Electrorheological-fluid-based microvalves

Xize Niu
Department of Mechanical Engineering, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, China

Weijia Wen[a)]
Department of Physics, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, China

Yi-Kuen Lee
Department of Mechanical Engineering, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, China

(Received 5 May 2005; accepted 12 October 2005; published online 5 December 2005)

We present the successful design and fabrication of push-and-pull microvalves that use a giant electrorheological (GER) fluid. Our multilayer microvalves, including the GER fluid control channel, the electrode, the flow channel, and the flexible membrane, are fabricated with polydimethylsioxane-based materials by soft lithography techniques. The GER effect is able to provide high-pressure changes in GER control channel so as to fully close and open an associated flow channel. The fast response time of the GER fluid and the push-and-pull valve design adopted assure fast switching time of the valve less than 10 ms and sound reliability. This GER-fluid-based microvalve has other advantages of easy fabrication and biocompatibility and is suitable for most microfluidic applications. © *2005 American Institute of Physics*. [DOI: 10.1063/1.2140070]

Microfluidic components are the key parts of micrototal analysis systems or the lab-on-a-chip systems widely used for deoxyribonucleic acid (DNA) sequencing, drug screening and delivery, in situ environmental monitoring and other biotechnology applications. Microvalves are fundamental building blocks in such devices to control microflows. Usually, the microvalves are controlled or actuated by piezoelectric elements,[1] pneumatic solenoid valves,[2] or hydrogels.[3] Recently, polydimethylsioxane (PDMS)-based valves and microfluidic devices have received intensive study due to their flexibility, biocompatibility, and easy fabrication using soft lithography.[2,4,5] However, an integrable and digitally addressable valve with fast response time in milliseconds is still lacking in microanalysis systems.

Electrorheological (ER) fluid is a type of colloidal suspension whose rheological properties can be varied through the application of an external electric field. Under an applied field on the order of 1–2 kV/mm, the ER fluids exhibit solidlike behavior, e.g., ability to transmit shear stress. The transformation from liquidlike to solidlike behavior is relatively quick, on the order of 1–10 ms, and reversible. Such a controllable variation of the rheological property implies a wide variety of potential applications, such as clutch, vibration damping, and valves.[6] Recently, a new type of ER fluid, with giant ER (GER) effect, has been developed.[7] The GER fluid, consisting of urea-coated 20 nm diameter nanoparticles (barium titanyl oxalate) suspended in insulating oil, e.g., silicone oil, can reach a yield stress above 200 kPa at an electric field of 5 kV/mm. This actually provides much benefit to operate the ER valve under the relative lower voltage and faster response time.

Micro-ER valves made with silicon and glass have been reported by Yoshida *et al.*[8] Nakano *et al.*[9] tested ER effects in SU-8 channels with indium tin oxide electrodes. However, these valves are designed mostly to control the flow of ER fluid itself, with two-port valves. To the best of our knowledge, a micro-ER valve that can control other liquid flows has not been reported to date. The main technological difficulties in designing microfluidic valves actuated by ER fluid are the arrangement of the parallel electrodes, effective actuation, and good bonding of different channel layers to an integrated microfluidic chip without any leakage. This letter describes our first effort to design a PDMS-based micro-ER valve to control the other flow channels (called the controlled channel) in microdevices that overcomes those difficulties mentioned above, while avoiding the complicated peripheries identified in the existing literature.[2]

Figure 1(a) depicts the schematic design of a PDMS-based multilayered *four-port* microvalve fabricated by soft lithography technique.[2,4,5] Compared to the *two-port* ER valve, the merit of the *four-port* microvalve is its integration of two independent flow channels, one of which is used for the flow of ER fluid, while another is for the flow of the controlled fluid, respectively. The fabrication process of such a valve chip started with the SU-8 mold made on glass wafers for the fabrication of the electrodes being interpolated into the ER channel on Layer 1. The material for electrodes is a composite synthesized by mixing PDMS (Dow corning 184) and carbon black (Vulcan XC72-R, Cabot Inc., USA) at a carbon concentration of 25% (w/w) to form so-called C+PDMS gel. The original gel was first injected into the mold pretreated with a demolding agent, squeezed by a plastic sheet, and then sandwiched between a pair of thick glass plates to ensure that the surface of electrodes could be flattened. After being cured at temperature of 80 °C for 1 h, a 300 μm thick C+PDMS hardened sheet was achieved. The sheet (film) resistance of the C+PDMS electrode was tested to be about 150 Ω/cm (ohms per square) by a four-point probe measurement. The zoomed-in image of C+PDMS

[a)]Author to whom correspondence should be addressed; electronic mail: phwen@ust.hk

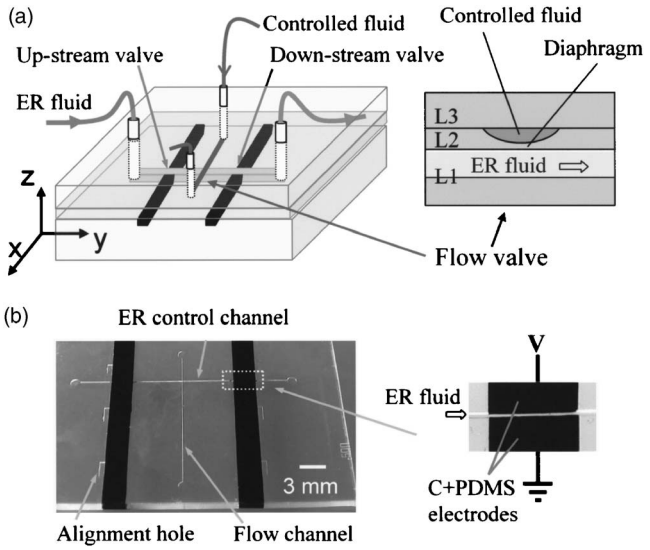

FIG. 1. Design, fabrication of the four-port micro-GER valve. (a) Schematic view of the four-port valve chip design. Layer 1 is the GER channel layer; Layer 2 is controlled flow channel layer; Layer 3 is the cover layer. The insert in the right depicts the cross section of different layers forming the flow valve. (b) Optical photograph of a made-up micro-GER valve chip. Insert shows the top-view image of the planner electrodes and the GER channel.

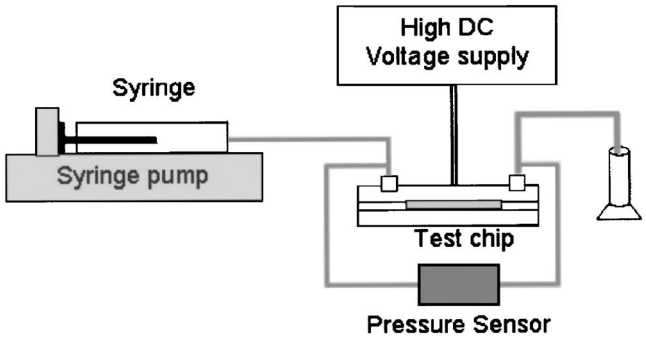

FIG. 2. Experimental setup for microvalve testing.

electrodes is shown on the right-hand side in Fig. 1(b), wherein the separation (gap) of two electrodes is 120 μm, the height and length are 300 μm and 3000 μm, respectively.

A second SU-8 mold was used to fabricate the ER channel with integrated C+PDMS electrodes. At first, the C +PDMS bulk electrodes were peeled off from the cured sheet described above, and then arranged on the second SU-8 mold along the ridge side of the channel mold under a microscope. More PDMS gel was poured onto the mold, placed under a vacuum for 10 min, and cured at 80 °C for 2 h to create Layer 1 on which the ER fluid channel (250 μm in height, 120 μm in width, and 2 cm in length) and the electrodes were tightly integrated.

The controlled liquid flow channel (located on Layer 2) was formed from a third mold. To avoid dead volume in the channel, an AZ4620 photoresist was used as the molding material. After the photoresist was developed and hard baked, a half-cylindrical channel was molded out on Layer 2. Treated with oxygen plasma (PDC-002, Harrick Sci. Corp., USA) for 1 min, Layer 2 was then bonded with Layer 1 to form a double layer, called Layer (1+2). It was later bonded with another 1 mm thick pure PDMS Layer (3) (used as a cover layer) to form a multilayered structure. Finally, two pairs of inlet and outlet ports connected to two channels were mounted on the top of Layer 3, which are shown in Fig. 1(a). The intersectional drawing of two channels (controlled fluid channel and ER-fluid channel) is depicted on the right-hand side in Fig. 1(a), wherein the controlled fluid and ER fluid flow along the x and y axes, respectively. It is noted from the drawing that a 35 μm thick flexible diaphragm (indicated in the drawing), formed at the intersectional area between two channels, separates ER-fluid channel and flow channel to ensure no interference between them when two fluids are in stationary flowing. Figure 1(b) shows an optically microscopic image of such a well-bonded microvalve chip (without inlet and outlet tubes), where the ER-fluid and controlled fluid channels are visible clearly.

The basic working principle of the ER-fluid-controlled microvalve is a push-and-pull actuation caused by the flexible diaphragm sandwiched between the ER channel and flow channel when an on-off voltage is applied to ER fluid. As shown in Fig. 1(a), a high-pressure source is connected to the ER channel from the inlet to push ER fluid in, while a vacuum source is connected to the outlet to suck ER fluid out of the channel. Two pairs of parallel electrodes are arranged along the ER channel forming an upstream valve and a downstream valve. If an adequate dc electric field is subjected to any pair of electrodes, the flow of ER fluid can be slowed or fully stopped due to the ER effect. The pressure in the GER channel between the two valves can therefore be modulated if the two valves are alternately opened and closed. Such a pressure change within ER channel will eventually result in the deformation of the flexible diaphragm with a vertical pull-and-push movement. Therefore, the liquid flow in the flow channel (Layer 2) will be controlled with the pressure change in ER channel. This push-and-pull mechanism has the advantage of faster response time compared with valves with only a push source, because the PDMS diaphragm has a low Young's modulus and cannot resume its original position very quickly by its own elasticity.

To assess the flow control characteristics, the ER effect of the GER fluid, and the pressure changes in the microchannels, a simple two-port straight channel is employed for these investigations. An experimental setup is depicted in Fig. 2, where a digital syringe pump (SP220i, World Precision Instrument, USA) is used to provide different constant flow rates, while a high dc power supply (Stanford Research Systems SRS PS310/1250V-25W), controlled by the LABVIEW© program, in conjunction with a high-power transistor-based control circuit, was connected to the electrodes. Pressure changes were measured with pressure sensors (Sensym ASCX15DN, Honeywell Inc., USA) by adjusting the nanoparticle concentration, the GER channel width (the distance of parallel electrodes), the length of the electrodes, as well as the electric-field strengths, respectively.

Figure 3(a) shows the experimental results of pressure change for respective pure silicone oil and GER fluids at different nanoparticle concentrations and mass flow rates carried out at zero-electric-field strength [here, the effective dimension of the testing channel is 0.24 mm(width) $\times$0.8 mm(height) $\times$20 mm(length), the length and separation of two parallel electrodes is 5 mm and 120 μm]. The data show that all GER fluid flows fall in the Newtonian flow

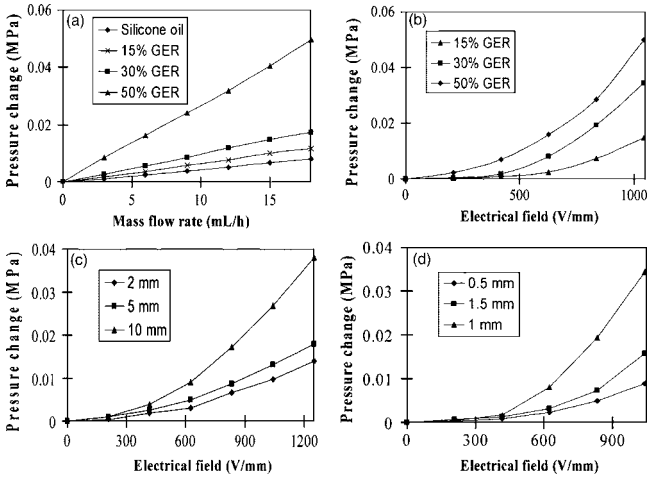

FIG. 3. Testing of pressure changes of GER fluid between the inlet and outlet of the microchannel ($W \times H \times L$: $0.24 \times 0.8 \times 20$ mm^3) under different conditions: (a) Pressure changes as a function of flow rate with different concentrations while the electric field is zero, (b) pressure changes due to ER effect as a function of the electric field at 6 mL/h mass flow rate, (c) pressure changes due to ER effect as a function of the electric field with different lengths of electrode, and (d) pressure changes as a function of the electric field with different widths of channels.

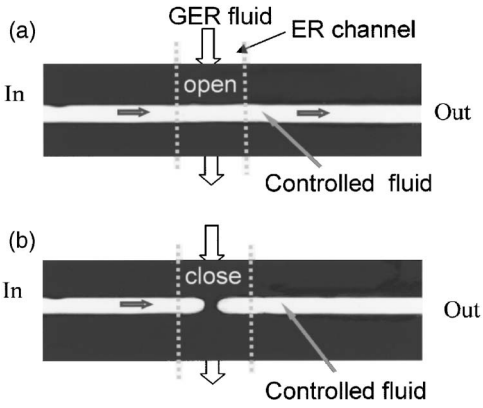

FIG. 4. Experimental microscope picture of (a) Open and (b) closed conditions of the controlled fluid channel.

regime for different concentrations. However, when an electrical field is applied, there is a distinct increase in the pressure changes, as shown in Fig. 3(b), tested at the same mass flow rate. The pressures increase exponentially as the electric-field increases resulted from the viscosity variations of ER fluid under different field strengths. From the curve, we note that even at a relatively low electric field of 1.2 kV/mm, the pressure changes due to the ER effect can reach up to 0.048 MPa for the ER fluid with a concentration of 50%. In our experiments, we found that, by further increasing the electric field up to 4 kV/mm, the GER fluid would change from liquid to near-solid state and result in the failure of the measurement since the value of the differential pressure was out of the measured range of the pressure gauge. Figures 3(c) and 3(d) show the pressure changes due to the ER effects in the microchannels with different lengths of electrodes and heights of channels, respectively. It is noted that the pressure changes in the same channel, and under the same electric field, increase linearly as the length of the electrodes increases.

As an example of realistic applications of our ER microvalve, the device shown in Fig. 1 was tested under on-off electric field to confirm its open-close functionality. In the experiment, the inlet and outlet of the ER-fluid channel were subjected to constant relative pressure of 0.041 MPa and −0.041 MPa, respectively. Deionized water mixed with the fluorescent dye (Rhodemine 6G) was pushed into the flow channel, with compressed nitrogen gas under a constant pressure of 0.02 MPa. Square waves of dc signals of 1500 V/mm that can fully stop the ER flow were applied to the upstream and downstream GER valves in contrast phase. When the upstream GER valve was open, the flexible diaphragm was pushed to the controlled fluid channel, which stopped the water flow. While when the downstream GER valve was open, the diaphragm was pulled back into the GER-fluid channel and the water flow was resumed. With a charge coupled device camera (Model No. 4912, Cohu Inc., USA) and Olympus IX70 inverted microscope, microscopic pictures, as shown in Figs. 4(a) and 4(b) were taken on the opened and closed statuses of water flow controlled by the GER valves. It is clearly seen that water flow could be fully controlled under an external electric signal. Such a push-pull microvalve is very reliable during days of operation because there are no mechanical friction and thermal problems.

Another experiment was performed to test the response time of the GER valve. A small amount of Rhodemine 6G powders, that has low solubility in silicone oil, was mixed into the silicone oil during sample preparation. Such silicone oil was pushed into the controlled flow channel and the movement of the dye particles was recorded at 30 frames per second. When the electric field of 1500 V/mm was switched on, the flow stopped promptly in the channel; when it was switched off, the flow resumed. By counting the bright dots from a single dye aggregate with a size of about 1–5 μm in one frame of the picture when the switching frequency of the electric field is higher than 30 Hz, we can approximately obtain the response time of on-off switching of the flow, which was found to be about 8 ms for one period, suitable for most microfluidic applications.

This work was supported by the Institute of Integrated Microsystems, HKUST (Contract No. I2MS01/02.EG08) and PROCORE Grant (Ref. No. F-HK18/05T). One of the authors (W.W.) thanks NSFC/RGC Research Grant N_HKUST605/04 for partial support. The authors thank Calbot Corp. for providing the free samples carbon black for this work.

[1] D. R. Reyes, D. Iossifidis, P. A. Auroux, and A. Manz, Anal. Chem. **74**, 2623 (2002).
[2] M. A. Unger, H.-P. Chou, T. Thorsen, A. Scherer, and S. R. Quake, Science **288**, 113 (2000).
[3] Y. Xia and G. M. Whitesides, Annu. Rev. Mater. Sci. **28** 153 (1998).
[4] D. Beebe, J. Moore, J. Bauer, Q. Yu, R. Liu, C. Devadoss, and B.-H. Jo, Nature (London) **404**, 588 (2000).
[5] S. Takayama, E. Ostuni, X. Qian, J. C. McDonald, X. Y. Jiang, P. LeDuc, M. H. Wu, D. E. Ingber, and G. M. Whitesides, Adv. Mater. (Weinheim, Ger.) **13**, 570 (2001).
[6] R. Stanway, J. L. Sproston, and A. K. EI-Wahed, Smart Mater. Struct. **5**, 464 (1996).
[7] W. Wen, X. Huang, S. Yang, K. Lu, and P. Sheng, Nat. Mater. **2**, 727 (2003).
[8] K. Yoshida, M. Kikuchi, J.-H. Park, and S. Yokota, Sens. Actuators, A **95**, 227 (2002).
[9] M. Nakano, T. Katou, A. Satou, K. Miyata, and K. Matsushita, J. Intell. Mater. Syst. Struct. **13**, 503 (2002).