Micro thermoindicators and optical-electronic temperature control for microfluidic applications

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The authors report the design and implementation of sensing and control of local temperature in polydimethylsiloxane microfluidic reaction chip, based on the fabrication of a microtemperature sensor with thermochromic color bars and the associated optical and electronic feedback controls. The thermochromic color bar demonstrates easy and accurate local temperature monitoring. In combination with a microheater, this contactless microchip temperature control approach may have wide application potentials in microchemical and microbiological analyses. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2776848]

Temperature is a basic environmental parameter which can affect many material properties. Various types of temperature sensors are available, such as fiber-optic sensors for high-temperature measurements,[1] sensors of organic thin film transistors,[2] etc. Recent interest on microfluidic chips for chemical and biological functions[3] has focused attention on temperature control in these systems, as thermal detection and control are important in microreactions and bioprocesses, e.g., experiments regarding DNA sequencing and cell biology applications.[4] Platinum thin film has been commonly used as a temperature sensor in microchips.[5] It has been reported that thermal microscopic scan, using fluorescent particles as sensor, has also been employed.[6] In another approach, infrared cameras are frequently utilized to not only obtain surface temperature distributions via images[7] but also constitute a feedback system for temperature control.[8] Low cost infrared sensors have been developed for these purposes.[9]

For its ease of fabrication, biocompatibility, and other merits, polydimethylsiloxane (PDMS) is considered as a primary base material for microchip fabrications.[10] However, owing to its weak bonding characteristic with metallic materials, it is difficult to implement microtemperature sensors inside PDMS chips during the soft lithographic fabrication process. In addition, since the material would shield signals from IR cameras, contactless sensing of local temperature inside the microchips is difficult. To solve the problems mentioned above, we present a design and fabrication of thermochromic microcolor bars, which provides a local temperature indicator inside the microfluidic chip which can be sensed optically. Together with the embedded PDMS/silver particle-based microheater and optical sensor,[12] the local thermal characteristics of microfluidic chips can be easily monitored and controlled through a feedback electronic system.

To show the functionality of our approach, we have designed a microfluidic chip for a well-known chemical reaction experiment, as shown in Fig. 1. The upper right inset is the top view of an image of the microfluidic chip, which is 32 mm in length and 10 mm in width. The color bars located at the lower layer consists of six different bars, each fabricated with a specific mixture of thermochromic particles (3–7 μm in diameter, Lijinkeji Co., Ltd.) and pure PDMS.[11] The color transition temperature for each of the six bars is arranged in sequence and ranges from 30 to 60 °C. For example, when the temperature exceeds a certain value, corresponding color bar(s) will transform from its original color to white. Every bar is associated with a circle (for optical sensing, see below), an arrow, and a digital number indicating its color transition temperature, as shown in Fig. 1. The contrast changes of color bars are very sensitive to the temperature which is calibrated by a hot stage which is preciously controlled by a thermocouple temperature control system. A microheater (synthesized with silver-PDMS composite) with an initial resistance of 69 Ω is also embedded in this layer to generate heat in the prespecified area. Detailed fabrication process for the microheater can be found in our previous work.[12] Microfluidic channels 200 μm in width and 100 μm in depth are located at the upper microchip layer. These channels have three functional sections: a heating section, a

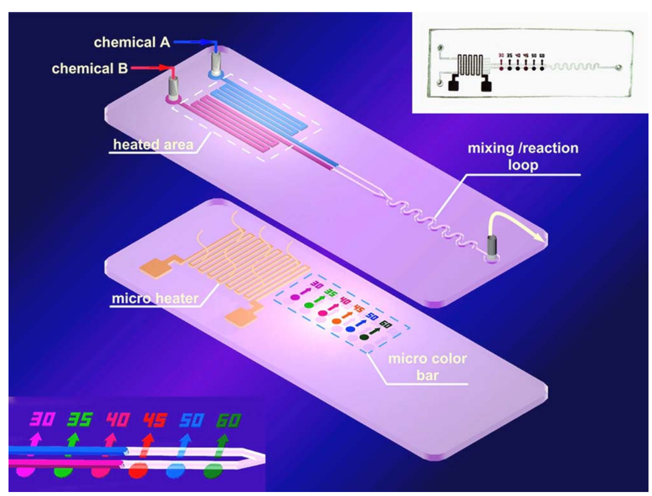

FIG. 1. (Color online) Schematic illustrations of the three-dimensional layered structure of the PDMS microreaction chip. The thermochromic color bars and microheater are located on the lower layer, while the microfluidic channels for chemical reactions are on the upper layer. The lower left inset shows an enlarged view of the thermochromic color bars and the upper right inset shows an image of the fabricated device.

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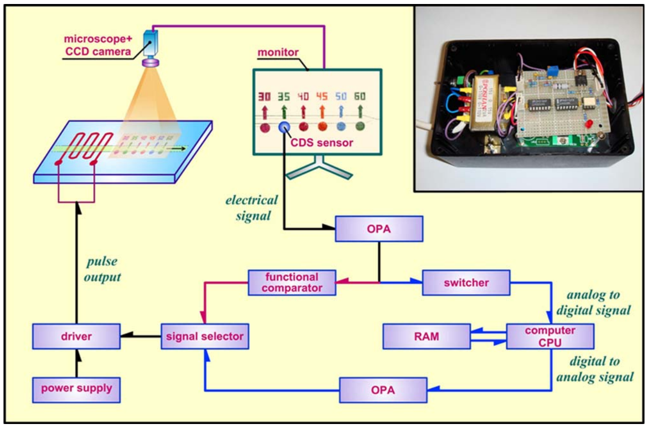

FIG. 2. (Color online) Diagrams of the optical-electrical temperature sensing and control processes. Combined with computer storage of calibrated control signals, this process can achieve accurate local temperature control in microfluidic devices. The process is operated via a control box shown in the upper right panel.

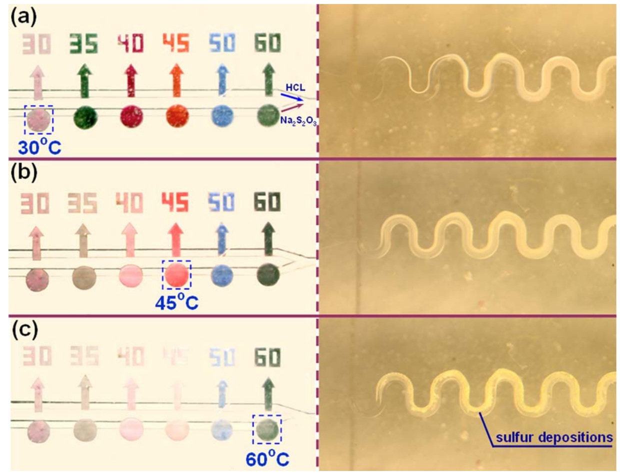

FIG. 3. (Color online) A demonstration of temperature controls in a microreaction involving sodium thiosulfate and hydrochloric acid. Left panels show the set target temperatures on the thermochromic color bars. Corresponding reactions are shown to the right. Here, the reaction product, sulfur, is what makes the loops clearly visible.

temperature detection section, and reaction loops. The heating section has two symmetric zigzag channels for heating the chemical solutions when two different chemicals (A in blue and B in red) are injected into the chip. When the two heated fluids flow through the temperature detection section, the solution temperature will cause the color bars (which are in contact with the microfluidic channels) to change color (lower left inset in Fig. 1), and in the process its temperature becomes apparent. After flowing through the temperature detection section, the two chemical solutions are mixed in the reaction loop, leading to a chemical reaction at the desired temperature.

In order to precisely control the local temperature inside the microfluidic chip, a temperature detection and feedback control system for the microheater is designed and constructed, shown as a flowchart in Fig. 2. A microscope connected with a charge coupled device (CCD) camera is positioned upright over the chip to monitor the color bar area. When color bars vary their contrast at different temperatures, their color images are detected by the CCD camera and displayed on a monitor. A photoconductive cell (CdS) (NORP-12, Silonex Inc.) would convert the detected image contrast (calibrated by a standard temperature control system mentioned above) into a digital electronic signal, input to the feedback system, as shown in Fig. 2. Thus, if the microfluidic temperature is set to be 35 °C, the CdS sensor will be deployed to focus on the circle area of the relevant color bar, denoting the CdS sensor induction zone. CdS sensor is sensitive to image contrast; e.g., when the induction zone is bright, CdS conductivity will have a high value; when the zone is dim, the conductivity will be reduced. Hence, the CdS sensor will detect color brightness from the induction zone in order to determine the on/off status of the microheater. This is achieved through an operational amplifier which amplifies the signal from the CdS sensor and passes it to a functional comparator (route in red color, Fig. 2). The functional comparator will determine the output status of the power supply. If a signal representing dim color in the induction zone is received (temperature is lower than that of the set temperature), the comparator will generate a trigger signal to turn on the microheater power supply so as to increase the temperature. When the temperature of the induction zone reaches the set temperature value, the corresponding color bar would change to white and the comparator will cut off the voltage output from the driver. In this manner, the feedback system adjusts the microfluid temperature.

In case the desired set temperature should be maintained for a long period of time, the analog control signal can be converted to digital form and stored in random access memory. The signal selector is then disconnected from the feedback loop and instead receive the control signal from the CPU after a reverse digital to analog conversion. In this way, the optical-electronic feedback control loop would serve only for the initial calibration purpose, with the subsequent temperature control independent from the microscope and the CCD camera.

A chemical reaction experiment was carried out to test the functionality of the thermochromic color bar and the associated temperature control aspects for our system. Liquid solutions of sodium thiosulfate and hydrochloric acid in concentrations of 3 and 6 mol/L, respectively, were injected into the microchannels at the velocity of 0.02 ml/m with a syringe pump. When the two chemical solutions were mixed, reaction occurred and sulfur (yellow in color) became visible. Hence, on the right panels of Fig. 3, the invisible sections of the reaction loop indicate not-yet-reacted chemical solutions, whereas the clearly visible sections indicate the presence of sulfur. The intensity of the reaction was observed to increase with the reagents' temperature, with more sulfur becoming visible in the loop channel. When the CdS sensor was set on the 30 °C color bar, the reaction was barely proceeding and sulfur particles were formed only in the last two loops, as shown in the right panel of Fig. 3(a). However, when the temperature was set at 45 °C, the reaction accelerated, with sulfur becoming visible after the first loop. A similar situation was observed when the temperature was set at 60 °C, whereby the reaction proceeded very quickly and large particles of sulfur were visible almost right after mixing. Images on the left panels show that as long as the appointed color bar reached the set temperature, even a very slight contrast change can be immediately detected by the sensor and a corresponding output signal to the control system was generated to accurately maintain the heater's status. The different reaction results validated our control system's

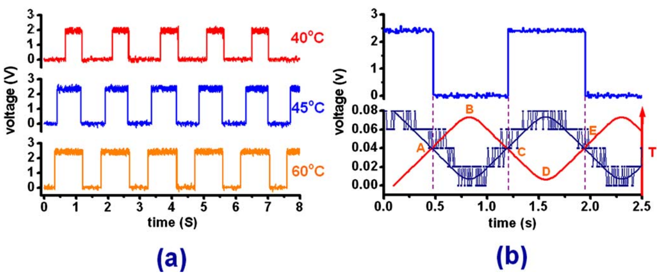

FIG. 4. (Color online) (a) Square-wave trigger pulse signals generated by the system to control the microheater. (b) CdS output voltage (fitted by the blue line on the lower panel) is juxtaposed with the predicted temperature variation (red line). The corresponding trigger voltage pulse train is shown on the upper panel. There is a systematic delay time of ~0.7 s.

capability in adjusting the temperature in microreactions within the desired range.

In order to quantitatively validate the temperature control, an oscilloscope was used to record synchronous signals to the microheater and the voltage output from the CdS sensor. Figure 4(a) shows trains of square waves for driving the microheater at the set temperatures of 40, 45, and 60 °C. It can be seen that at fixed pulse amplitude, a higher set temperature of the microheater requires longer pulse duration, with slightly increased duty cycle as well. In Fig. 4(b), the CdS voltage output for 45 °C set temperature (lower panel), fitted by a dark blue line, is compared with the corresponding triggered pulse (upper panel). As the temperature of the color bar rises and the contrast becomes lighter, the resistance of the sensor decreases, thus bringing down the voltage output. Hence, by reversing the voltage output of the CdS sensor in the blue line, the temperature variation tendency is obtained, indicated by the red line. It can be seen that once the desired temperature of 45 °C at point A is reached, the trigger pulse (upper panel) is turned off, but the temperature is seen to keep rising to peak B before decreasing to 45 °C again at point C. When the trigger pulse to the heater is turned on at the next pulse, there is a delay for the heater to heat up the fluid; hence, the temperature decreases to point D before rising up again to point E. It can be seen that voltage from the CdS sensor is in very small values and the response time is measured to be ~0.7 s. Hence, stable temperature can be maintained with only small fluctuations owing to the response time of the system.

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