPC) board.

Liquid phase photopolymerisation was used to create the actuator structure with paraffin cavities and integrated heaters, as well as to create the channel structure. UV curing epoxy was also used when bonding the channel structure to the actuator membrane and a mask was used to avoid curing in the channels. The mask designs for channel structure fabrication and bonding are shown in Fig. 3. The membrane diameter is 2 mm and the channel is 100 µm deep and 1 mm wide.

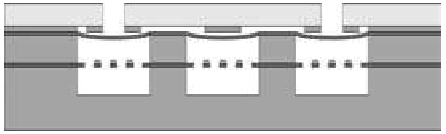


Fig. 1. Cross section of the pump showing the valves and the pumping chamber**.**

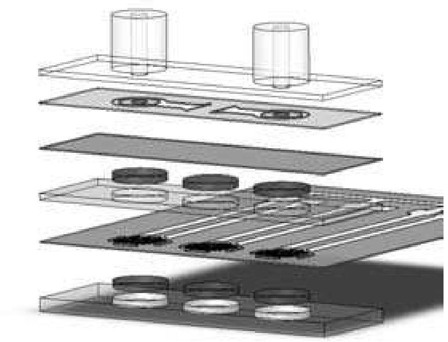


Fig. 2. Exploded view of the pump.

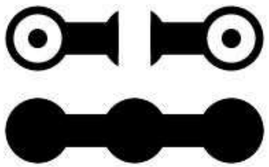


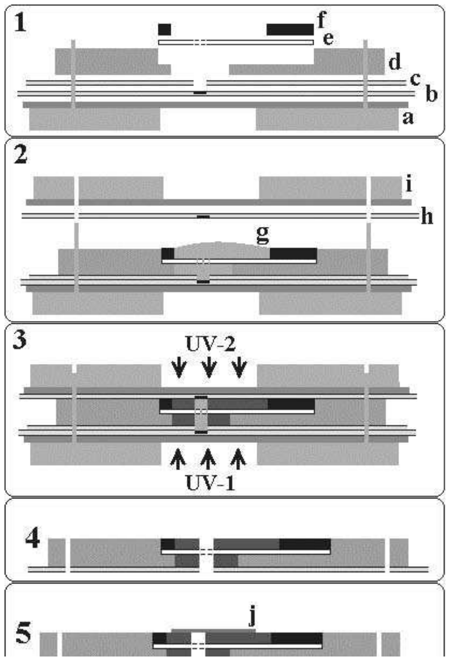
Fig. 3. Mask designs for creating the channel structure with liquid phase polymerisation (top) and bonding the channel structure to the actuators (bottom).

FABRICATION AND EVALUATION

Actuator

The fabrication steps are illustrated in Fig. 4. To build the actuator, a fixture for aligning masks and to create a stiff clamping was used. The fixture consisted of a brass bottom and top plate with alignment pins and glass covered through holes for UV light passage. (1) A mask for defining the paraffin cavity was placed in the fixture, followed by another mask with holes over the cavities. The hole mask was later used to protect the surroundings from contamination of paraffin. A teflon spacer, which at the same time works as a mould, was placed over the masks. The heater film was placed in this mould fixture and was fixed in position by a inner aluminium frame. The heaters, made of a polyimide (PI) and copper laminate (Espanex SB 12-50-12 AE), were patterned using standard photolithography (S1813 Shipley resist) and wet chemical etched in a sodium persulfate solution ($Na_2(SO_4)_2$), 320g/l water for 5 min at 50 ^oC. The through holes and the contour of the heater film were milled with a circuit board plotter (LPKF, Protomat C60). (2) The mould was filled with Epoxy (EPOTEK OG 198-50) and a mask for defining the paraffin cavities was placed on top of the mould. The fixture top was then added and secured with clips. (3) The epoxy was cured first in UV light (Dymax 5000EC System, 400 W metal-halide lamp) for 15 s from both sides. (4) The fixture and cavity defining masks were removed and uncured epoxy was rinsed with acetone. A cleaning rinse was made with ethanol before blow-drying with N_2. The actuator body was then clamped in the fixture and post-cured for 30 s from each side.

(5) An adhesive tape was used as membrane (PI-tape TESA 51408) and was attached to the actuator body. (6) The actuator body was then placed on a hot-plate (>60 ^oC) with the membrane side facing the hot-plate. Paraffin wax (Sigma-Aldrich 76228, melting point 44-48^oC) was placed over the actuator and melted into the cavities. After coagulation of the paraffin the hole mask was removed and (7) a backing of epoxy (EPOTEK OG 142-13) was applied and cured in UV light for 30 s. A 1 mm thick polydimethylsiloxane (PDMS) frame was used as mould. The actuator was removed from the teflon spacer leaving the inner frame bonded with the encapsulated actuator. Finally, copper wires, 100 µm in diameter, were soldered to the heater connector pads.



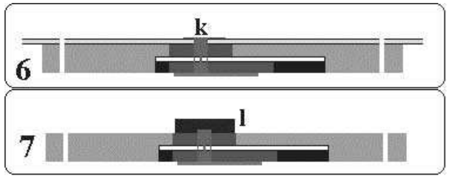


Fig. 4. Process scheme illustrating the actuator fabrication.

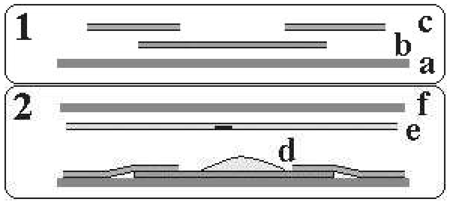
a – Brass frame with glass window and alignment pins
b – Shadow mask
c – Hole mask
d – Outer moulding frame
e – Heater film
f – Inner moulding frame
g – Structure epoxy
h – Shadow mask
i – Brass frame with glass window and alignment holes
j – Membrane film
k – Paraffin
l – Encapsulation epoxy

Channel structure and bonding

The fabrication steps of the channel structure and its bonding to the actuator are illustrated in Fig. 5. Fluid connection holes with a diameter of 0.6 mm were drilled in a 150 µm glass substrate or lid. (1) The lid was then added to a glass slide with a 50 µm tape (PI-tape TESA 51408) that also determined the height of the channel structure. (2) A drop of epoxy (EPOTEK OG142-13) was then pressed between the lid and the structure mask, taped on another glass slide. (3) The epoxy was selectively exposed with UV-light through the mask for 20 s (Dymax 5000EC system, 400 W metal-halide lamp). (4) After rinsing uncured epoxy with acetone and ethanol, the structure was post cured in UV light for 30 s.

(5) The channel structure was fixed with tape on a mask for UV bonding. This mask prevents epoxy from curing at the channel and valves. The actuator was then aligned and clamped on the lid with epoxy (EPOTEK OG198-50) in-between. (6) After exposure with UV-light through the mask, the uncured epoxy in the channel was rinsed with acetone, ethanol and finally water using a syringe with a rubber tube. The bond was then post cured for 30 s with UV-light, leaving the completed pump (7).

EPOTEK OG198-50 was used for its good adhesion to the membrane, and OG142-13 for its good suitability to make structures with lower heat generation during curing.



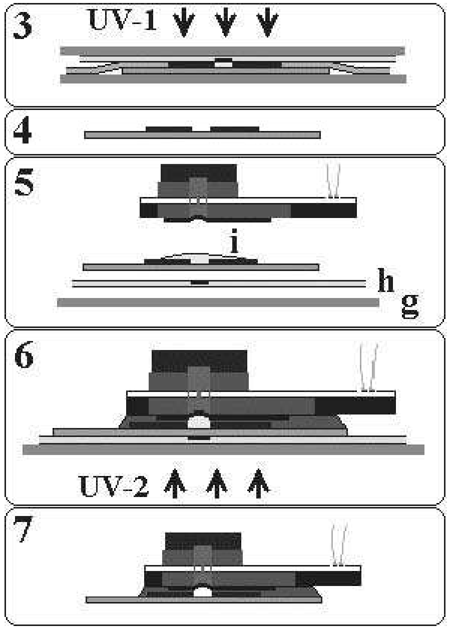


Fig. 5. Process scheme illustrating channel structuring and bonding.

a, f, g – Glass backings
b – Glass lid
c – Distance tape
d – Structure epoxy
e – Structure mask
h – Bonding mask
i – Bonding epoxy

The actuator was evaluated for itself with respect to frequency dependence on the stroke length. The stroke was measured using a length gauge (Heidenhain MT25).

For characterization of the valves, a syringe pump with water was used to apply a pressure over the valves, measured with a pressure gauge (Honeywell 13C5000PS5K with Upchurch Scientific pressure gauge tee) connected in-between. The flow rate was adjusted to give a constant pressure rise. For higher pressures an unclamped or, at the highest pressures, a clamped nanoport (Upchurch Scientific) was used.

Waveform generator and power supply (WFG400, PA19-14, FLC Electronics, Sweden) Waveforms with an amplitude of 2-3 V and period times of 30 s were used in a sequence to drive the pump at zero pressure difference. The flow rate was calculated from observation of meniscus propagation in the outlet tube.

RESULTS AND DISCUSSION

The completed pump is shown in Fig. 6. The lateral resolution reported for the rapid prototyping liquid phase photopolymerization in the reference [2] is 70 µm. In the present work, the main difference is the use of a UV-the smallest parts, 250 µm wide and 100 µm deep, of the channels, and has therefore not been evaluated further at this point.

The results from the actuator characterization are shown in Fig. 7. The results shows that the actuation frequency can be increased and still provide a useful stroke for pump and valve applications.

A flow rate of 74 nl/min was measured when running the pump for 30 minutes, Fig. 8.

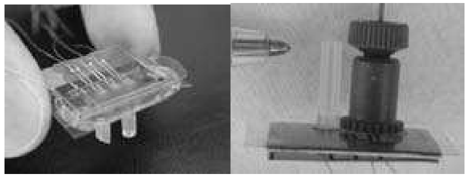


Fig. 6. Paraffin reservoirs with integrated heaters visible through the backside epoxy (left panel) and inlet/outlet silicone elastomer and nanoport connectors (right panel).

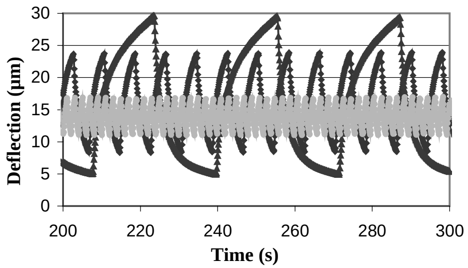


Fig. 7. Deflection of actuator membrane using square waves with frequency 1/32, 1/8 and 1/2 Hz at 2 V drive voltage. The zero level is the original non-activated state and the graph shows the behaviour at steady state after 200 s running period.

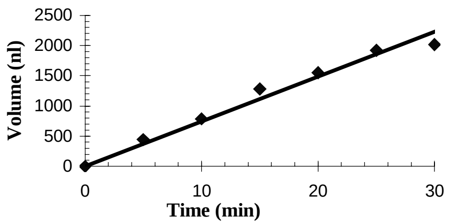


Fig. 8. Performance of the pump during 30 minutes of operation. The average flow rate was 74 nl/min.

Some aging effects, deriving from paraffin leakage under the membrane, were observed and can be evaluated further. These effects rendered in degradation of the pump performance after approximately one hour or longer total pumping times.

An unclamped valve could handle pressures up to 0.2 MPa, with a nanoport connection the valve was tested up to 0.35 MPa, and with a clamped nanoport pressures up to 0.92 MPa were measured. No visible leakage was our definition of ability to hold a pressure in this evaluation.

CONCLUSION AND OUTLOOK

The flow rate can be further increased by using better waveforms with higher frequencies. Also, an optimisation of the pump performance could surely be made by changing valve and pump chamber dimensions as well as channel and actuator sizes. Methods for bonding membranes can also be further developed.

The rapid prototyping technique used here could possibly be transferred to a very fast industrial manufacturing of paraffin actuators. The epoxy process can be developed further regarding resolution and aspect ratio.

A method for detecting very small leak rates can be utilized in the future. This could

The high pressure sustained by the valve presented in this work suggests the realisation of an extremely strong micropump in a near future.

Acknowledgements

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