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Micro-heaters embedded in PDMS fabricated using dry peel-off process

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ABSTRACT

The present report describes a reliable fabrication method of micro-heaters embedded in polydimethylsiloxane (PDMS), and shows the characterization of the micro-heaters. Metallization of PDMS is achieved using a dry peel-off process which involves modifying the surface properties of the substrate and metal patterns through self-assembled monolayer (SAM) and manually peeling off the PDMS with embedded metal layers. Thus, micro-heaters embedded in PDMS can be fabricated by a simpler and easier way compared to a conventional method (*e.g.* patterning a conducting composite of PDMS using a razor blade). As a result, Au micro-heaters embedded in PDMS were successfully fabricated without any chemical swelling and contamination. Micro-heaters on a glass substrate were also fabricated for comparison with those embedded in PDMS. For heating up to 90 °C, the micro-heaters embedded in PDMS needed only ∼90 mW compared to those fabricated on the glass substrate needed ∼530 mW. Moreover, we could not observe any degradation of the micro-heaters by thermal stresses that confirmed by repeatability (10 thermal cycle with a range of 25–89 °C) and stability test (20 min at 90 °C). Micro-heaters took less than 60 s to reach the target temperature (90 °C) and spent less than 60 s to drop to room temperature. The spatial temperature distribution was not significantly varied with materials of the substrate (*i.e.* PDMS or glass).

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1. Introduction

The development of lab-on-a-chip (LoC) systems has been one of the dominant themes in analytical instrumentation for chemical and biomedical analysis applications over the past decade [1–3]. Among the components, heat generation and thermal control are critical in LoC for precise control of protein synthesis and the amplification of nucleic acid molecules using polymerase chain reaction (PCR) [4–8]. Also, chemical synthesis in micro-reactors requires precise temperature control [9,10]. Heating blocks [4] or heating wires [10,11] can be used for the temperature control of LoC devices while the temperature control can be achieved much precisely by micro-heaters [5–8].

Polydimethylsiloxane (PDMS) is one of the most popular polymer for LoC devices because it is optically transparent, flexible, chemically resistant, bio-compatible, inexpensive and easy to fabricate [7–10]. Usually, heaters for PDMS LoC devices are fabricated on a glass substrate, and assembled with PDMS micro-fluidic channels. On the other hand, micro-heaters embedded in PDMS have the advantages such as flexibility, rapid prototyping and greater compatibility with existing PDMS chips [12].

A conducting composite of PDMS has been investigated by some groups who create a mixture of various fillers (*e.g.* carbon black powder or silver platelets) and PDMS prepolymers, thus producing an inherently conducting PDMS [12–14]. To pattern the conducting PDMS, the gel-state conducting PDMS is molded into photoresist (PR) patterns, then unnecessary portions of the gel are removed from the mold surface using a razor blade. However, this method is restricted by two reasons: (1) a razor blade can damage mechanically the mold (*e.g.* PR or PDMS) and (2) large volumetric change of conducting PDMS deteriorate the spatial resolution of heating area (coefficient of thermal expansion: 310 ppm $^{\circ}C^{-1}$ for PDMS [15]). Another approach, metallization of PDMS, also have been investigated through a self-assembled monolayer (SAM) as a molecular adhesive [16–20]. Recently, we demonstrated that (3-mercaptopropyl)trimethoxysilane (MPTMS) can drastically promote the adhesion between Au and PDMS using a liquid deposition method [18]. Also, we showed the fabrication of Au micro-patterns embedded in PDMS without any chemical contamination of PDMS during the transfer process [19].

Here, we demonstrate a simple process for fabrication of Au micro-heaters embedded in PDMS. The key point in fabricating the Au micro-heaters is the embedding of a metal layer into PDMS

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carried out by a "dry peel-off" process, which involves modifying the surface properties of the substrate and metal patterns through SAM treatment and manually peeling off the PDMS with embedded metal layers [19]. Moreover, characterization was conducted for evaluating resistance, time response, area for effective heating, temperature repeatability and temperature stability. Also, we characterize micro-heaters on a glass substrate for comparison with those embedded in PDMS.

2. Experiment

2.1. Fabrication procedure

Chemicals and experimental details are noted at S1 in Supplementary material. The fabrication strategy of Au micro-heaters embedded in PDMS is schematically shown in Fig. 1. A Si wafer was treated with a piranha solution [H_2SO_4 (98%)/H_2O_2 (30%), 3:1 (v/v)] for 10 min to clean the surface of the Si substrate and grow a fresh thin oxide layer. After dehydration at 150 °C for 10 min, a sparse MPTMS layer was formed on the Si substrate using vapor deposition for 10 min (Fig. 1a). A thin Au layer (thickness: 100 nm) deposited onto the substrate by thermal evaporation was lithographically patterned and wet-etched (Fig. 1b). The substrate with both Au patterns and photoresist (PR) patterns were immersed in perfluorodecyltrichlorosilane (FDTS) solution (5 mM in hexane) for 5 min (Fig. 1c). After PR removal, the substrate with Au patterns was treated with an ethanolic solution of 20 mM MPTMS for 2 h (Fig. 1d). A 10:1 (by weight) mixture of PDMS base/curing agent was poured on the substrate (thickness: 3 mm), then heat-cured in an oven at 60 °C for 3 h, and then maintained at room temperature for 12 h (Fig. 1e). Finally, the PDMS with Au micropatterns was manually peeled off from the Si substrate (Fig. 1f).

For the comparison, Au micro-heaters were also fabricated on 1 mm thick glass substrate (Micro-slide glass, S9111, Matsunami, Japan). Au (100 nm) was deposited on the glass substrate with Cr (10 nm) as an adhesion layer. After lithography and wet-etching of Au/Cr layers, PR was removed with acetone and ethanol, followed by rinsing with deionized water and drying with a stream of air.

2.2. Design of the micro-heaters and characterization

The micro-heaters were designed with different geometric shapes (Fig. 1g). The width of heating wire was 40, 80 and 160 μm with a length of 15 mm. Pads for electrical connection to power supply were designed for 3 × 3 mm. Joule heating was applied to the micro-heaters operations. To apply the voltage and measure the electrical resistance, DC voltage current source/monitor (6240A, ADCMT, Japan) was used. Copper wires and Au micro-heaters were electrically connected with silver paste (Electroconductives, D-362, Fujikura Kasei Dotite, Japan).

A simple setup was utilized to characterize the micro-heaters (Fig. S1 in Supplementary material). The temperature of micro-heaters and peripheral area (*i.e.* PDMS or glass) were monitored by infrared (IR) thermo-microscopy (FSV-GX7700, Apiste). The emissivity for the IR imaging was set to 0.86 for the PDMS and 0.95 for the glass substrate [21,22]. Because of a large difference of emissivity between PDMS (0.86) and Au (0.02), it is difficult to measure the temperature of both PDMS and Au, simultaneously. Thus, we set the emissivity of the substrate materials (*i.e.* 0.86 for PDMS or 0.95 for a glass) to the thermo-microscope, and measured the highest temperature of the target area. The point of the highest temperature in PDMS part is adjacent to the Au micro-heaters.

3. Results and discussions

3.1. Fabrication results

Au micro-heaters embedded in PDMS were successfully fabricated using a dry peel-off process (Fig. 1h and i). Because a sparse MPTMS layer between Si and Au made the moderate adhesion, the Au layer was not exfoliated during the wet-process (*e.g.* photolithography, etching, and rinsing). Buckling and wrinkles were observed at the surface of Au micro-heaters, but it did not critically affect the performance of the micro-heaters.

3.2. Electrical resistance

At the first attempt, the electrical resistance of Au micro-heaters was measured. For theoretical calculation, a simple Eq. (1) for resistance was used where R is the resistance, ρ_R is the electrical resistivity, L is the length and A is the cross-sectional area:

$$R = \rho_R \frac{L}{A} \tag{1}$$

We measured the resistance when the applied voltage was 1 V. The results of experiments and theoretical calculation were agreed well (Fig. S2 in Supplementary material). Thus, we can assume that the micro-heaters were not broken or cracked after the transfer from the donor substrate (*i.e.* Si wafer) to PDMS.

3.3. Temperature with respect to the applied voltage and power consumption

To have a comprehensive understanding of the relationship between the temperature and applied voltage, the temperature of micro-heaters was measured during increasing the applied voltage

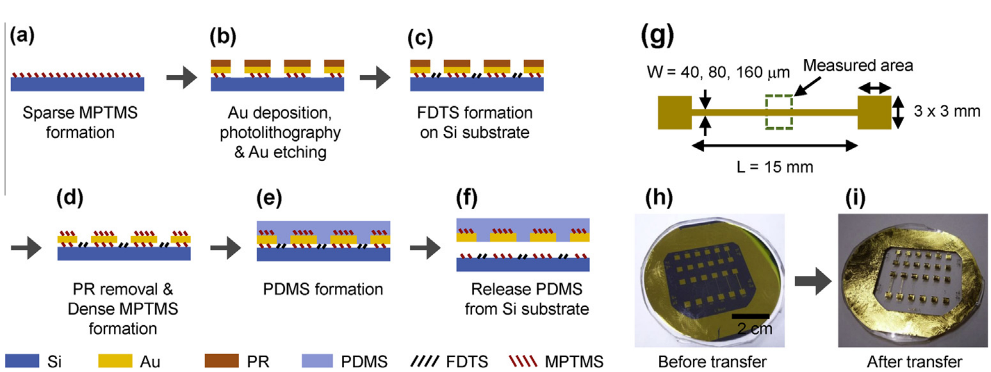

Fig. 1. (a–f) Schematic illustration of the fabrication process of Au micro-heaters embedded in PDMS. (g) Design of the micro-heaters, an optical image of Au micro-heaters, (h) before the transfer and (i) after the transfer.

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