# Rapid prototyping of microfluidic switches in poly(dimethyl siloxane) and their actuation by electro-osmotic flow

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**Abstract.** This paper describes a procedure for rapidly and conveniently prototyping microfluidic devices that are useful with aqueous solutions. A design (with diameters of channels  $\geqslant 20~\mu m$ ) is created in a computer-aided design program and printed at high resolution on a transparency. This transparency is used as a mask in photolithography to create a master in positive relief photoresist: casting poly(dimethyl siloxane) (PDMS) against this master yields a polymeric replica containing a network of bas-relief channels. The channels are closed and sealed irreversibly by oxidizing the replica and another flat substrate (PDMS, glass, silicon, silicon oxide) in an oxygen plasma and bringing the two surfaces into conformal contact. Oxidation of the polymer allows the formation of a seal without using adhesives; it also generates channels that support electro-osmotic flow (EOF) and fill easily with aqueous solutions. Two microfluidic devices—a fluidic switch and a side channel flow controller—have been fabricated using this rapid prototyping methodology. These devices were tested using aqueous solutions as the test fluid and actuated by EOF.

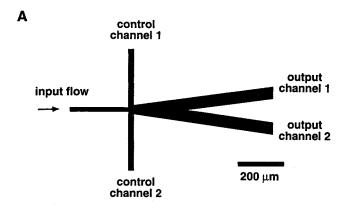
# 1. Introduction

We have developed a rapid prototyping methodology that makes it possible to fabricate microfluidic components and systems rapidly and conveniently in poly(dimethyl siloxane) (PDMS). This methodology has four key components: (i) a method for generating patterns of channels rapidly that have dimensions (20–100  $\mu$ m) that are in the range used normally for microfluidic applications; (ii) a material of fabrication (the polymer, PDMS) that has useful properties for microfluidic systems that use aqueous solutions as the working fluid; (iii) a process (oxidation in a plasma) that modifies the surface properties of the polymer in a way that allows the microfluidic channels to support electro-osmotic flow (EOF) and that causes them to fill easily and spontaneously with liquids, such as water, that have high surface energies; (iv) a procedure based on the same plasma oxidation that seals the channels during fabrication that produces leak-free joints. We have illustrated this methodology by fabricating two simple microfluidic components—a fluidic deflection switch and a side channel flow controller-in oxidized PDMS.

The recent development of microfluidic systems has been motivated in large part by a substantial interest in microscale total analysis systems ( $\mu$ TAS) [1–4].  $\mu$ TAS are small, integrated, often chip-based devices that perform many of the fundamental functions of analytical chemistry

laboratories—sample preparation, separation, detection, quantification and read-out. These systems are based on microfluidic components such as pumps, valves and channels [5,6]. New components and new methods of fabricating these components and of integrating them into  $\mu$ TAS are needed.

Polymers are attractive materials for fabricating microfluidic systems [7–9]. PDMS, in particular, is tough, optically transparent, amenable to fabrication using a number of procedures, and inexpensive [7, 10-13]. polar, impermeable to aqueous solutions and well-suited for working with aqueous, biological samples. It is swelled by many organic solvents and hence unsuited for use with non-polar organic fluids. We have developed a method for rapidly prototyping microfluidic systems in PDMS, and have previously benchmarked the methodology by fabricating a micro-capillary electrophoresis ( $\mu$ CE) system [10], a reconfigurable diffraction grating [12], and liquidcore waveguides [14]. The method is based on creating patterns of positive relief photoresist (a 'master') by contact photolithography using a high-resolution transparency as the photomask [15]. A prepolymer of PDMS is then cast against the master; curing the polymer and releasing it from the master yields a replica containing a network of microfluidic channels in bas-relief. The channels are closed and sealed irreversibly simply by oxidizing the replica and a flat (PDMS,



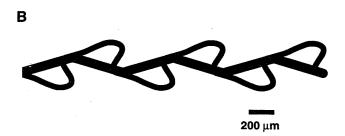


Figure 1. (A) Detail of the design of the fluidic deflection switch. An input flow of liquid enters the switch. Hydrostatic or electro-osmotic pressure is applied at two control channels. Depending on the relative pressures in the two control channels, the input flow is directed into one of the two output channels.

(B) Detail of the design of the side channel fluidic controller.

glass, silicon, silicon oxide) in a plasma and bringing them into contact. Here we present two microfluidic components that we have fabricated using this system.

We based our design of the microfluidic deflection switch on fluidic amplifiers that were proposed and fabricated on large size scales (widths of channels  $\sim 0.5-1$  mm) in the 1960s [16, 17]. Figure 1 shows an enlargement of the computer-aided design (CAD) of channels that we used to fabricate this device. At low Reynolds numbers (Re < 2300, in the laminar flow region), the direction of fluidic flow in the main channel of such a device (figure 1(A)) varies continuously with the pressure arising from fluidic flow at the control channels, and therefore the main flow can be directed into one of two output channels: this device is known as a jet deflection amplifier. Recently, Furlan and Zemel [18] created microscopic (diameter of narrowest channel = 50  $\mu$ m) version of the jet deflection amplifier by micromachining silicon and using nitrogen gas as the test fluid. They also demonstrated a wall attachment amplifier that relies on a turbulent flow attaching to one of the walls of the main channel; this phenomenon only occurs at high Reynolds numbers, so was not observed in the low Reynolds number flows of liquid in the microscopic devices described here.

We based our side channel flow controller on the design of a fluidic rectifier proposed by Tesla [19]; an enlargement of the design of our device is shown in figure 1. As in the design of Tesla, this device is composed of a main fluidic channel with several side channels branching off on both sides of this main channel. In this type of device, the flow of fluid from the main channel into the side channels only occurs in one

direction, if Reynolds numbers are high enough to make the momentum of the fluid important in determining its behavior.

An important aspect of the microfluidic devices described here is that the polymer is oxidized in an oxygen plasma. Oxidation introduces silanol (SiOH) groups at the surface of the naturally hydrophobic polymer and makes the surface of oxidized PDMS resemble the surface of silica in its wettability properties [20]. This change in the surface chemistry that occurs on plasma oxidation has three consequences for microfluidic systems. First, when two oxidized surfaces are brought into contact they seal irreversibly through the formation of siloxane (Si-O-Si) bonds [10]. Second, the surface of the polymer is hydrophilic so the channels can be filled easily with aqueous solutions, a process that can be difficult in microfluidic systems fabricated from hydrophobic polymers. Third, under neutral or basic solutions, the silanol groups are deprotonated (SiO-), the surface of the polymer is negatively charged, and the channels support EOF toward the cathode [10]. EOF has been used widely as the method for pumping fluids in many microfluidic systems; we have also used EOF to actuate the switch and flow controller.

### 2. Experimental section

#### 2.1. Rapid prototyping of masters

Designs of networks of microfluidic channels (diameters  $>20 \mu m$ ) were created in a CAD program (Freehand 7.0) [15]. High-resolution transparencies were produced (Herkules PRO image setter, 3386 dpi, Linotype-Hell Corporation, Hauppauge, NY) from the CAD files with the design clear and the background ink. Transparencies were used as masks in photolithography on negative photoresist (SU-8 50, Microlithography Chemical Corporation, Newton, MA) spin-coated onto silicon wafers to create masters. We spin-coated at 5000 rpm for 20 s to create the features of photoresist, and hence channels, 50  $\mu$ m deep. After development in propylene glycol methyl ether acetate (Aldrich, Milwaukee, WI), the masters were placed in a desiccator under vacuum for 2 h with a vial containing a few drops of tridecafluoro-1,1,2,2-tetrahydrooctyl-1trichlorosilane (United Chemical Technologies, Bristol, PA). This fluorosilane passivates the surface of the master and facilitates the removal of cured PDMS.

## 2.2. Molding

A 10:1 mixture of PDMS prepolymer and curing agent (Sylgard 184, Dow Corning, Midland, MI) was stirred thoroughly and then degassed under vacuum [10]. Tall glass posts (~7 mm diameter, ~2 cm tall) were placed on the master to define reservoirs (figure 2). The prepolymer mixture was poured onto the master (and around the glass posts) and cured for 1 h at 65 °C. After curing, the PDMS replica was peeled from the master and the glass posts were removed.

#### 2.3. Sealing

A PDMS replica was rinsed in ethanol and dried under a stream of argon; a glass slide was cleaned in heptane and ethanol and then dried. The two substrates were placed in a plasma cleaner (PDC-23G, Harrick, Ossining, NY), and oxidized for 1 min. Immediately after removal from the plasma cleaner, the substrates were brought into conformal contact and an irreversible seal formed spontaneously [10].

# 2.4. Filling the channels and EOF

The channels were filled by filling a reservoir with an aqueous solution of 25 mM Tris(hydroxymethyl)aminomethane (Tris)–192 mm Glycine (Gly) and pulling vacuum on the other reservoirs [10]. The liquid in the reservoir of the main channel also contained 1  $\mu$ m diameter fluorescent beads (Polysciences Inc, Warrington, PA). EOF in the control channel was induced by applying a potential between the two reservoirs connected by this channel through Pt electrodes connected to a high-voltage power supply (CZE1000R, Spellman, Hauppauge, NY).

#### 2.5. Detection

The motion of the beads in the fluidic flow was followed using a microscope (Leica DMRX, Heidelberg, Germany) and an Hg lamp and fluorescence filters. Fluorescence images of the beads were grabbed from a charge coupled device (CCD) camera connected to a frame grabbing card.

#### 3. Results and discussion

# 3.1. Rapid prototyping of microfluidic devices

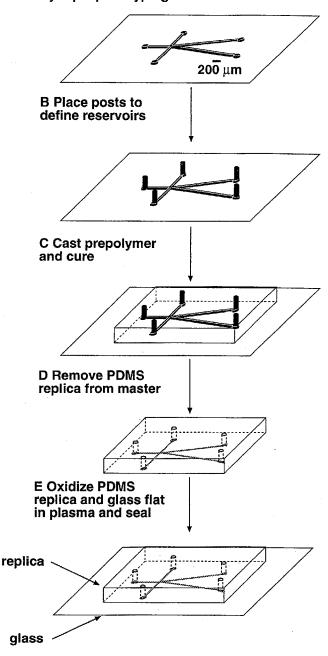
Figure 2 outlines the scheme for the rapid prototyping of microfluidic devices. Experimental details of the rapid prototyping method are given in section 2 and in a paper describing a  $\mu$ CE device [10]. For the devices presented here, we sealed the PDMS replicas, which contained a bas-relief pattern of channels, against glass substrates. The plasma oxidation and sealing seemed relatively insensitive to the details of the procedure, provided first, that sealing of the PDMS and glass was performed soon (<24 h) after curing the PDMS and second, that the two surfaces were brought into contact within a few minutes (ideally, less than a minute) after they were removed from the plasma chamber. If the freshly oxidized surfaces were allowed to stand for longer periods, they would lose their ability to form a seal. We have not defined the processes involved in this time-dependent change in surface properties.

# 3.2. Flow of liquids in microscopic channels

We induced the flow of liquids in the channels in two ways: hydrostatic pressure and electro-osmosis. In both systems we maintained flows at low Reynolds numbers (Re  $\sim 0.005-0.25$ ). Flow by hydrostatic pressure in a channel was induced simply by having a difference in the height of liquid between two reservoirs connected by the channel. The surface of oxidized PDMS is negatively charged under neutral or basic solutions and therefore, EOF is induced in a channel by applying an electric potential between two reservoirs at either end.

We characterized the flow of liquids in microfluidic devices by adding 1  $\mu$ m diameter fluorescent beads to the liquids being studied and using fluorescence microscopy to monitor the motion of these beads in the flow.

# A Fabricate master by rapid prototyping



**Figure 2.** Scheme of the rapid prototyping of microfluidic devices in PDMS. (A) A high-resolution transparency containing the design of the device, created using CAD, is used as the mask in photolithography to produce a positive relief of photoresist on a silicon wafer. (B) Glass posts are placed on the wafer to define reservoirs for liquid. (C) A prepolymer of PDMS is then cast onto the silicon wafer and cured at 65 °C for 1 h. (D) The polymer replica of the master containing a negative relief of channels is peeled away from the silicon wafer, and the glass posts are removed. (E) The PDMS replica and a glass flat are treated in an oxygen plasma discharge for 1 min. When the two oxidized surfaces are brought into conformal contact, an irreversible seal forms between them: this seal defines enclosed microscopic channels.

#### 3.3. Flow deflection switch

Figure 1(A) shows diagrams of the fluidic switch; this design was based on that of a fluidic amplifier [18]. Figure 3(A)

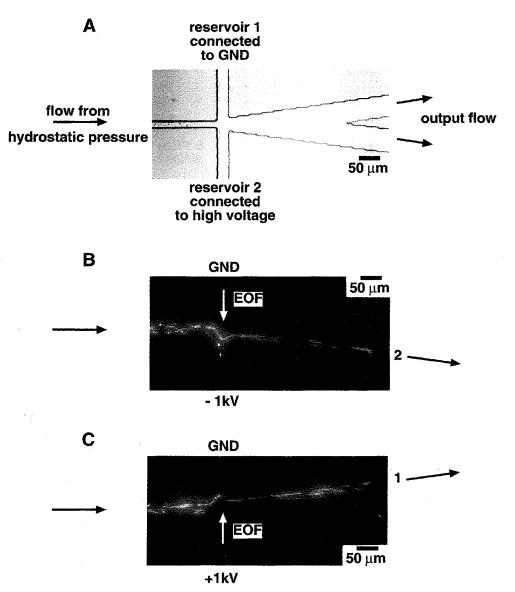


Figure 3. Actuation of the fluidic deflection switch by EOF. (A) Optical micrograph of the switch fabricated in PDMS; the lighter areas are microscopic channels and the darker area is PDMS sealed against glass. An aqueous solution of 25 mM Tris-192 mm Gly containing 1  $\mu$ m diameter beads labelled with a fluorescein fluorophore was pumped into the switch by a hydrostatic pressure difference between the input and output reservoirs. The reservoir of control channel 1 was connected to ground; the reservoir of control channel 2 was connected to a high-voltage power supply. (B) Fluorescence image from the beads when illuminated by light from a Hg lamp, when a negative potential (-1 kV) was applied to control reservoir 2. This potential generated an EOF toward the cathode and caused the flow of beads to be deflected into output channel 2. The beads appeared streaked as they flowed because the image was grabbed from a video signal with a refresh rate of 60 Hz. (C) Fluorescence image from the beads when the polarity of the applied voltage was reversed, i.e. to +1 kV. The change in polarity reversed the direction of EOF in the control channels; the stream of fluorescent beads was directed into output channel 1.

shows an optical micrograph of the device fabricated in PDMS. In our device, the flow of a liquid (water) from an input source was directed into one of two output channels by the application of pressure at two control ports (figure 1(A)). At the low speeds ( $\sim 1~\rm mm~s^{-1}$ ) and viscosity (0.001 kg m<sup>-1</sup> s<sup>-1</sup>) of the liquid in these experiments, the Reynolds number of flow in this device was low (Re  $\approx 0.05$ ). The value of Re is well below the transition Reynolds number (Re  $\sim 2300$ ), above which turbulent flow occurs, so the flow was laminar. For the velocities of liquid we could generate, the device could therefore only be operated in jet deflection mode; a wall attachment device was not a practical option.

The principle of operation of the switch is the same as the amplifier. The input flow can go into one of two output channels. The output channel into which the fluid flows was determined by the pressures applied at the two control ports (figure 1(A)). If the pressure at each control port was equal then the main flow was not deflected and partitioned into both channels. By applying a greater pressure at one of the control ports, the flow was directed into one of the output channels. We actuated the fluidic deflection switch both by EOF and hydrostatic pressure.

Figures 3(B) and (C) show the effect on an input flow of EOF in the control channels of the switch. A flow of an aqueous buffer (25 mM Tris-192 mM Gly) containing fluorescent beads from the input channel was created by hydrostatic pressure, i.e. the level of liquid was higher in the input reservoir than in the output reservoirs. The velocity of the fluorescent beads in the flow was about 1 mm s<sup>-1</sup>. The control channels and their reservoirs also contained buffer, and were balanced

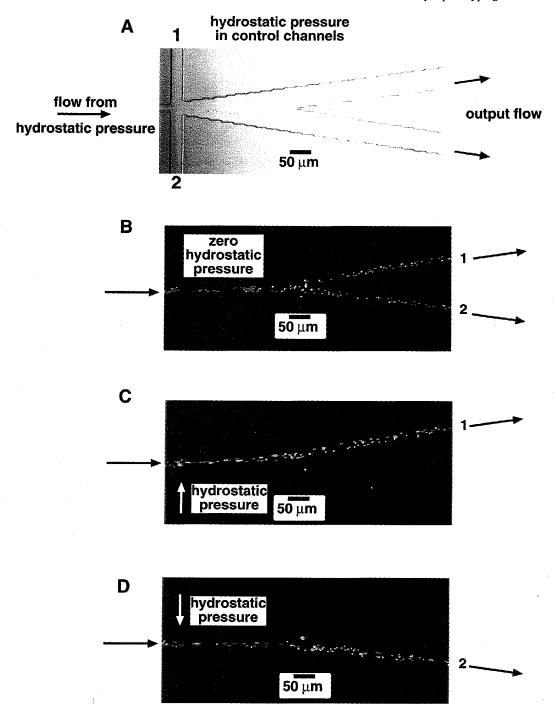


Figure 4. Actuation of the fluid deflection switch by hydrostatic pressure. (A) Optical micrograph of the fluid deflection switch. The same solution as in figure 3 was pumped by hydrostatic pressure into the switch. (B) Fluorescence image of the flow of beads when the levels of liquid in the reservoirs of the two control channels were the same. There was no net hydrostatic pressure in the control channels, the input flow of liquid was not deflected, and the flow partitioned into the two output channels. (C) About 50  $\mu$ l of liquid was added to the reservoir of the control reservoir 2 to produce a net hydrostatic pressure in the control channels (shown by a white arrow). The fluorescence image shows that the flow of liquid that resulted from this pressure was deflected into output channel 1. (D) Fluorescence image of the flow of beads when the direction of the hydrostatic flow in the control channels was reversed (shown by white arrow) by removing about 100  $\mu$ l of liquid from reservoir 2 and adding it to the reservoir of control channel 1. The pressure difference in the two control channels that resulted from a difference in height of the liquid in the reservoirs was about 20 Pa.

hydrostatically. An electric potential (1 kV) was applied across the two control channels; this potential set up EOF. Depending on the direction of EOF, the flow of beads was directed into one of the two output channels. In figure 3(B), the potential of the reservoir connected to control channel 2 was set to -1 kV, with the control channel 1 connected to ground. The surface of oxidized PDMS is negative so EOF was to-

ward the cathode; this flow diverted the main flow into output channel 2. Simply by reversing the polarity of the applied voltage, the direction of EOF was reversed, i.e. it was toward control channel 2, and the main flow was directed into output channel 1 (figure 3(C)). At high (>2 kV) potentials, the EOF in the side channels was strong enough to divert the flow of beads into the control channel at the negative potential.

There is a possible alternative explanation of switching; the direction of the flow of beads could be determined by EOF in the main channel itself. We do not believe, however, that this explanation of switching is correct as the reservoirs connected to the input and output channels were floating electrically so there was little or no EOF in these channels.

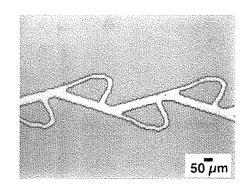
We also actuated the switch by hydrostatic pressure; this experiment is shown in figure 4. When the reservoirs of the control channels contained the same height of liquid, there was no net flow in the control channels, the input flow was not diverted and partitioned equally into the two output channels (figure 4(B)). A difference in the height of the liquids produced a net flow in the two control channels, and caused the input flow to be directed into one or other of the output channels (figures 4(C) and (D)). We actuated the switch hydrostatically simply by adding about  $50 \,\mu$ l to one of the reservoirs of the control channels. This volume resulted in a height difference of about 2 mm of liquid, i.e. a pressure difference of 20 Pa between the two control reservoirs.

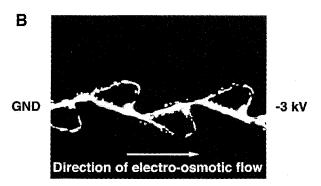
The microfluidic switch presented here could operate as a control element in  $\mu$ TAS to direct analytes in a network of microscopic fluidic channels. The use of this switch would have two main advantages. First, the use of electric fields and electro-osmotic pumping are compatible with the methods that are used currently in  $\mu$ TAS to transport and analyse samples. Second, the switch contains no moving parts: this property reduces the chance of malfunction and clogging due to stiction of moving parts. The fact that this non-mechanical switch functioned with beads shows that it will also tolerate some level of suspended solids: a serious limitation of switches that use micromachined silicon valves with moving parts is that they are easily clogged by particulate matter.

There is also a disadvantage to using this type of switch in  $\mu$ TAS; the method of actuation is 'invasive', i.e. the flow of liquid in the control channels will leak into the main flow. The liquid in the control channels has to be compatible with the solution under analysis. There may also be leakage from the main flow into the control channels. This switch works best in a continuous flow microfluidic circuit, but could also, in principle, be implemented in systems in which the flow is not continuous, despite the possibility of backflow. Backflow could be reduced by selectively changing the surface energy of the exit of the switch, for example, by applying a hydrophobic coating.

# 3.4. Side channel fluidic controller

We fabricated a network of channels (figure 1(B)) based on the design for a fluidic rectifier [19]. Figure 5(A) shows an optical micrograph of the device we fabricated in PDMS. In order to generate sufficient flows in this device for it to operate, we pumped a solution of buffer using EOF; we could not generate high enough flows by hydrostatic pressure using the few mm of hydrostatic head that was convenient to use with this system without applying external pressure. Figures 5(B) and (C) shows fluorescence images of beads flowing in this channel under electro-osmosis in two opposite directions; it is clear that the flow pattern of the fluorescent beads in this channel depended on the direction of EOF. In one direction





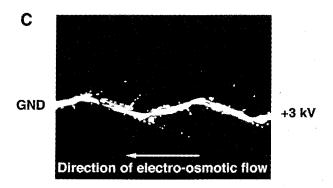


Figure 5. (A) Optical micrograph of a side channel fluidic controller. The light areas are channels; the dark areas are PDMS. (B) Fluorescence image of the same solution of fluorescent beads used in figures 3 and 4 when an electric potential was applied that gave rise to EOF from left to right. The fluorescence indicates that the momenta of the beads carried them into the side channels. (C) Fluorescence image of the beads when the polarity of the applied voltage was reversed. EOF was from right to left; the fluorescent beads emptied the side channels and the momenta of the beads in the central channel did not take then into the side channels.

(figure 5(B)), the momenta of the beads naturally took them into the side channels. When the direction of EOF was reversed (by reversing the polarity of the applied potential), those beads already in the side channels flowed out, and the momenta of the beads in the main channel prevented them from entering the side channels.

Figure 5 shows that the flow of the beads can be controlled by the geometry of the side channels. We choose, therefore, to call this device a side channel fluidic controller. We did not observe rectification of the flow in the main channel using this device, presumably because flow was

generated by strong EOF in the main channel that could not be reduced by the weaker flows in the side channels. Rectification in the side channels was observed; however, the beads flowed in the side channels in one direction but not in the other. This device can therefore also be considered as a side channel rectifier.

#### 4. Conclusions

Rapid prototyping provides a straightforward, inexpensive and rapid way to realize designs for microfluidic devices. Designs are created directly by the researcher in a computer drawing package and transferred to an inexpensive transparency that is used as the mask in photolithography. Moulding a polymer against the resulting master yields a replica that contains networks of bas-relief channels that are sealed simply by oxidation and contact with another oxidized polymeric or ceramic surface. The whole process takes about 24 h and therefore reduces significantly the turnaround time for evaluating ideas for microfluidic devices. Adopting the approach described here could therefore significantly speedup the exploratory stages of  $\mu$ TAS. Once microfluidic devices have been prototyped in this way, the successful concepts can be transferred to  $\mu$ TAS using materials and methods more suited to mass production and incorporation into integrated systems.

In addition to rapid prototyping, the use of PDMS has several other benefits for microfluidic systems. First, oxidized PDMS supports EOF; this flow can provide fluidic pressure to actuate microfluidic devices. This property opens up the possibility of incorporating microfluidic devices, such as the switch, into  $\mu$ TAS. Second, oxidized PDMS is hydrophilic, and microscopic channels can be filled easily with water. The formation of bubbles is a serious limitation for microfluidic systems fabricated from hydrophobic polymers, for which water has a tendency to dewet from the walls of the channels. Third, PDMS is optically transparent and therefore lends itself to several detection schemes (e.g. optical and fluorescence microscopy) that are used commonly in  $\mu$ TAS. Fourth, PDMS is not brittle, and these microfluidic systems are robust physically.

## **Acknowledgments**

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