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Magnetically Actuated Micropumps Using an Fe-PDMS Composite Membrane

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ABSTRACT

In this paper we describe a novel Fe-PDMS composite that can be used to create magnetically actuated polymeric microstructures. The composite is formed by suspending <10μm iron particles in polydimethylsiloxane (PDMS) at concentrations ranging from 25-75% by weight. Material properties and processing capabilities have been examined, and to demonstrate the usefulness of this material we have designed, fabricated and tested two prototypical micropumps that utilize an Fe-PDMS actuator membrane.

Keywords: MEMS, magnetic actuator, microactuator, micropump, PDMS composite, PDMS bonding, magnetic micropump

1. INTRODUCTION

Micro total analysis systems (μTAS) or lab-on-a-chip systems contain several components with functions including sample preparation, fluid control, analyte separation and detection, and data acquisition. In order to realize handheld μTAS, all of these components must be miniaturized, however fluid control components remain unsatisfactorily large relative to other subsystems. Although many different kinds of microvalves and micropumps have been presented, most researchers use benchtop syringe or peristaltic pumps to power their microfluidic devices, indicating a further need for reliable microfluidic actuators that can be easily integrated with other components. The goal of disposable microsystems for biomedical applications adds biocompatibility, low-cost, and low power consumption to the criteria for such an actuator. This paper presents a novel Fe-PDMS magnetic composite that, in application, can meet all of these requirements.

Since 1980, micropumps have been designed around almost every available MEMS actuation principle to yield piezoelectric, electrostatic, shape-memory alloy-based, thermal, thermopneumatic and magnetic devices. A recent and comprehensive review of these is presented in [1]. Among the many different microscale actuation mechanisms, magnetic actuation has certain advantages over other methods. In particular, it has been shown to produce large forces (10s of μN's) capable of affecting large displacements (100s of μm's) [2]. Previous magnetic micropumps have utilized several magnetic materials to achieve actuation, beginning with electroplated soft magnetic materials (e.g. Permalloy/NiFe) in the 1990s. For example, a silicon-based micropump with a Permalloy membrane was presented in [3]. It consisted of a 7μm-thick Permalloy film on a 17μm-thick, $8 \times 8 mm^2$ silicon membrane which could be deflected 23μm via the device's integrated inductors.

Hard magnetic or permanent magnetic materials with a high remnant magnetic moment, M_r, can be activated with lower-strength magnetic fields and will thus require lower power levels to actuate. Due to a lack of readily available and reliable deposition and micromachining processes, hard magnetic materials have not been used until recently. Since 2000 several magnetic micropumps using hard magnetic materials have been reported. These devices use bulk permanent magnets, permanent magnet powder composites, or ferrofluids.

The micropump presented in [4] used a single cylindrical permanent magnet, which was centered above the pump chamber and held in place with epoxy on top of the pump's PDMS membrane. A separate permanent magnet mounted on a micromotor shaft induced periodic deflection of the membrane, and pump flow was rectified through the use of two ball check valves. Similarly, a peristaltic design using three permanent magnets embedded in the pump membrane

has been presented [5]. The magnets alternate in polarity, and are sequentially actuated by three separate permanent magnets mounted on a micromotor shaft to generate peristaltic flow.

Bulk permanent magnets aside, another permanent magnetic material frequently used in micropumps is ferrofluid, which is a colloidal suspension of permanent magnetic particles in a liquid carrier. When no magnetic field is present, the magnetic moments of the particles are randomly distributed and the ferrofluid has no net magnetization. When a magnetic field is applied to the fluid, the permanent magnet particles quickly align to create a homogenous magnetic liquid that reacts in proportion to the gradient of the field and the magnetization value of the particles. Most ferrofluid-based designs implement the ferrofluid in contact with the liquid to be pumped in some sort of microchannel, and thus the two liquids must be immiscible. Oftentimes the externally actuated ferrofluid slug serves as a piston. In this way it pushes liquid through the microchannel and also seals the pump inlet and outlet as necessary during a pumping cycle. The microchannel can be linear [6, 7] or circular [8] but in either case the operational principles are equivalent.

The permanent magnet-based membrane micropumps described so far were successful in achieving large displacements and increased flow rates over previously reported devices, however the use of bulk permanent magnets or ferrofluids constrains device dimensions and can complicate the manufacturing process. As an alternative, magnetic PDMS composite actuator in which powdered permanent magnetic material was mixed with PDMS was reported recently [9, 10]. However, the magnet particles (NiFeB) had a large size (~ 100 μm) and had to be ground and then mixed with PDMS. In this paper, we introduce a novel Fe-PDMS composite that can be used to create magnetically actuated structures, particularly membranes for micropump or microvalve applications. The small iron particles (< 10 μm) render the PDMS magnetically susceptible, with the strength of the magnetic response being proportional to the concentration of iron used. The processing capabilities and material properties of the composite have been investigated, and prototypical micropumps have been designed, fabricated and tested to demonstrate the composite's applicability.

2. FE-PDMS COMPOSITE

The iron particles used in our Fe-PDMS are commercially available (Sigma-Aldrich; St. Louis, MO) and are shipped as 99.9+% <10um. No additional grinding or filtering was performed on the powder. The PDMS used is Sylgard 184 (Dow Corning; Midland, MI). PDMS is traditionally mixed at a 10:1 ratio of prepolymer base to polymerizing agent, cast as desired, degassed to remove air bubbles and ensure mold filling, and then cured. In our work the polymer mixing ratio was adjusted anywhere from 3:1 to 25:1. This allowed us to alter the PDMS' material properties and also helped to promote bonding between polymeric parts.

To create the Fe-PDMS composite, iron particles are added to the PDMS polymerizing agent and mixed thoroughly by hand. This mixture is then added to the PDMS prepolymer base and mixed again. Initial dispersion of the iron particles in the less-viscous polymerizing agent helps prevent aggregation of the particles during suspension. The composite is most useful when the weight ratio of iron ranges from 25 to 75%. Below 25% the concentration of iron is too low to support magnetic actuation, and above 75% the integrity of the PDMS becomes compromised.

Fe-PDMS retains many of the properties of pure PDMS, including moldability, elastomeric behavior, good adhesion to silicon and glass substrates, and the ability to permanently bond to other PDMS parts. The composite has several advantages over the previously discussed means of magnetic microactuation. In contrast to micropump designs that utilize permanent magnets embedded in a PDMS membrane, our Fe-PDMS composite simplifies device design in three ways: 1) membrane thickness is not limited by the dimensions of a magnet; 2) bulk permanent magnets or Permalloy pieces do not have to be positioned within the prepolymer or otherwise integrated into the membrane; and 3) the composite structure will have homogenous and isotropic material properties. Additionally, low-cost micron-scale iron powders are commercially available and do not require any additional preparation.

2.1 Material Properties

Mechanical testing was performed to determine the Young's modulus and Poisson's ratio of Fe-PDMS and to determine how these values relate to the composite's iron concentration. Samples of 10:1 PDMS were cast in a custom mold so as to have the required dog-bone shape for tensile testing. Samples contained zero, 50 or 75% iron by weight. Poisson's ratio was measured to be 0.5 for all samples; results for the Young's Modulus measurements are presented in Table 1.

Although there is no chemical interaction between the two components in our composite, Fe-PDMS was seen to have a higher Young's modulus than regular PDMS, and the value increases with Fe concentration. Additionally, subtle changes in the material properties arise if the orientation of the iron particles within the PDMS is nonrandom (unpublished data). The base and polymerizing agent mixing ratio has a large effect on the modulus value of regular PDMS: there is an exponential decrease in the modulus value as the amount of polymerizing agent decreases [11].

The PDMS base and polymerizing agent have different densities however their mixing ratio has a small effect on the polymer's final density. Also, addition of the iron powder does not greatly change the volume of the composite. The density of PDMS at different mixing ratios has been published [11] however empirical validation of these results proved difficult. No effort has been made to measure the density of the composite, however the results in [11] can be used with the density of iron to approximate the density of any particular blend of Fe-PDMS.

Samples of 50wt% Fe-PDMS were tested in a magnetometer and the magnetization curve obtained is presented in Figure 1. With negligible hysteresis present, we can conclude that the ferromagnetic properties of iron are unchanged by their suspension in PDMS. This absence of hysteresis is also expected to help simplify future numerical modeling of the composite and its magnetic response.

Table 1. Measured mechanical properties of Fe-PDMS composite.

Fe (wt%)	*Young's Modulus (MPa)*	*Poisson's Ratio*
0	2.14	0.5
50	2.56	0.5
75	2.91	0.5
Pure Fe	2.11×10^5	0.5

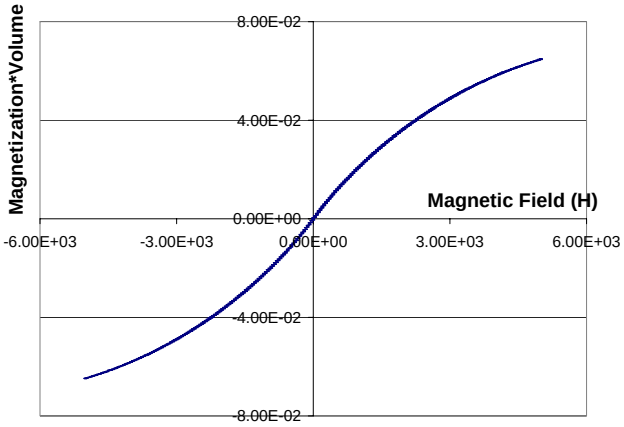


Figure 1. Magnetometer results for 10:1 50wt% Fe-PDMS.

2.2 Processing capabilities

Distinct areas of Fe-PDMS can be cast within lager areas of pure PDMS. This can be done to create confined areas of magnetic susceptibility within a membrane structure (i.e. directly over a pump chamber) or to preserve the optical transparency of a membrane in areas that are not intended for actuation (e.g. in some interrogation region of the microfluidic path). To create these areas, pure PDMS is cast but not cured. The desired volume of Fe-PDMS is transferred by pipette into the region of interest. If no external magnet is present, Fe-PDMS will remain where it is deposited with some diffusion into the surrounding pure PDMS. If an external magnet is placed near the region of deposition, the Fe-PDMS will be confined to that location and the iron particles will align with the magnetic field lines present. Figure 2 shows one quadrant of a 1cm-diameter circle of Fe-PDMS constructed within a 5mm-thick membrane of regular PDMS by this method.

Our Fe-PDMS composite has exhibited molding capabilities similar to those of pure PDMS. Figure 3 depicts a microchannel (60 µm width, >100 µm depth) molded from Fe-PDMS to demonstrate this. The diameter of the iron particles used does not limit the minimum moldable feature size since PDMS alone will fill any voids smaller than the particle size. Fe-PDMS cannot be reliably spincoated; the result in an uneven, though approximately radially symmetric, distribution of iron particles.

Although the majority of iron particles in the composite are fully encapsulated in PDMS, it is expected that some may partially breach the surface and thus be vulnerable to oxidation. To verify this, a 20 x 20 x 2 mm^3 sample of composite

was immersed in regular water for 48hrs. Microscopy revealed three small areas of oxidation on the surface. Although minute amounts of surface oxidation would not greatly alter the material or magnetic properties of the composite, in a μTAS application this could lead to sample contamination. Thus, to prevent surface oxidation an Fe-PDMS membrane can be protected in two ways. First, the composite membrane may be sandwiched between very thin layers of regular PDMS, or regular PDMS may be spincoated onto the composite surface to protect any surface-bound iron particles. Second, a surface coating such as Parylene (polyparaxylylene) may be applied, but doing so will change the material properties of the composite structure and complicate design. The former method is therefore suggested, and protected against oxidation in a repeat experiment.

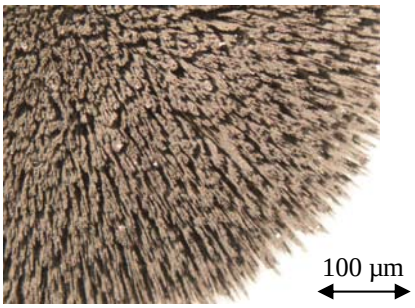


Figure 2. Magnetically aligned Fe particles within PDMS.

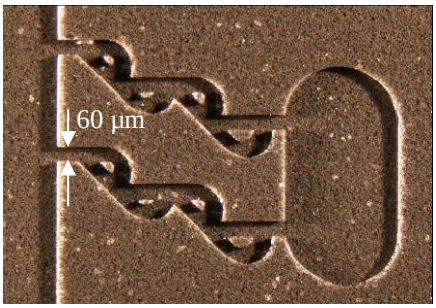


Figure 3. Micromolded Fe-PDMS composite. Channel width is 60 μm.

2.3 Fe-PDMS bonding

PDMS readily adheres to itself non-permanently, and this adhesion can be used to facilitate device assembly (see section 3.3 on pump assembly). The highest ratio Fe-PDMS used in this research, 25:1, is slightly tacky to the touch and has the best adhesive properties when in contact with itself, PDMS or Fe-PDMS of other ratios, silicon, glass, or any other smooth surface. Adhesion is signficantly weaker between lower-ratio parts under the same circumstances.

In order to bond our device's multiple layers to one another, a novel PDMS bonding process had to be developed. Without access to an oxygen plasma setup (typically employed to activate PDMS surfaces), published bonding techniques could not be replicated. Serendipitously, the bonding process we developed encourages PDMS layers of dissimilar mixing ratios—and thus dissimilar material properties—to be bonded, which supported the use of a rigid pump chamber and more flexible pump diaphragm in constructing our micropump prototypes.

Pump chambers were made from 3:1 PDMS whereas pump diaphragms were made from 25:1 PDMS. Diffusion of polymerizer from the 3:1 part to the 25:1 part does not support bonding alone: a thin layer of polymerizer was placed between the parts to aid bonding. A microcontact transfer technique was developed to apply this thin layer. A 1 ml droplet of polymerizing agent is placed near the end of a 2.5 cm-wide glass microscope slide. A duplicate slide is then placed on top, perpendicular to the bottom slide. It is pressed upon until the liquid film edge meets the slide edges, at which point the top slide is slid away to reveal a 2.5 cm^2 thin layer of polymerizing agent on both slides. The pump chamber is carefully placed on the film, picked up, and placed in contact with the pump diaphragm. The layers are then clamped or weighted and baked overnight in an 80°C oven.

In microfluidics it is often desirable to bond PDMS to a glass substrate in order to seal a microchannel or provide a rigid base for the device. We have developed a glass-PDMS bonding procedure based on the above method that works in situations where a PDMS or Fe-PDMS structure is to be attached to a planar (not micropatterned) substrate. To attach a 25:1 Fe-PDMS part to a glass substrate, a layer of 3:1 PDMS is spincoated onto the substrate. The following spincoating parameters reliably produced a micron-range thickness: 500 rpm @ 100 rpm/s for 10s followed by 1000 rpm @ 300 rpm/s for 30s. The thin 3:1 layer is cured and will be strongly bonded to the glass substrate. The part to be attached is then mounted on the PDMS-coated glass using the microcontact transfer technique described above.

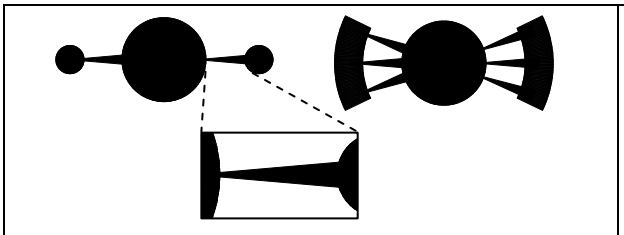
3. PUMP DESIGN AND FABRICATION

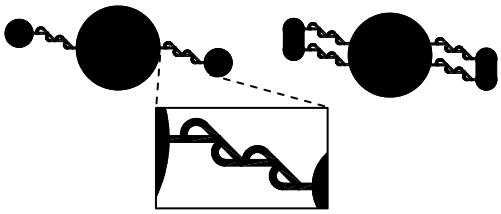
3.1 Pump design

Two types of valveless micropumps were designed, fabricated and tested to demonstrate the usefulness of our Fe-PDMS composite. For flow rectification, the first uses a diffuser/nozzle combination and the second uses a Tesla valvular conduit. The primary figure of merit for such structures is their fluidic diodicity, or ratio of flow in the positive direction to the amount of backflow for a single pump cycle. The diodicity of these structures is heavily defined by their geometries, and critical values are listed in Table 2. Both styles of pumps were built in two different sizes to have a pump chamber diameter of either 6 or 12 mm. Here we present the performance of the small diffuser/nozzle pump and the large Tesla pump. Other designs, including a circular peristaltic pump, have been fabricated but not fully tested.

The pumps are operated by periodically moving a permanent magnet under the pump chamber. Doing so causes oscillation of the composite membrane, which compresses the pump chamber and generates fluid flow. Continuous and periodic actuation with an NdFeB magnet was achieved using a miniature DC gear motor and crankshaft. A DC power supply provided electronic control over the pump's speed. In this arrangement, the stroke volume and maximum flow rate of pump is dependent on several operational parameters, including magnet location, magnet field strength, and motor speed.

Table 2. Pump designs and dimensions.





Diffuser Angle	10°	*Channel Width*	60 µm
Max. Diffuser Width	650 µm	*Curve Radius*	550 µm
Min. Diffuser Width	175 µm	*Shortest Flow Path*	6720 µm
Diffuser Length	2716 µm	*Intersection Angle*	45°
Chamber Diameter	6 mm	*Chamber Diameter*	12 mm
Depth	660 µm	*Depth*	660 µm
Chamber Volume	74.6 mm^3	*Chamber Volume*	18.6 μm^3
Final Pump Dimensions	20 × 15 × 4 mm	*Final Pump Dimensions*	40 × 15 × 4 mm

3.2 Pump fabrication

Pump fabrication can be divided into four tasks: SU-8 mold construction, pump chamber molding, Fe-PDMS membrane molding, and device assembly (see Figure 4). Pump designs were transferred to a photoresist layer spin-coated on a glass substrate to serve as pump chamber molds. In this process, the photoresist thickness determines the depth of the pump chambers and features, and as such the SU-8 process was adjusted in order to realize very thick (0.5-1.5 mm) photoresist layers. Besides not spin-coating the SU-8, significant deviations included SU-8 degassing, extended pre- and post-exposure bake times, gradual temperature ramping, and the use of ultrasonic agitation during development.

Pyrex glass substrates (2 × 2 × 0.125in) supported the photoresist molds. Substrates were cleaned in Piranha solution (1:3 H_2O_2:H_2SO_4), rinsed with deionized water, and dehydrated on a 120°C hotplate. SU-8 2035 photoresist (MicroChem; Newton, MA) was dispensed by weight to produce approximate thicknesses of 0.5, 1.0 and 1.5 mm. The photoresist-coated substrates were then degassed for approximately one hour, and large bubbles were manually burst if necessary. The density of SU-8 can be assumed to be 1.20 g/cm^3, however a correction factor should be used since considerable solvent loss occurs during degassing and baking.

The SU-8-coated glass substrates were baked on a digital hotplate preheated to 65°C for 180min to ensure sufficient heat transfer through the thick glass substrate and photoresist layer. The temperature was then gradually ramped to 95°C. Thirty minutes after the hotplate temperature reached 95°C it was turned off and allowed to gradually cool to room temperature with the substrates still in place (approximately 90min).

An exposure energy of 800 mJ/cm^2 was administered to the 1.0 and 1.5g samples, while 1000 mJ/cm^2 was used on the 2.0g sample. After exposure, the substrates were placed on a room-temperature hotplate which was then set to 65°C. Five minutes after reaching 65°C, the hotplate was ramped again to 95°C. The substrates were allowed to bake at 95°C for 30min before turning off the hotplate to cool to room temperature with the substrates in place. The SU-8 features were developed with sonication, which greatly reduced the necessary development time and provided well-defined high aspect ratio features. Minimizing the SU-8's contact with the developer solution also helped prevent delamination of the photoresist from the substrate.

Pure PDMS (3:1) was cast approximately 1 mm over the SU-8 mold features to create the pump chambers. Meanwhile, sheets of Fe-PDMS (25:1, 60wt%) were cast between glass plates to ensure a uniform thickness and then cut to serve as pump diaphragms. To do so, a mold with the desired thickness is created and Fe-PDMS is poured into the mold cavity. A transparency film is carefully laid over the surface to avoid any air bubbles. With the film in place, a rigid substrate is placed on top and the entire assembly is clamped or weighted and cured. The pumps described here used a 1mm-thick Fe-PDMS diaphragm, although thinner sheets could be reliably produced by this same method.

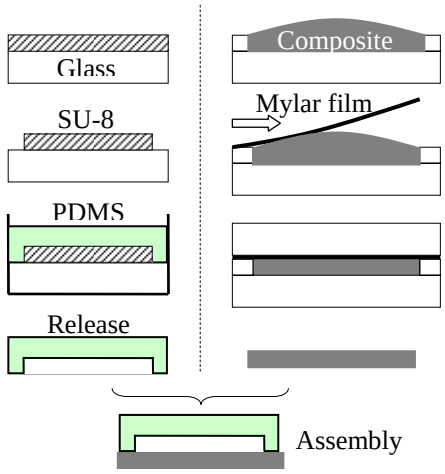


Figure 4. SU-8 pump chamber mold construction (left), Fe-PDMS membrane construction (right), and assembly.

3.3 Pump assembly

The two primary pump parts—the pump chamber and actuation membrane—can be assembled in several different configurations as shown in Figure 5. Since PDMS readily adheres to itself, simply aligning the parts and press-fitting them together will suffice for some applications (Figure 5a). In this configuration the pumps can withstand low-pressure testing and usage, and the pump parts can be easily disassembled for inspection, cleaning, or interchanging. For higher-pressure testing and usage, the parts can be clamped together between two rigid substrates and maintain these same qualities (Figure 5b). For permanent assembly, the parts can be bonded together using the technique described herein. If desired, the pump's fluid connections can be moved to the membrane side in order to free the actuation side, as in Figure 5c. Since the membrane is much softer than the pump chamber, doing so requires the tubing to be supported by an additional layer of PDMS or some other substrate. The membrane cannot contact any other material above the pump chamber or it will adhere and potentially not deflect during actuation.

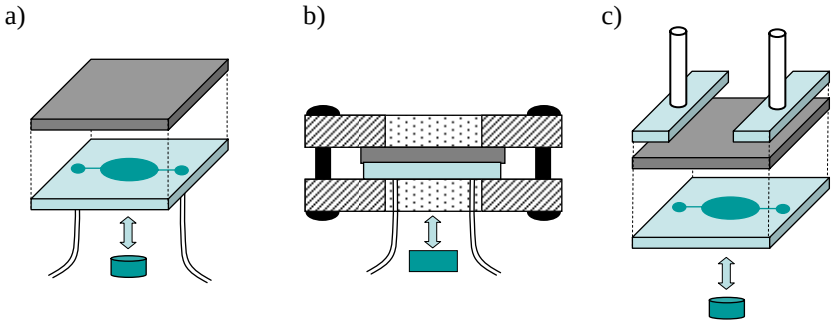


Figure 5. Different pump assembly methods.

Note that since Fe-PDMS can only displace in the direction of the actuating magnet, actuation must take place on the side opposite from the composite membrane. Photographs of two assembled micropumps are shown in Figure 6.

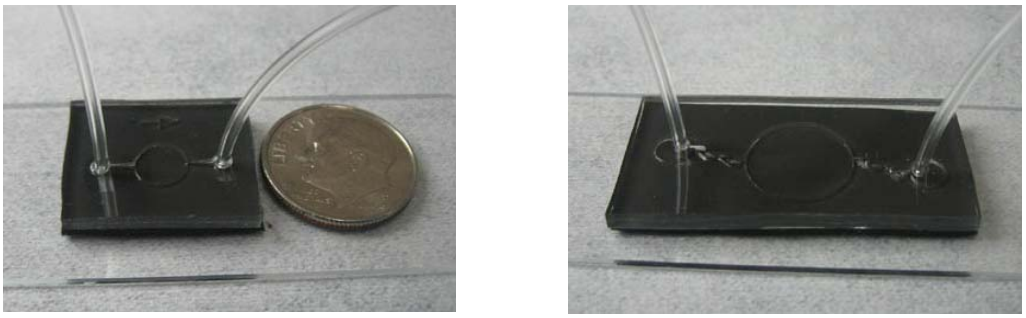


Figure 6. Photographs of the assembled diffuser/nozzle (left) and Tesla (right) micropumps.

4. PUMP PERFORMANCE AND DISCUSSION

The experimental setup used to test the two micropumps is shown in Figure 7. A crankshaft was used to transform the rotational motion of a DC micromotor into translational motion of a magnet. Ni-coated NdFeB magnets with a thickness of 6.33mm were used to test both micropumps. For the 6mm diffuser/nozzle pump, the magnet was 9.5 mm in diameter; for the 12 mm Tesla pump the magnet was 12.7 mm in diameter. These magnets were placed at the end of the crankshaft, centered over the pump chamber, and came within 1 mm of the pump bottom.

The pump inlet was submerged in a reservoir of deionized water. Both the inlet and outlet tubes were made of Tygon and had an inside diameter of 0.02 in. The pumps were syringe-primed such that the outlet tube was only partly filled with water. During pumping, the progression of water in the outlet tube was measured and the volume of water moved was calculated.

In oscillating displacement pumps such as these, flow is pulsatile due to the periodic nature of the magnetic actuation. Also, flow rate is proportional to the frequency and amplitude of the membrane actuation. The actuation frequency is controlled via the electric motor voltage. The actuation amplitude can be controlled in three way: 1) the size or strength of the magnet employed can be changed; 2) one can vary how close the magnet gets to the pump; or 3) the concentration of Fe in the actuator membrane can be changed.

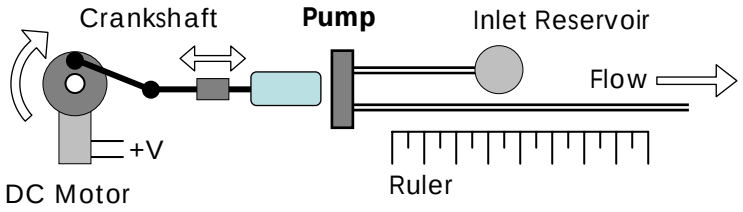


Figure 7. Experimental setup for micropump testing, as viewed from above (pump inlet and outlet are on the same plane).

Figure 8 and 9 are the flow rate versus actuation frequency curves for 12 mm Tesla micropump and 6 mm diffuser/nozzle micropump, respectively. The flow rates for the micropumps ranged 0-14 µL/min and 0-35 µL/min each. Smaller diffuser/nozzle micropump showed higher flow rate than the larger Tesla micropump. It was because the flow rate through the large Tesla pump was limited by the conduit dimensions. The large chamber could not fill entirely before the next actuation. In both micropumps, the flow rate increased as the actuation frequency increased and after certain maximum values, the flow rate decreased. This is because the actuation amplitude is decreased at higher actuation frequency. The frequency at which the flow rate is maximized varies according to the pump design and dimension. Also note that the larger standard deviations were shown for the higher flow rates.

The valveless pumps presented here cannot operate against any backpressure; a net reversal in fluid flow will be observed if the pump outlet is raised above the inlet. Additionally, the pumps are not self-priming and fluid must be introduced into the system before operation. These two issues can be resolved by replacing the current flow rectification structures with a pair of passive check valves.

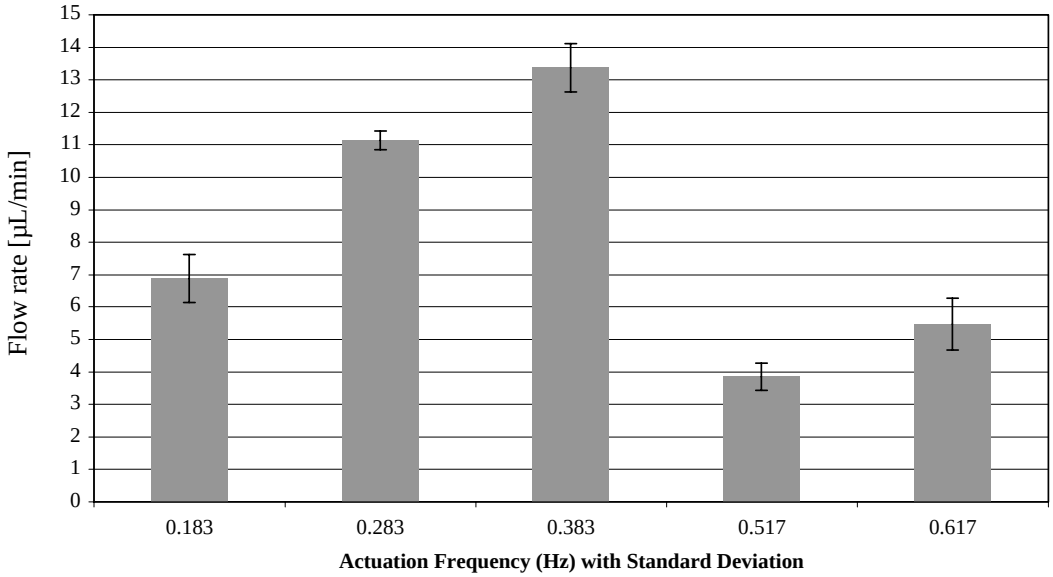


Figure 8. Flow rate versus actuation frequency for 12 mm Tesla micropump.

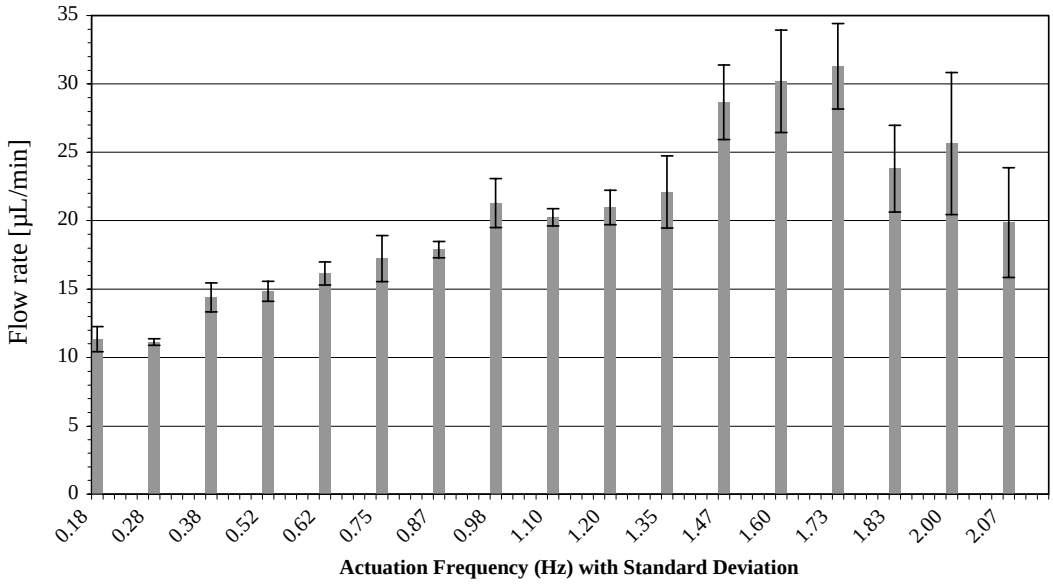


Figure 9. Flow rate versus actuation frequency for 6 mm diffuser/nozzle micropump

5. CONCLUSIONS AND FUTURE WORKS

The material properties and processing capabilities of a novel Fe-PDMS composite have been examined as a potential membrane actuator for microfluidic devices. The mechanical and magnetic properties of the composite layers with different composition have been measured. The molding and bonding characteristics have been investigated and the optimum processing conditions have been explored.

The applicability of our Fe-PDMS composite material has been successfully demonstrated through the prototypical micropumps discussed here. Micropumps with diffuser/nozzle design and Tesla conduit design have been fabricated, and their pumping performances have been evaluated with water. More prototypes including peristaltic micropumps are presently under evaluation.

Plans for future work include further experimentation with the composite properties as well as optimization of the micropumps' performance. Further materials testing as well as numerical modeling or computer simulation will enable this optimization. Ideally the moving magnet used in the experimental setup here will be replaced with a square-wave driven electromagnet. Also, check valves may be incorporated to bring the fluidic diodicity of the pumps closer to unity. Check valves will give the pumps some resistance to backpressure, and may also allow the pumps to be self-priming.

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