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Development of a Novel 2-Dimensional Micro-Heater Array Device with Regional Selective Heating

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Abstract

This paper reports the development of a micro-heater array device that can be selectively heated at an arbitrary location. To confirm the heating characteristics of this micro-heater array device, heating experiments using a thermo-responsive gel were conducted. Since a micro-heater can supply heat with a rapid-response on a micro-scale, various applications have been studied. Based on these characteristics, micro-heaters have often been used in recent research applications involving biological cells. To expand the versatility of the micro-heater, the development of micro-heater array systems that can supply heat selectively at an arbitrary location is required. In this work, to support micro-heater applications in the field of biochemistry, the design and materials of a micro-heater array device were optimized and a fabrication process was established. Furthermore, the usefulness of this device was verified using a thermo-responsive gel, and control of the temperature distribution on a glass substrate was successfully demonstrated. This micro-heater array device can be heated with regional selectivity, and each region can be controlled to an arbitrary temperature, so the device is also capable of generating temperature gradients.

Keywords: micro heater, heater array, temperature control, temperature gradients

1. Introduction

A micro-heater can reliably supply heat and has a rapid response. Because micro-heaters can be fabricated from a variety of materials, they can also offer high reliability, long operational lifetimes, inexpensive fabrication, and high shape flexibility, such as a coil shape. Based on these characteristics and their high response at the micro-scale, micro-heater arrays for a number of applications have been investigated. Moreover, there have been numerous reports of the fabrication of micro-heater systems using micro electromechanical systems (MEMS) technology.

On a macro-scale, nichrome (NiCr) and tungsten (W) both have a high electrical resistance, and are commonly used in heater elements. Since nichrome has a high ductility and good corrosion and acid resistance, it can be sealed in an extra-fine tube for use at the micro-scale. Moreover, since these materials can be fabricated using MEMS technology, their integration on a chip is possible (Sima et al., 2013; Das & Akhta., 2013; Mamanee et al., 2008; Lyulevich & Yaminskii, 2000; Weir et al., 2009; Shukla et al., 2009; Haque et al., 2008; De Luca et al., 2015). As the use of MEMS can enable the construction of integrated devices, the heater element can be integrated with other functionality. For example, in order to control the temperature with a high accuracy, a heater element and a temperature sensor can be integrated on the same chip. In this case, a heater element and a thermistor (a type of temperature sensor) were fabricated from nickel (Ni) and platinum (Pt), and therefore had a resistance that varied more strongly with temperature than sensors made from other materials (Jiang et al., 2012; Resnik et al., 2011; Tao & Tsai, 2002; Yoon et al., 2002; Yan et al., 2008). There are many other cases where combinations of different material properties are used for a variety of purposes. Aluminum (Al) has a high resistance to organics, so it is being used in micro-heater systems for application in organic environments (Santra et al., 2009). Indium tin oxide (ITO), a type of transparent electrode, is being used as a micro-heater system in combination with a glass substrate for microscopic observation (Arai et al., 2005). In many micro-heater devices, the basic heating principal involves supplying an electrical voltage to a heater element.

Since a micro-heater can supply heat with a rapid response at the micro-scale, various applications have been investigated. In the field of engineering, for example, the development of high-sensitivity sensors has been reported. The developments of inertial sensors such as gyroscopes and acceleration sensors have also been reported. A working principle is based on convective heat transfer and thermistor signals (Dau et al., 2006; Dau et al., 2007). An inertial sensor based on the thermo-resistive effect has many advantages over those based on other methods such as piezo resistance or capacitance effects. For example, the structure of a sensor that uses the thermo-resistive effect is simple, and has high impact and vibration resistance. The development of a flow-sensor was also reported, based on the same principle (Sasakii et al., 2005). Other sensors that can employ micro-heater systems are humidity-sensors, gas-sensors, pressure-sensors and infrared-sensors (Dai et al., 2006; Roy et al., 2012; Zhou et al., 2015; Hwang et al., 2011; Bruyke & Puers, 2000; Zhou et al., 2013). Moreover, micro-heaters have also been used in research involving biological cells in recent years, and are expected to enable control of the expression of cell function and alteration of cellular morphology. Many biochemical experiments have been performed using micro-chips called Bio-MEMS or micrometer-scale total analysis systems (μ-TAS), micro fluidic devices such as microchannels, and chambers made of polydimethylsiloxane (PDMS). Applications that combine this microfluidic chip and a micro-heater system have been reported. Although configurations that combine both systems are simple, their intended applications are diverse, and include molecular and cellular analysis such as polymerase chain reaction (PCR), trapped glucose analysis, enzyme analysis, and DNA analysis (Ha et al., 2008; Paranjape et al., 2003; Arata et al., 2006; Deng et al., 2005; Lee et al., 2008; Cheng et al., 2013).Many applications that do not use a PDMS chip have also been reported. For example, cell manipulation using a thermo-responsive polymer and a drag release system has been reported (Takeuchi et al., 2013; Chau & Melvin, 2012).

To expand the versatility of biochemical applications using micro-heaters, further development of micro-heater array systems is required. Because localized heating and generated temperature gradients can be accomplished using multiple heater elements, more detailed experimental conditions can be established. Specifically, micro-heaters with a two-dimensionally arrayed structure as a Digital Mirror Device (DMD), can be heated with regional selectivity at an arbitrary location, and can be used to establish a process flow at low cost and in a short time. Although there have been many reports of micro-heater fabrication, there have been few reports of the development of micro-heater arrays. The fabrication of micro-heaters with a one-dimensionally arrayed structure has been reported (Han et al., 2005), as has the fabrication of a two-dimensionally arrayed structure consisting of a limited number of micro-heater elements (Liu et al., 2006). However, using previous micro-heater array fabrication methods, it is difficult to extend the process to a large area or a high density. Because most micro-heater devices generate heat when a voltage is applied to the heater elements, lead wires are required to supply this voltage to the heater element. As shown in Figure 1 (a) and (b), for one-dimensional and two-dimensional arrays of only a few micro-heaters, adjacent lead wires do not interfere with each other. On the other hand, when many micro-heaters are placed two-dimensionally at a high density, a structure must be devised to accommodate the lead wires (Figure 1(c)). If the electrodes and lead wires are located on the lower side, such as in a DMD, the fabrication process and structure will be complicated and difficult to achieve on a glass substrate. Unfortunately, particularly in biochemical applications, the use of a glass substrate is desirable. Therefore, in this research, we attempted to develop a micro-heater array device that is applicable to the field of biochemistry.

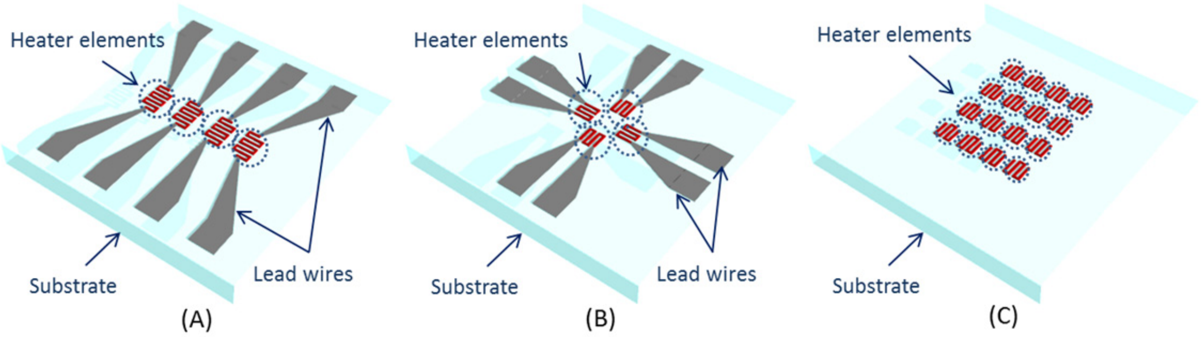

Figure 1. Schematic layouts of arrayed heater elements. Heater elements enclosed in dashed blue circles are placed on the substrate, and lead wires for the application of voltage to the heater elements are connected to both ends of the heater elements. (A) In one-dimensional micro-heaters, adjacent lead wires do not interfere with each other. (B) In two-dimensional micro-heaters with only a few elements, adjacent lead wires do not interfere with each other. (C) When many micro-heaters are placed two-dimensionally at a high density, it is difficult to place the lead wires due to space limitations

2. Experimental

2.1 Design of Micro-Heater Array Device

The design guidelines for this work included the ability to heat selectively at an arbitrary location and applicability to biochemical experiments. A micro-heater array device was designed on the basis of these design guidelines. First, the heater elements were integrated digitally on a glass substrate that is easy to prepare at a low cost and is widely used in biochemistry experiments. Moreover, to heat with regional selectivity, a wiring structure was invented. A micro-heater array device fabrication process suitable for high-precision microfabrication and large-area fabrication was adopted from MEMS technology.

In this micro-heater array device, heating is accomplished by supplying an electrical voltage to a heater element. To simplify the fabrication process and structure, the heater elements and lead wires were fabricated using the same material, but this is only possible in arrays of many heater elements if the line width of the heating elements is narrower than the line width of the lead wires. The heating elements and lead wires were fabricated from chromium (Cr), which is a suitable material for micro-fabrication and is very stable, both electrically and mechanically.

To realize regionally selective heating at an arbitrary location, a lead wire structure was devised. In this work, a structure in which the lead lines are grade crossings with overpasses was invented. Crossover points at which two lead wires cross are insulated using oxidized silicon, which is composed of the same materials as the glass substrate. This wiring structure enables heating with regional selectivity, and this micro-heater array device was fabricated from only Cr and oxidized silicon materials. Moreover, these materials are stable, both electrically and mechanically, many proven in developing MEMS device, and are highly reliable.

Figure 2 shows the design of the micro-heater array device, which includes two-dimensionally arrayed heater elements and a crossing structure that lead lines are not energized. To fabricate this device using MEMS technology, the device was divided into three layers. The first layer contains heater elements and lead wires without crossover points. The second layer places an insulator over the lead wires at the crossover points. Finally, the third layer adds additional lead wires over the insulator to connect the decoupled lead wires in the first layer.

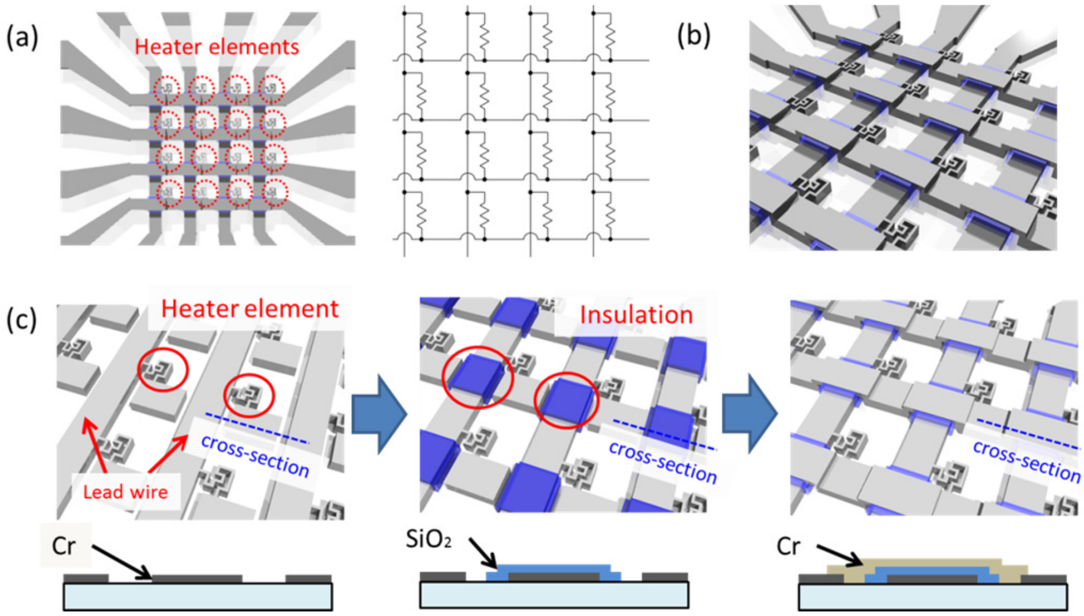

Figure 2. Design of the micro-heater array device. (a) Top view of micro-heater array device, in which heater elements are mounted in a 4-by-4 matrix on the substrate, along with a circuit schematic of the heater elements and lead wires. The top-to-bottom lead wires and the right-to-left lead wires do not interfere with each other. (b) A birds-eye view of the micro-heater array device. To realize the circuit schematic, a structure in which the lead lines are grade crossings with overpasses was designed. (C) Birds-eye and cross-sectional views of the structure. To fabricate this device using MEMS technology, the device was divided into three layers. The first layer contains heater elements and lead wires without crossover points. The second layer places an insulator on the lead wires at the crossover points. Finally, the third layer adds a lead wire over the insulator to connect the decoupled lead wires in the first layer

2.2 Fabrication Process and Conditions

In this work, 80 µm x 80 µm heater elements were placed at intervals of 200 µm in a 4-by-4 matrix on the substrate. The width of the heater elements was 10 µm, the width of the lead wires was 80 µm, and the insulators were 100 µm x 100 µm. The fabrication process flow is shown in Figure 3.

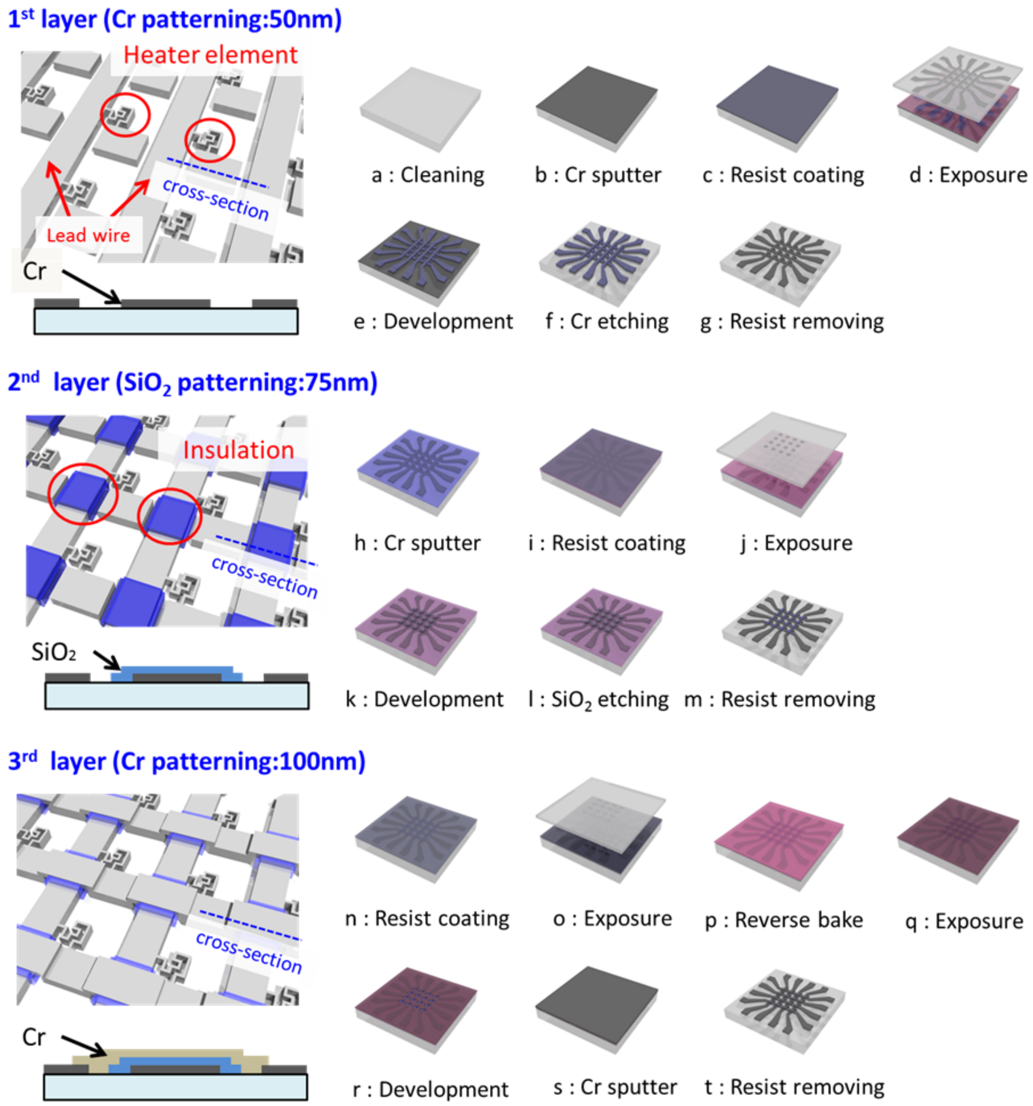

Figure 3. Fabrication process flow. (a-g) Heater elements and lead wires without crossover points are fabricated from Cr. A 50-nm-thick Cr film is sputtered on the glass surface, and heater elements and lead wires are patterned by photo-lithography and Cr-etchant. (h-m) Insulators on the lead wires at the crossover points made are fabricated from oxidized silicon. A 75-nm-thick SiO_2 film is sputtered, and insulators are patterned by photo-lithography and HF. To ensure proper insulation, the SiO_2 film must be thicker than the Cr film. (n-t) Other lead wires made of Cr are fabricated over the insulators to connect the decoupled lead wires in the first layer. 100-nm-thick Cr is patterned using a lift-off method. To ensure electrical connectivity, the Cr film must be thicker than the SiO_2 film

First, heater elements and lead wires without crossover points are fabricated from Cr. After the 1.0-mm-thick glass (Micro Slide Glass, Matsunami) surface is cleaned using ethanol and O_2 plasma-ashing, a 50-nm-thick Cr film is sputtered onto the glass surface using a radio frequency (RF) sputtering system (EB1100 PVD Equipment for R&D/Small-Scale Production; CANON ANELVA CORPORATION), and a 2.0-µm-thick AZ1500 photoresist (AZ1500 38cp; AZ Electronic Materials) is spin coated (4000 rpm, 30 s) onto the substrate (Figure 3 a, b, and c).

After the construct is baked for 3 min at 110°C, the photoresist is exposed to UV light (20 s) using a mask aligner (MA-10; MIKASA CO., LTD.), and then developed using an NMD-3 developer (Figure 3 d and e). After the heater elements and lead wires are patterned by Cr-etchant, the resist is removed using AZ remover and O_2 plasma-ashing (Figure 3 f and g).

Second, oxidized silicon insulators are fabricated over the lead wires at the crossover points. A 75-nm-thick SiO_2 film is sputtered using an RF sputtering system (SVC-700LRF; Sanyu Electron Co., Ltd.), and a 2.0-µm-thick AZ1500 (38cp) photoresist is spin coated (4000 rpm, 30 s) onto the substrate (Figure 3 h and i). After the construct is baked for 3 min at 110°C, the photoresist is exposed to UV light (20 s) using a mask aligner, and then developed using the NMD-3 developer (Figure 3 j and k). After the insulators are patterned using Pure Etch (Pure Etch ZE250; Hayashi Pure Chemical Ind., Ltd.) a type of hydrofluoric acid (HF), the resist is removed using AZ remover and O_2 plasma-ashing (Figure 3 l and m). To ensure proper insulation, the lead wires must be covered by the SiO_2 film. Therefore, the SiO_2 film must be thicker than the Cr film.

Third, additional Cr lead wires are fabricated on the insulators to connect the decoupled lead wires in the first layer. This third layer is patterned by a lift-off method. This is because the use of a Cr etchant would also etch the heater elements and lead wires in the first layer. A 500-nm-thick AZ5206 (AZ5206; AZ Electronic Materials) photoresist is spin coated (5500 rpm for 40 s and 6000 rpm for 2 s) onto the substrate, the construct is baked for 5 min at 90°C, and then the photoresist is exposed to UV light (6 s) using a mask aligner (Figure 3 n and o). To fabricate a reverse-taper structure suitable for lift-off, the construct is baked for 5 min at 120°C and the photoresist is exposed to UV light (6 s) using a mask aligner (Figure 3 p and q). After the photoresist is developed using the NMD-3 developer and the surface is cleaned by O_2 plasma-ashing, a 100-nm-thick Cr film is sputtered onto the glass surface using an RF sputtering system (EB1100 PVD) (Figure 3 r and s). To ensure electrical connectivity, the insulators must be covered by the Cr film. Therefore, the Cr film must be thicker than the SiO_2 film. After the AZ5206 photoresist is removed using AZ remover, the micro-heater array device is complete (Figure 3 t).

2.3 Heating Experiment Conditions

To verify the usefulness of this device, heating experiments were conducted using a thermo-responsive gel. The thermo-responsive gel was a thermo-responsive polymer solution with 10 wt% poly (N-isopropylacrylamide) (PNIPAAm). Above 32°C, the PNIPAAm solution became clouded and generated a white solid. Below 32°C, the thermo-responsive gel reversibly liquefied from a white solid to a liquid. To monitor the heating characteristics, discolorations of the thermo-responsive gel were observed using an optical microscope (MX-10C; HIROX Co., Ltd.).

3. Results

3.1 Fabrication Results

A structure that enables crossing of the lead wires as required for two-dimensionally arrayed heating elements on the glass substrate was devised, and the materials comprising the micro-heater array device were optimized, thereby establishing a fabrication process flow. First, microscopic views after the patterning of the oxidized silicon are shown in Figure 4, confirming that the Cr lead wires were covered by oxidized silicon. Second, microscope views after the final process are shown in Figure 5. The oxidized silicon patterning is sandwiched between the two lead wires. When the continuity was tested, the lead wires were isolated. These microscope views and continuity tests confirmed the successful formation of the structure with properly crossed lead wires. Therefore, a micro-heater array device that can be heated with regional selectively was successfully fabricated.

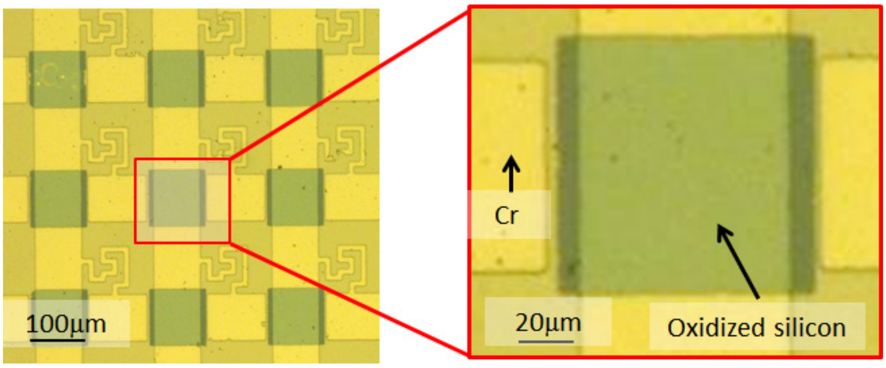

Figure 4. *Microscope photographs* after the patterning of the oxidized silicon

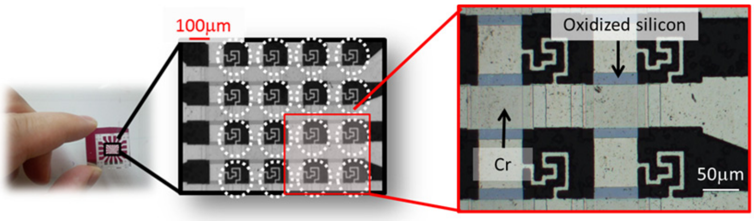

Figure 5. Microscope views *after the final process. The* oxidized silicon patterning is *sandwiched between the two lead wires*

3.2 Heating Results

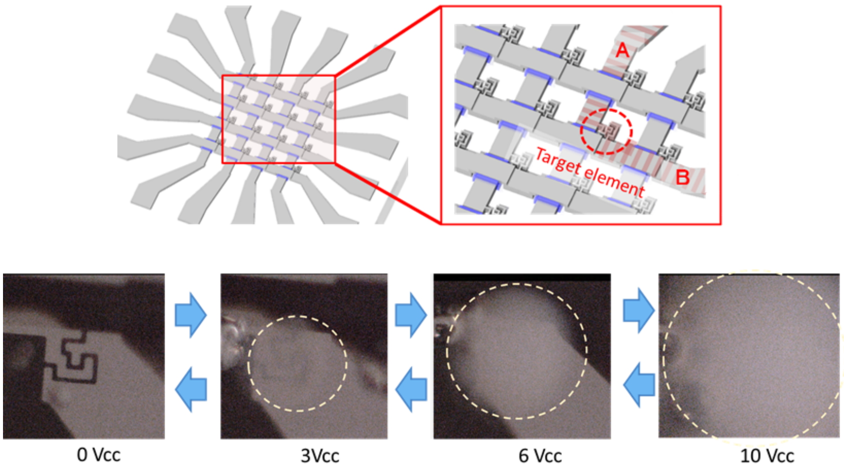

Figure 6. Results of heating by applying a voltage. An arbitrary target micro-heater element on the device was heated. A voltage was applied to the two lead wires connected to the ends of the selected heater element (A-B). The white discoloration is cured gel. Moreover, the white solid area increased with increasing voltage

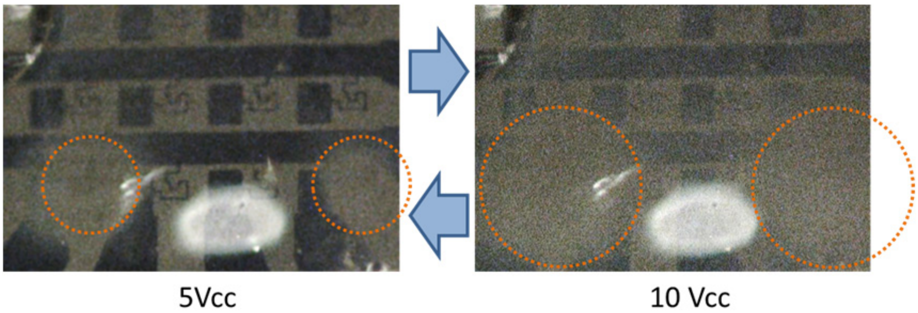

Figure 7. Simultaneous heating of a number of micro-heaters was also confirmed. Elements to which a voltage was applied are indicated by dotted circles

To confirm the heating characteristics of this micro-heater array device, a 10 wt% PNIPAAm solution was dropped onto the device and heating experiments were conducted. As shown in Figure 6, an arbitrary micro-heater on the device was heated. A voltage was applied to the two lead wires connected to the ends of the heater element. The 10 wt% PNIPAAm solution became clouded and generated a white solid when the voltage was applied, and the boundary between the white solid and liquid was 32°C. Moreover, the white solid area increased with increasing voltage. Therefore, the temperature of the micro-heater can be controlled by changing the voltage. As shown in Figure 7, simultaneous heating of a number of micro-heaters was also confirmed. Based on these results, it should also be possible to apply different voltages to different micro-heaters, bringing them to different temperatures as a result. Therefore, the generation of a temperature gradient on the glass chip is possible.

4. Discussion

This micro-heater array device can be heated with regional selectivity and controlled to arbitrary temperatures. Therefore, it can also generate temperature gradients. This device expands not only the versatility of biochemical applications, but also engineering applications such as sensors by using micro-heater. When this micro-heater device and a thermo-responsive gel are combined, a system that can alter its hardness with regional selectivity can be established. The cellular morphology and differentiation of various cells are influenced by the hardness of the culture environment (Maskarineca et al., 2007; Tan et al., 2003). Therefore, analysis of the relationship between cellular morphology and the hardness of the culture environment used of this device provides useful knowledge of cancerization principles. Moreover, since the holding of micro-objects such as cells used for gelatinization is possible, cellular sheets of arbitrary shape can fabricated by simultaneously heating a number of micro-heaters. Using PIPAAm thermo-responsive polymer, which can control the hydrophilic-hydrophobic temperature changes, high speed hydrophilic-hydrophobic patterning is possible. Therefore, highly efficient separation of proteins, steroids, amino acids, and peptides should be possible. And when this micro-heater device and a thermo-responsive gel are combined, and the thermo-responsive gel included a drug liquefied from a solid to a liquid, regional drug release is also possible. Therefore elucidation of the functional expression of cells may also be possible. As this fabrication process was established using MEMS technology, the materials used to form the heater elements and lead wires could be converted to other materials that can be deposited by sputtering or chemical vapor deposition (CVD), depending on the application. Although the width of the heater elements in this work was 10 µm, fabricating the heater elements by electron beam (EB) lithography would enable the fabrication of scaled-down heater elements such as nano-heaters.

5. Conclusion

Because a micro-heater can be supplied with heat rapidly and at the micro-scale, various applications have been studied. Specifically, micro-heaters have recently been used in biological cell research. To expand the versatility of biochemical applications of the micro-heater, the development of micro-heater array systems that are heatable with regional selectivity at arbitrary locations is required. In this research, the design and materials of a micro-heater array device were optimized and a fabrication process was established. A micro-heater array device that can be heated with regional selectivity was fabricated, and the usefulness of this device was verified using a thermo-responsive gel. Control of the temperature distribution on a glass substrate was successfully demonstrated. The micro-heater array device that was developed in this research has several advantages. First, it can be manufactured at low cost. Second, it can be heated with regional selectivity. Third, since heat is generated in proportion to the applied voltage, the generation of a temperature gradient and temperature control are also possible. Fourth, the device can be integrated with sensors, actuators, and circuits by adopting semiconductor processes. Therefore, this work not only expands the versatility of biochemical applications using micro-heaters, but also enables new applications of micro-heater devices.

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