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Abstract

Valves for microfluidic systems have, for various reasons, proven to be difficult to fabricate, cumbersome to operate, and/or unreliable. We have explored the performance of a novel microfluidic valve formed by creating a flow channel past a Peltier junction. When the Peltier junction is used as a thermoelectric cooler it is possible to freeze the fluid in the valve, forming an ice plug that blocks flow through the valve. This type of valve is fundamentally leak-free, has no moving parts, and is electrically actuated. We have fabricated several experimental prototypes and evaluated their performance. We find that they are reliably capable of closing in less than 100 ms, and of opening substantially faster.

Introduction

A potential new application for thermoelectric devices is in the fabrication of microfluidic valves. [1] Microfluidic technology is focused on producing fluidic analogs of the integrated microelectronic circuit. The ultimate goal is to reproduce an entire biological or chemical laboratory process on a microchip. Fluid handling systems on the macroscopic scale incorporate pipes, valves, pumps, and fluid storage reservoirs, as well as specialized components such as heat exchangers and filters. Microfluidic systems should, ideally, be able to incorporate all these functions into an integrated device of an appropriate size. As can be expected, pipes are relatively easy to manufacture, even with micron, or submicron dimensions. Various technologies have been developed for pumping on microscopic scales, including centrifugal systems (where the entire device is rotated at high speeds) and electro-kinetic systems.

In contrast to pipes and pumps, valves have proven to be particularly difficult on the microscopic scale. Reliable fluid valves have been in use in macroscopic systems for centuries. These valves depend on various types of mechanical actuators that move an obstruction across an opening to stop the flow. In principle, one might expect simply to be able to shrink such valves to microscopic dimensions. This fails, however, for two main reasons. The first is that traditional valves have complex geometries that are difficult and expensive to reproduce on microscopic scales. The second reason is that, as sizes decrease, it becomes progressively more difficult for mechanical actuators to provide sufficient force to seal the valve closed. At the same time, as the size decreases, pinhole leaks due to mechanical imperfections in the sealing surfaces take on a proportionally greater significance; a leak rate of 100 microliters per day will not be noticed in a kitchen faucet, while a similar leak rate in a microfluidic device could result in a complete loss of the sample within a few minutes. For these reasons, an entirely new concept is needed for microfluidic valves.

One concept that appears to have some potential is the Peltier-actuated microvalve, in which valving action is accomplished by using the Peltier effect to freeze the working fluid in a segment of the pipe. The resulting ice plug blocks the flow. When the Peltier cooler is turned off (or reversed), the ice plug melts and flow resumes. This valve actually works better as it gets smaller because the time required to freeze enough fluid to block the flow channel decreases. Compared to conventional valves, the Peltier-actuated microvalve has several notable advantages. Among these are that the valve has no moving parts, is electrically actuated, is leak free, is unaffected by particulate contamination, and has the characteristics of a straight pipe when the valve is open.

The Peltier-actuated microvalve is particularly suited to biomedical applications because most biological fluids are water-based and can easily be frozen using the Peltier effect. When combined with suitable technology for flow channels and pumps, the Peltier-actuated microvalve becomes an enabling technology for a versatile biomedical "lab-on-achip." An important requirement for biomedical applications is that cross-contamination between samples be avoided. This is best accomplished by disposing of the hardware after each use. It is possible to build separable versions of the Peltieractuated microvalve, such that the thermoelectric elements are on a reusable module and the fluid channels are in a disposable module. [2]

In some applications, it is desirable to reduce the valve actuation time to the minimum possible. This would be important, for example, when the valves are used cyclically to control the inlet and out of a displacement pump. Our recent work has been focused on developing designs for fast Peltieractuated microvalves. Previously we reported preliminary results from a numerical thermal model of the valve. [3] In this paper we report on the design and performance of valves optimized using the results of the thermal modeling. The performance of the valves is also compared with the modeling results.

Valve Design

The key to making the valves fast is to use a single linear junction, keeping it as short as possible, and having a small flow channel in good thermal contact with the junction. It is also essential to provide good heat sinking to keep the valve from overheating.

Our test valves are fabricated from single linear junctions of bismuth telluride, with each of the n and p legs having a length of 300 microns. The legs are sandwiched between 1mm blocks of sliver and copper, which provide both electrical contacts and heat sinking for the hot-side junctions. In addition, the entire junction structure is mounted on a 6-mmthick aluminum substrate that provides both structural support and additional heat sinking. The conduction layer is separated from the heat sink layer by a thin electrically insulating layer to prevent shorting of the TE junction. The flow channel is formed in a polymeric substrate attached to the surface of the junction. A schematic diagram of a single valve is shown in figure 1.



Figure 1: Schematic diagram of Peltier-actuated microvalve.

Test valves are fabricated with n-type and p-type bismuth telluride elements that are 300 microns thick, providing a total junction length of 600 microns. The junction is sandwiched between 1-mm-thick silver and copper blocks. The silver and copper can serve equally well for both electrical and thermal conductivity. The reason for using one of each is to make it possible to visually determine the polarity of the junction. The junction is mounted between two properly spaced holes in a printed circuit board. The heat-sink substrate is coated with thin layer of an electrical insulator (paint, or spin coat have both worked successfully). The PC board with the junction is then bonded to the heat-sink substrate using epoxy. The bonding orientation is such that the conduction traces on the PC card, as well as the silver and copper blocks in the linear junction, are adjacent to the heat-sink substrate. After curing, the PC card and the linear TE junction are ground down to a thickness of 300 microns. This produces a junction, such as that illustrated in figure 1, in which the legs are each 300 microns long, 1 mm wide, and 300 microns deep. A photograph of a single linear junction is shown in figure 2.



Figure 2: Photograph of single linear junction without flow channels.

A one-dimensional heat transfer analysis for a single linear thermoelectric junction provides a closed form solution for the largest steady state temperature difference that can be maintained:

$$\Delta T_{\max} = -\frac{\Pi^2}{8\rho k}$$
 [1]

where Π is the Peltier coefficient, ρ is the resistivity, and k is the thermal conductivity. This shows that the steady state temperature of the junction depends only on the material properties and is independent of geometry. The current density at which the maximum temperature difference occurs is given by:

$$I_{\Delta T \max} = \frac{\Pi}{\rho L}$$
 [2]

where *L* is the length of the thermoelectric element.

Both equations 1 and 2 refer to steady-state conditions. Valve cycling is, of course, a transient event, and the rate of cooling depends in a complex manner on the current density. This is best studied with numerical models. [2, 3] The general principles are illustrated in figure 3, which shows the temperature as a function of time for a linear junction at various current densities. The Peltier junction in this case 325 microns thick and has an overall length of 1 mm (500 microns per segment). The width is undefined in this two-dimensional model. At very low current densities, the junction never cools to the point of freezing. As the current density increases, the minimum temperature decreases, up to a point. As the current is increased beyond the optimum current density, the valve will continue to cool more quickly, but ohmic heating begins to dominate, and the valve eventually overheats. At very high current densities, the ohmic heating quickly overwhelms the Peltier cooling, and the valve never freezes.

Experimental Results

Because of the small size of the valve, it is not practical to directly measure valve temperature. In addition, because of the homogeneity of the ice that forms, it is generally difficult to see whether any ice is present on the valve junction. We have found two practical diagnostics of valve function. The first is to measure the transient electrical properties of the junction in response to a step change in current. The second is to use high-speed digital video microscopy to observe flow through the valve.



Figure 3: Valve temperature as a function of time at various current densities.

At any given current, the voltage drop across the valve is determined by the resistivity of the bismuth telluride and the thermoelectric voltage due to the temperature difference between the hot and cold junctions. When power is first applied to the valve, there is no temperature difference between the hot and cold sides, so there will be no thermoelectric voltage, and the voltage drop across the valve will be only resistive. As the temperature difference increases, the total voltage drop across the valve will increase. When the device reaches thermal equilibrium, the voltage drop as a function of time gives an indication of the time constant of the valve.

In testing the valve, we used an externally-programmable current-controlled power supply (Kepko BOP 20-10). This supply has a delta-function rise time less than one ms for transitions from no current to 5 A. A National Instruments AT-MIO-16XE-10 A-to-D and D-to-A board operating under LabView was used to control the power supply and to measure the voltage drop across the valve.

Figure 4 shows a plot of voltage drop as a function of time for a typical valve when the current is switched to 3 A. (the rise time on the current was less than 1 ms.) The voltage drop is initially between 84 and 85 mV, but rises rapidly, reaching 89 mV within 150 ms. The equilibrium value is just under 90 mV. It is not clear from this data, however, at what point the valve freezes the fluid.

Actual valve operation can be imaged using high-speed video. Figure 5 shows two frames taken from a typical video. In this figure, a flow channel has been attached to a junction

such as that seen in figure 2. The flow channel in this case has a width of about 500 microns, and a depth of about 50 microns, and is covered with acrylic to allow visualization of the flow. The channel is filled with water and the water is seeded with 7-micron-diameter red beads to enhance visibility of the flow. Flow is from right to left.



Figure 4: Voltage drop across a single valve as a function of time in response to a 3 A current. Data from two separate runs with the same valve are shown, and almost completely overlap.



Figure 5: Two frames from a video of the valve in operation. In the top image, the fluid is flowing through the valve. In the lower image, an ice plug at the valve junction blocks the flow.

The difference between the two frames in figure 5 is that water is flowing in the first frame while, in the second frame, the flow is blocked by an ice plug at the junction. The ice plug itself is difficult to see in a still image because it is free of cracks or other imperfections such as those that typically make ice cubes visible in a glass of water. The most observable distinction between the two images is the slight motion-induced blurring of the particles in the upper frame. Watching the video, however, it is very easy to see whether the fluid is moving. It is also possible to observe the growth of the ice plug due to the change of the index of refraction associated with the phase change.

The video system used to obtain the images of figure 5 is capable of imaging at frame rates in excess of 1000 frames per second. In practice, such high frame rates are both difficult (because of the lighting requirements for the short exposure times) and unnecessary; we find that the current version of the valve typically operates on time scale of 10s of milliseconds, so frame rates of 250 Hz are sufficient to explore the transient behavior of the valve.

As noted above, theory indicates that the junctions used in our valves should have a maximum temperature difference at a current of about 3 A. To date, we have no way of directly measuring the cold-side junction temperature. We are, however, able to note the range of current over which ice can be formed at the junction, causing the valve to close. Although it varies from one valve to another, probably due to slight variations in geometry, the typical current range over which these valves can operate is 2.5 to 5 A.

For the video experiments, we used the same power supply and National Instruments A-to-D and D-to-A board described above. An additional digital output channel was used to trigger the camera, providing timing information. Thus, the computer would trigger the camera at the same time that it would switch the current in the junction from zero to the desired value. After a predetermined period (typically 200 ms), the current would be switched back to zero. By noting the frame numbers in the video where the flow stopped and then resumed, we could determine the cycle time of the valve. The results of several tests are summarized in figure 6.



Figure 6. Valve cycle time data at various current levels.

Although the data are widely scattered, two trends are visible (if only weakly). Numerical modeling indicated that the time required to close the valve should decrease with increasing current density, and this is seen, at least at the lower current levels. At higher current levels, the timing trend appears to level off. This could be due to any of a number of factors, and is still being investigated. The overall timing agrees fairly well with the numerical model of this valve, which indicates an expected closing time of 55 ms. More interesting is that the time required to open the valve appears to reach a maximum when the valve is operated at optimum current. This is most likely due to the fact that the size of the ice plug blocking the flow also is maximum at this current. Since the opening process involves heat leaking into the valve from the surroundings, one would expect that a larger ice plug would take longer to melt. At very high current levels, the ice plug is relatively small. At the same time the temperature of the rest of the valve is probably slightly elevated due to both ohmic heating and heat dissipation at the hot side of the junction, so the heat loading on the ice plug when the valve is shut off would be larger, leading to rapid melting.

Conclusions

We have developed a design for a relatively fast-acting Peltier-actuated microvalve, along with reliable methods for manufacture. The operating time of the valve agrees fairly well with expectations, both analytical and numerical. The cycle time, at less than 100 ms, is fast enough to be of interest in a number of microfluidic applications.

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References

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