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Toward orientation-independent design for gas recombination in closed-loop electroosmotic pumps

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Abstract

Direct current operation of electroosmotic pumps consumes water and generates electrolysis gas bubbles that can block the working electrode and build up pressure in the pump housing. The formation of the electrolysis gases can degrade pump performance and can lead to failure. Although catalysts have been used to recombine the electrolytic gases, current designs are sensitive to orientation relative to gravity making them inappropriate for many portable applications. Here we propose a gas recombination system design that operates independent of pump orientation. The design uses Nafion-sheathed electrodes, a catalyst chamber, and an osmotic membrane to capture and recombine electrolytic gases as well as return the liquid recombination products to the electrolyte reservoir. The pump was tested for 24 h at six major orientations with constant current fluxes. The measured gas chamber pressures and applied pump potentials suggest that the recombination system is functional at all orientations for driving currents of 3 mA and less.

Keywords: Electroosmotic pump; Orientation independent; Gas recombination

1. Introduction

Electroosmotic (EO) pumps have no moving parts and are capable of generating high flow rates (on the order of 0.13 ml/(min cm^2 V) [1]) and large pressures (on the order of 1000 Pa/V [2]). Electroosmotic pumps offer some advantages over other micropumps for microchannel cooling applications [3,4] and integrated bio-analytical systems [5]. EO pumps have been fabricated from a variety of materials including packed and sintered of glass beads [6], porous glass frits and monoliths [7–10], polymer beads [11], porous silicon [1], as well as planar pumps microfabricated in silicon and glass [12]. Laser and Santiago reviewed a large array of micropumps including EO pumps [13].

EO pumps require applied potentials greater than the electrochemical reaction overpotential resulting in Faradic current and formation of electrolytic gases. The electrical current and resultant electrolytic gas generation have been discussed in Yao et al. [7] and Yao and Santiago [8]. The majority of EO pumps use buffered aqueous solutions resulting in hydrogen and oxygen electrolysis gases. These gases can block EO pump channels reducing effective pump area and flow rate. In a closed-loop system, electrolysis consumes the working fluid and (if the system is sealed) increases the system pressure resulting in poor long-term performance and unstable operation. For portable applications such as miniature fuel cells [14] methanol pumping via EO and handheld lab-on-a-chip devices EO pumps must operate for long periods (over 6 h) independent of pump orientation.

Electrodes have been separated from pumping media by an ion exchange membrane [15–17], including Nafion [18–20] as well as gel or liquid salt bridges [21]. These strategies have been shown to be effective in preventing bubbles from blocking the pumping media thereby improving long-term pump

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performance. In these designs the gases are vented to the environment and the working fluid is consumed. Yao and Santiago [8] incorporated platinum-based catalytic meshes that recombined hydrogen and oxygen to water thereby conserving the working fluid. In the latter design, electrolysis bubbles form on the platinum wire electrodes and rise and flow to the catalyst via buoyancy. This recombination strategy fails under many orientations and when the catalyst mesh is wet by the working electrolyte (by momentary changes in orientation or splashing upon sudden movement). A recent study on ac electroosmotic pumping [22] asserts that bubble generation at the electrodes can be avoided by using asymmetric pulses with zero net electromigration current. Their approach is explained in terms of the non-linearity of the low-potential (e.g., <100 mV), low-frequency (few Hz or less) region of the current–voltage curve of electrochemical systems; and they achieve order 10 nl/s flows with a few volts externally applied potentials and 1 nA currents. We expect such pumping to be limited to very low-pressure capacity (e.g., a 3 μm hydraulic diameter channel with a −100 mV zeta potential and an effective voltage drop of order 1 V along its active length generates order 300 Pa). This also translates to very low thermodynamic efficiency compared to dc EO pumping, given a non-negligible load applied to the pump. This approach's applicability to high flow rate applications (say 0.01 ml/s or greater) is an open question.

In this study, we demonstrate a sealed, closed-loop electroosmotic pump integrated with Nafion-sheathed electrodes, a catalyst recombiner, and osmotic membrane for long-term, orientation-independent pumping. This pump operates continuously in multiple orientations without refilling or replacement of the working fluid. We sheathe platinum wire electrodes in Nafion (DuPont Fuel Cells, Wilmington, DE) ion exchange membranes to capture electrolytic gases and route them to the catalyst mesh—an idea first presented by Mosier et al. [20]. These ion exchange membranes are largely impermeable to bulk liquid flow and gases, but allow electromigration of anions [23]. The key feature and novelty of the current design is the compact, stack configuration of a gas chamber, a PTFE-membrane-encapsulated recombiner, and a DI water outlet fitted with an osmotic membrane to recover water back into the main flow loop. The application of an osmotic membrane for this purpose was first proposed by Santiago and Zeng [10].

2. Experimental setup and theory of operation

In this section we present the design and theory of operation of a closed-loop, orientation-independent EO pump with gas recombination. Fig. 1a shows the pump system schematic. The EO pump assembly is based upon the design presented by Yao and Santiago [8] and will not be repeated here. The pumping medium is a 40 mm diameter, 4.5 mm thick porous glass frit (ROBU Glasfilter-Geraete GmbH, Germany) with an effective pore radius of 1.1 μm. In this work we sheathe platinum wire pump electrodes (0.13 mm o.d., Omega, Stamford, CT) with two layers of Nafion ion exchange membrane tubes (0.84 mm

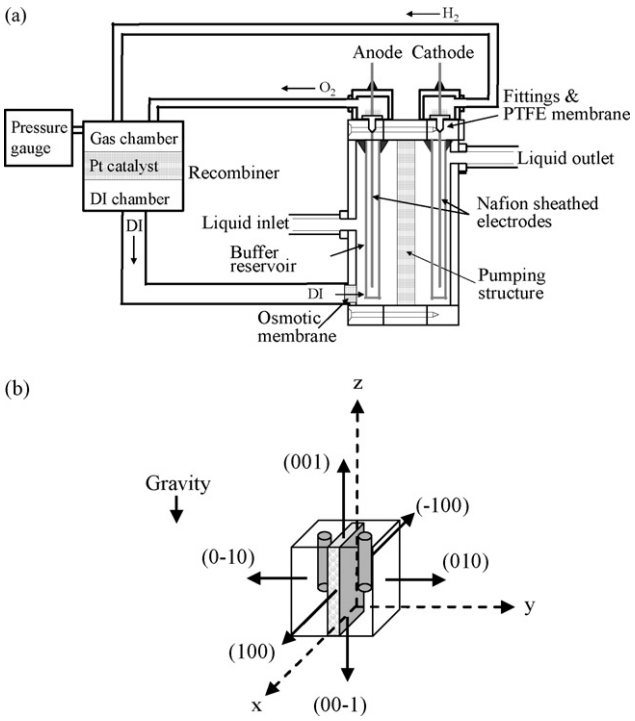

Fig. 1. (a) Schematic of closed-loop, orientation-independent EO pump system with gas recombination. The gases form at the platinum wires that are encased in the Nafion ion-exchange tubing. The gases are routed to the gas chambers through the Nafion tubes. The PTFE tubing prevents the passage of liquids to the catalyst mesh. Hydrogen and oxygen recombine at the mesh surface forming pure water. The water diffuses through the osmotic membrane rejoining the working electrolyte. (b) Orientation coordinate system for pump operation.

o.d., 0.65 mm i.d. and 1.34 mm o.d., and 1.07 mm i.d., Perma Pure LLC, Toms River, NJ). The electrolysis gas bubbles form on the platinum electrode and are routed to the catalyst recombiner through the Nafion tubes. Nafion is a porous polymer with anionic surface groups that makes it permeable to cations and polar compounds [24]. The material has a high flow resistance to anions and nonpolar compounds and therefore serves as a barrier to the electrolytic gases. Nafion conducts positively charged ions and weakly conducts negatively charged ions and electrons. We found that a single layer of the Nafion material resulted in some bubble formation on the frit side of the Nafion barrier (probably electrolytic gases) on the outside of the membrane. We therefore opted to use a second layer (a second, concentric tube) of Nafion around the electrode to minimize this effect. Electrolyte liquid was wicked into the annular gap between the Nafion tubes and the central tube space. We sealed one end of each electrode tube with epoxy and epoxied the other end to a polymer HPLC fitting (Upchurch, Oak Harbor, WA). Polytetrafluoroethylene (PTFE) membranes were glued atop of the HPLC fittings. PTFE membranes are permeable to the electrolysis gases yet inhibit the passage of electrolyte solution. The HPLC fittings connecting the electrode regions to the recombiner assembly separate the electrolyte reservoirs from the gas chambers.

As shown in Fig. 1a, stainless steel gas tubes connect the PTFE-fitted HPLC fittings to the recombiner assembly. The recombiner consists of a platinum catalyst partially coated with

teflon particles (AECL, Mississauga, Canada) where hydrogen and oxygen gases mix and recombine forming water. The partial teflon coating on the platinum enhances water removal via capillary forces [25].

In our design, we further completely sealed the catalyst within a PTFE membrane package that keeps the catalyst layer dry. This PTFE–catalyst–PTFE assembly separates the recombiner into a gas chamber and a chamber for recombined DI water. A PTFE tube connects the deionized water (DI) chamber to the buffer reservoir for returning recombined water. As shown in the schematic, this return passage is fitted with an osmotic membrane (GE, Trevose, PA) between the return tube and the reservoir. This osmotic membrane prevents buffered electrolyte solution from flowing back to the DI chamber. More importantly, this component creates an osmotic pressure between the DI-filled tube and the main flow loop which pumps newly formed DI back into the circulation loop [10]. The entire gas control system allows Faraday reactions at the electrode surfaces to feed ion current to the EO pump, while keeping electrolysis gases out of the main flow loop. The electrolysis gases are fed back to the PTFE-shielded-catalyst assembly and the newly formed DI water is pumped back into the main flow loop via osmotic pumping. Pressure differences due to electrolysis and osmosis make the entire electrolytic gas/water recovery process flow independent of gravity. A schematic defining six primary unit normal vectors of the pump is shown in Fig. 1b. In this Cartesian system, gravity is aligned in the negative z-axis (0,0,−1). We adopt a pump orientation nomenclature where the top of the pump is directed towards one of the six primary unit normal vectors. For example, Fig. 1b shows the pump upright in the (0 0 1) orientation where the top face is aligned opposite to gravity (e.g., (0,0,−1) denotes an upside down pump). We here present performance data for the EO pump at these six primary orientations.

A dc power supply (Kepco, Flushing, NY) provided constant current through the EO pump system. The electrolyte solution was 5 mM sodium borate buffer (Sigma–Aldrich, St. Louis, MO). During each experiment we monitor the total pump voltage and the gauge pressure (Omega, Stamford, CT) of the gas mixture chamber. All the signals were collected by a data acquisition multiplexer (Agilent, Palo Alto, CA) and recorded on a PC. In constant current mode, the voltage applied to the pump electrodes is a measure of the total electrical resistance on the pump circuit (e.g., so that high voltage may indicate bubbles in the main flow loop). The pressure in the gas chamber is a measure of the activity of the catalyst.

3. Experimental results and discussion

We tested our EO pump system without the catalyst assembly in the (0 0 1) orientation and with the catalyst assembly at all six primary orientations. Fig. 2a shows the gauge pressure of the gas chamber at 3 mA constant current operation over a 24 h period. Without the catalyst, the pressure increased continuously and reached 1 atm in 7.5 h. When we deactivated the applied potential the pump pressure decreased at a rate that was less than 5% of the generation rate. We attribute this decrease

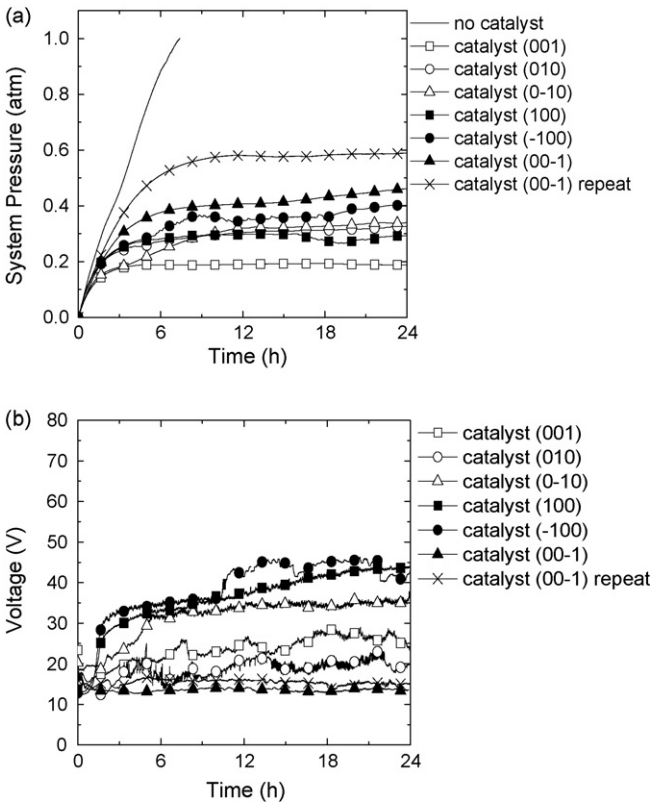

Fig. 2. (a) Differential pressure in EO pump housing operated at 3 mA for 24 h in six orientations and without catalyst (the experiment without a catalyst was in the (0 0 1) orientation). (b) Electrical potential applied to EO pump under constant current control at 3 mA. This potential is a measure of total pump resistance at each orientation.

in pressure to low level leaks in the fittings and seals as well as permeability of the pump housing to hydrogen and oxygen. We repeated the sans catalyst experiments several times and estimated that the pressure–time slopes were reproducible to within 2.5%.

We then characterized the pressure in the gas chamber with the catalytic recombiner assembly. Fig. 2a shows the chamber pressure at each orientation. The pressure at each orientation initially increases and then asymptotes. The steady state pressure and transient time scale vary. For example, at an orientation of (0 0 1) the pressure reaches a steady state value of 0.19 atm after 3 h. The (0,0,−1) orientation has the highest pressures (∼0.6 atm) and takes more than 6 h to reach a steady value. Note that the two (0,0,−1) orientation experiments have different steady state pressures, and there is a small, nonzero slope for orientations (0,0,−1), (−1,0,0), and (1 0 0). A steady state pressure is reached when the rate at which gases are produced equals the rate at which gases are recombined and to a lesser extent, leaks from the structure.

We hypothesize that the equilibrium pressure of each orientation is largely a function of the catalyst active area. The active area of the catalyst is effectively reduced due to some accumulation of DI water on the catalyst surface. This reduction in catalysis rate results in increased pressure in the recombination region. The partial flooding of the catalyst is affected by gravity and therefore depends on the orientation of the system. The

(0,0,−1) "upside down" orientation or any "side" orientations (0 1 0), (0,−1,0), (1 0 0), and (−1,0,0) seem to most strongly reduce the catalyst area and result in higher equilibrium pressures. Although the steady state pressures are not identical in each experiment, the roughly asymptotic behavior to a steady state pressure suggests that the gas separation and recombination are functional. We observed no gas bubbles outside of the double Nafion sheaths in any of these experiments, with and without the catalyst assembly.

We also monitored the electrical potential applied across the pump. The pump was operated in 3 mA constant current mode and the applied potential represents the integrated electrical resistance of the EO pump system. Fig. 2b shows the applied potential over a 24 h period for the six orientations tested. In all orientations except (0,0,−1), the applied potential rapidly increased over the first few hours and then fluctuated around a stead state value. The lowest voltage (suggesting the lowest electrical resistance) was obtained with the pump upside down in the (0,0,−1) orientation. The large resistances for the side orientations (e.g., 0 1 0, 1 0 0, −1,0,0) are probably due to some trapping of bubbles in the Nafion tubes. The (0,0,−1) orientation, shows the highest steady state pressure (∼0.6 atm). This high pressure compresses the gas volumes in the Nafion tubes, yielding the smallest bubbles and minimal blockage (so electrode-to-buffer resistance is minimum).

In portable applications such as a laptop microprocessor cooling or handheld bio-analytical devices, an EO pump may experience a sequence of orientations in series. The sequence of orientations may also affect performance of the EO pump system. We consider two sequences of four orientations: the first two runs were (0 0 1), (0,−1,0), (0,0,−1) to (0 1 0); the third run was (0 0 1), (0,0,−1), (0,−1,0) to (0 1 0). The pump was operated at a constant current of 3 mA and the orientation was rotated in 6 h intervals.

Fig. 3 shows the gas chamber pressure as a function of time during the sequences. The data shows that the chamber pressure remains bounded over the two orientation sequences. Note that there is a significant decrease in the pressure when the pump is rotated away from the upside-down (0,0,−1) orientation. This suggests that gases were quickly released from the Nafion tubes and flowed to the catalyst assembly to recombine into water. Overall, these preliminary experiments suggest that the pump can be oriented at virtually any orientation and through a variety of orientation sequences.

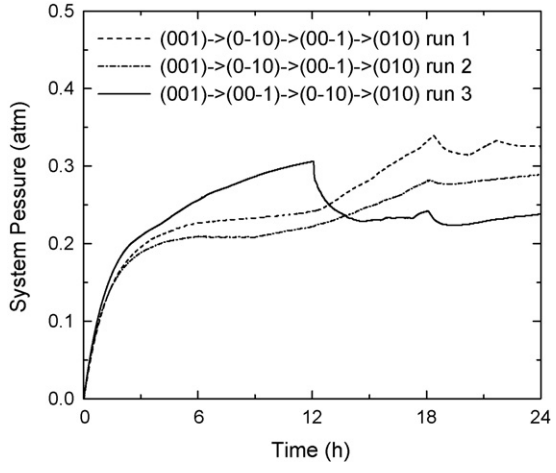

Fig. 3. Pressure traces for the pump system operated at 3 mA. For these data, we changed the system orientation every 6 h in the sequences shown in the legend.

We also tested the pump at higher driving currents. We performed four experiments at orientation (0 0 1) and 5 mA. At this higher current we observed intermittent voltage spikes as high as 200 V, which was the voltage limit of the power supply. These voltage spikes indicate large electrical resistances most likely caused by smaller effective ion migration cross-section area due to gas accumulation in the Nafion tubes. Over multiple realizations we found that the initial spike occurred at times ranging roughly from 2 to 14 h. After the initial spike, further voltage spikes were frequent and led to eventual failure of the pump.

4. Conclusions

We present an orientation-independent gas control and recombination device for electroosmotic pumps. The design uses Nafion-sheathed electrodes fitted with PTFE membranes, a PTFE-sealed catalyst chamber, and an osmotic membrane. The system prevents electrolysis gases from entering the main flow loop of the pump, and recombines these to form pure water. The system returns newly formed water back into the main flow loop via osmotic pumping. The pump and recombination system were tested for 24 h at six major orientations with constant current fluxes of 3 mA. The system was also shown to function as it was rotated through two-, four-position orientation sequences over a 24 h period. At a higher driving current (e.g., >5 mA), the pump and recombiner system failed due to accumulation of gases in the Nafion tubes. Important future work would include a study of proton conduction and diffusion through Nafion as a function of driving potential, membrane thickness, and species concentration. Overall, the experiments presented here suggest that a sealed, orientation-independent electroosmotic pump loop is possible.

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Biographies

Mr. Che-Wei Lin has a masters of science degree in mechanical engineering from National Chiao Tung University, Taiwan. During the project described in this paper, Mr. Lin was a researcher in the Industrial Technology Research Institute's (ITRI) in Taiwan. ITRI is a non-profit research and development organization with the goal of accelerating the industrial development of Taiwan. Mr. Lin led ITRI projects in microsystems research and liquid-cooled microelectronics. He is an expert in microfabrication techniques including silicon bulk micromachining and LIGA. Mr. Lin was a visitor of the Stanford Microfluidics Laboratory from August 1, 2004 to January 31, 2005.

Shuhuai Yao received her BS degree in engineering mechanics from Tsinghua University, Beijing, China, in 2000, and the MS and the PhD degrees in mechanical engineering from Stanford in 2001 and 2005. Her graduate research focused on the development of microfluidic and MEMS devices for electronic cooling systems and bio-analytical applications. She has published the theoretical and experimental research work of miniaturized, high-flow rate electroosmotic micropumps and the integration of a recombination system for control of electrolytic gases. Currently, she is a postdoctoral fellow in Lawrence Livermore National Laboratory, where she is engaged in developing ultrafast microfluidic mixers for studies of protein folding kinetics.

Professor Jonathan D. Posner earned PhD (2001) degrees in mechanical engineering at the University of California, Irvine. In addition, he spent 18 months as a fellowship student at the von Karman Institute for fluid mechanics in Rhode Saint Genese, Belgium. As a research scientist at Neophotonics Corporation he developed an aerosol based, laser pyrolysis nanoparticle reactor for optical films and fuel cell electrolytes. He also spent 2 years as a postdoctoral fellow in the Microfluidics Laboratory. Dr. Posner is currently an assistant professor at Arizona State University in the Department of Mechanical and Aerospace engineering and director. His interests include microscale transport phenomena, fluid dynamics, electrokinetics, and optical diagnostics as they apply to the physics and design of micro/nanofluidic bioanalytical and energetic devices. Applications of his research include: development of protein separation microdevices, nanomaterial toxicity, microfluidic fuel cells, and ion-channel biosensors. Dr. Posner was honored for his excellence in experimental research by the von Karman Institute for Fluid Dynamics and his work has appeared on the cover of *Applied Optics* and the *Journal of Microfluidics and Nanofluidics*.

Alan Myers received his BS degree in metallurgical engineering from the University of Illinois at Chicago in 1984 and his PhD degree in materials science from the University of Illinois at Urbana Champaign in 1991. He joined the etch group of the Portland Technology Development division of Intel in 1992 and is currently a member of the External Programs group. After working from 2002 through 2005 as an Intel Researcher in Residence at Stanford University investigating advanced cooling solutions for microprocessors, he is now on assignment at IMEC in Belgium working on developing Extreme Ultraviolet Lithography for high-volume manufacturing operations. Alan has published over 20 archival journal and conference articles and currently holds 20 patents with an additional 18 patent applications pending with the US patent office.

Professor Juan G. Santiago has PhD in Mechanical Engineering from the University of Illinois at Urbana-Champaign (UIUC); where he received five fellowships as a doctoral candidate. He was a Senior Member of the Technical Staff at the Aerospace Corporation (1995–1997), where his work included the development of flow diagnostics for micronozzles. Prof. Santiago received a Ford Foundation Postodoctoral Fellowship (1997), and worked as a Research Scientist at UIUC's Beckman Institute (1997–1998). Santiago is an associate professor in the Mechanical Engineering Department at Stanford. He specializes in microscale transport phenomena, electrokinetics, and microfluidic system design. His research includes the optimization and development of novel microsystems for pumping liquids, on-chip electrophoresis, sample concentration methods, and miniature fuel cells. The applications of this work include microfabricated bioanalytical systems

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