Modelling and fabrication of low operation temperature microcages with a polymer/metal/DLC trilayer structure

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Received 8 September 2005; received in revised form 17 February 2006; accepted 8 March 2006
Available online 24 April 2006

Abstract

Multi-finger microcages with metal/diamond like carbon (DLC) bilayer and polymer/metal/DLC trilayer structures were analyzed, simulated and fabricated. Modelling by finite element analysis showed that a soft polymer can be used together with DLC to form a normally closed microcage which can be opened by 90° angle at a temperature of ∼400 K, which is much lower than that of a metal/DLC bilayer structure previously demonstrated. The opening temperature for a microcage is independent of the finger dimensions, but is simply determined by the thermal expansion coefficient difference of two materials used. Microcages with SU8/DLC bilayer and SU8/Al/DLC trilayer structures have been fabricated and fully closed microcages with diameters of ∼40 μm have been obtained. Initial electrical tests have showed these devices open by more than 90° at a temperature lower than 150 °C, roughly in agreement with the theoretical analysis.

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Keywords: Microcage; Microgripper; Thermal actuator; DLC; Trilayer; Bilayer

1. Introduction

Microgrippers such as microcages and microtweezers are important microtools with great potential for applications in micro-precision, micro-control and microfabrication [1,2], and those with small dimensions of a few tens of micrometers in diameter have the potential for use in biological and bio-medical applications [3–5]. In particular a microcage fixed on a multi-freedom robotic arm could be used to capture, transport and manipulate bio-cells for dissection and injection etc., which is a requirement for single cell proteomics. In this case it is necessary to be able to select a single cell, and transport the specimen to a platform to carry out various measurements using nanoelectrodes. There is great interest in direct observation of cells (or infected tissue) and their response to drugs injected in to them for drug discovery and cancer research, all of which rely on cell (or tissue) selection and capture. Unlike the glass pipette or microtweezer, the microcage captures the specimen by confining or trapping it without applying a force directly on it, thus avoiding potential mechanical damage to the specimen. The primary requirements for such biological microtools are low operation temperature, low power consumption and small dimensions. Attempts have been made by many research groups to develop microcages for biological applications. Most of these have been made from multilayer structures with a ‘normally open’ state and large dimensions >500 μm in diameter [4]. These devices consist of bilayer structures utilizing thermal stress for actuation, and require a constant power supply to close during operation, leading to a sharp rise in the temperature of the device and the surrounding environment. A normally closed microcage of size >500 μm was developed with a pressure as the actuation force by Kim and co-workers [5]. In this case, the cage can be opened by applying pressure to the membrane on which the microcage was made. However, the fingers have a fixed curvature, and are unable to open widely. It also requires an external pressure source to actuate the device, and so is not suitable for portable applications. In this study, normally closed multi-finger microcages with dimensions down to ~40 μm have been successfully fabricated [6,7]. The devices were made from a metal and diamond...
like carbon (DLC) bilayer structure. The highly compressively stressed DLC layer lifts the bimorph fingers upwards once they are released from the substrate, forming a closed microcage. The device can be opened by passing a current through the metal layer in millisecond pulses at a power of a few tens of milliwatts. This device is superior to others in that it is normally closed, and is opened by the pulsed current—hence there is no need to supply a constant power to keep the device closed. However, the actual operation temperature of the microcages is too high for practical biological applications. Here we report the modelling and fabrication of a new type of microcage with a low operation temperature using a polymer/metal/DLC trilayer structure.

2. Theoretical analysis

For the metal/DLC bilayer microcage structure, the DLC layer possesses a high compressive stress, while the metal has no residual stress. Once a bilayer structure is patterned, and released from the substrate, the residual stress in the DLC layer makes the bilayer curl upwards. The radius of curvature, $R$, is expressed by [8,6]

$$\frac{1}{R} = \frac{6\varepsilon(1 + m)^2}{d[3(1 + m)^2 + (1 + mn)(m^2 + (mn)^{-1})]} = \varepsilon S$$

(1)

where $\varepsilon$ is the strain and is related to the stress, $\sigma$, by $\varepsilon = \sigma/E$, $E$ being the Young’s modulus, $d = d_1 + d_2$, with $d_1$ and $d_2$ being the thicknesses of the DLC and the metal layers, respectively, $n (= E_1/E_2)$ and $m (= d_1/d_2)$ are the ratios of the Young’s moduli $E_i$ and the layer thickness. $S$ is a constant for the fixed materials and a particular structural configuration. The curvature is linearly correlated to the stress of the DLC layer, and a DLC with a high stress can be used to form a microcage with small dimension. The curvature of the metal/DLC bilayer structure can also be adjusted through control of the layer thickness ratio and the finger length. Thus a normally closed microcage can be obtained by using appropriate materials of a given material stress and thickness ratio.

Once a closed microcage is formed, a raised temperature will generate a thermal stress on the topside of the bilayer, leading to opening of the microcage. The opening temperature of the microcage device with a fixed finger length $L$ is mainly determined by the difference in the thermal expansion coefficients (TEC) of the two materials used. Assuming $\theta$ is the inclination angle of the fingers, then the change of the finger angle from $\theta_1$ to $\theta_2$ is expressed as [7]

$$\theta_2 - \theta_1 = \frac{180L}{\pi} \left( \frac{1}{R_2} - \frac{1}{R_1} \right) = \frac{180L}{\pi} \Delta\alpha \Delta T S$$

(2)

where $\Delta\alpha$ is the TEC difference of two materials, $\Delta T$ is the temperature rise, and $R_1$ and $R_2$ are the radii of curvature before and after the temperature change, respectively. For a normally closed microcage with a diameter in the range of 20–100 $\mu$m, $S$ is in the range of 0.5–2 $\times 10^{-6}$ m$^{-1}$. $\Delta\alpha\Delta T$ is the thermal strain generated by Joule heating. The opening angle of the device is proportional to the product of $\Delta\alpha$ and $\Delta T$. As discussed previously, a 90° opening is sufficient to capture a micro-object in practical applications, hence Eq. (2) can be rearranged as follows:

$$\Delta T = \frac{\pi}{2LS\Delta\alpha}$$

(3)

The opening temperature of the device is inversely proportional to the difference in TECs. In order to reduce the operation temperature, it is better to select two materials with a large difference in TECs. It is clear that DLC is one of the best materials to be used as the bottom layer to fabricate the microcages, not only because it has a high compressive stress to form a closed microcage, but also because it has a small TEC, which maximizes the thermal stress for the bilayer structure, and opens the microcage easily at a lower operation temperature. For the top layer, a material with a high TEC is most desirable. Fig. 1 shows the dependence of the opening temperature on TEC of a top layer material with $S$ as a parameter. The TEC of semiconductor materials is generally small, and typically less than $5 \times 10^{-6}$ K$^{-1}$, hence they are not suitable for such ‘normally closed’ devices with low operation temperature. The TEC of metals are typically in the range of 10–30 $\times 10^{-6}$ K$^{-1}$, leading to an opening temperature between 420 and 780 K, which is sufficient for use in micro-precision and control applications, but too high for practical biological applications. The TECs of polymers are in the range of 30–200 $\times 10^{-6}$ K$^{-1}$, which is much larger than those of metals. A microcage with a polymer/DLC layer will have an opening temperature between 320 and 420 K. PMMA, polyimide and SU8 are common polymers available for microelectronic industrial applications with a similar TEC of 50–55 $\times 10^{-6}$ K$^{-1}$ [9,10], which corresponds to an opening temperature of 380–390 K. Microfabrication process for these polymers are widely available, making them a better choice for this device application. Further decrease of operation temperature can be achieved by using other polymers with higher TECs such as PDMS and nylon [11]. A question may arise as to whether a polymer with a low Young’s modulus will be strong enough to balance the DLC layer which has one of the highest Young mod-
A finite element analyses (FEA) based on FEMLAB software (a commercial available plug-in software for MATLAB\textsuperscript{TM})\textsuperscript{1} was opened by raising the temperature using the metal heater, a process may vary. imide as they have similar TEC values, though the fabrication process are electrically non-conductive materials. Therefore, an electrical resistive heating element is required to generate Joule heating. SU8 is chosen as the polymer in this work, but the results are also applicable to PMMA and polyimide as they have similar TEC values, though the fabrication process may vary.

3. Finite element simulation

In order to clarify whether a soft polymer can be used together with DLC layer to fabricate a closed microcage and can be opened by raising the temperature using the metal heater, a finite element analyses (FEA) based on FEMLAB software (a commercially available plug-in software for MATLAB\textsuperscript{TM})\textsuperscript{1} was used to model the curvature and performance of the bilayer and trilayer structures as a function of temperature. A 2D-plane stress model using a parametric non-linear solution was employed to solve this non-linear and large deformation problem. A step of 0.2 GPa was used in the simulation. For simplicity, only the constant temperature mode was used in modelling the actuation at high temperatures. This is sufficient to compare the simulation with the experimental results and to optimize the device structures. As all the fingers are identical with a symmetric structure, only a cross-section of one finger was chosen for modelling. Ni was used as the metal layer for a bilayer structure with $\alpha = 12.7 \times 10^{-6} \text{K}^{-1}$, while Al was used as the heating element for the trilayer structure with $\alpha = 21.3 \times 10^{-6} \text{K}^{-1}$. The material properties used in the simulation are listed in Table 1.

For the trilayer structure, it was found that the curvature of the structure is unaffected by the presence of an SU8 layer less than 100 nm thick if the Ni layer is much thicker than 100 nm. This implies that a thin polymer can be used as a coating layer to minimize the heat loss from the surface of a bilayer microcage as the thermal conductivity of polymers is normally more than two orders of magnitude smaller than those of metals and the DLC layer. As the polymer thickness increases, it becomes an effective active blocking layer to modify the curvature of the trilayer structure, and eventually becomes the dominant force to balance the DLC layer, and forms the curled trilayer structure. At an optimized thickness ratio and finger length, a closed microcage using a polymer/Al/DLC trilayer can be formed. Fig. 3(a) and (b) show the curvature of a polymer/Al/DLC trilayer structure at room temperature for two different DLC stresses. The thicknesses of the DLC, Al and SU8 layers are 85 nm, 100 nm and 1 µm, respectively, and the finger length is 200 µm. For a DLC layer with a stress of 1 GPa, the finger only curls slightly. Increasing the stress in the DLC layer to 6 GPa causes the fingers to curl by nearly 180° at room temperature—similar to that of the Ni/DLC bilayer structure. Fig. 4 shows the dependence of the radius of curvature on the compressive stress of the DLC layer for devices with a finger length of 200 µm. The curvature of the finger is a linear function of stress, in agreement with the analytical model (see Eq. (1)).

As the temperature rises, the top SU8 layer expands much more than that of the DLC layer, and generates a compressive thermal stress on the topside of the trilayer, leading to the opening of the fingers. Fig. 3(c) and (d) show the opening of the trilayer structure at 400 and 500 K, respectively. The device opens by 90° at 400 K, a much lower temperature than the 700 K required for the Ni/DLC bilayer structure [7]. Fig. 5 shows the curvature angle of the microcages as a function of operation temperature for the measured and simulated results. The circles with error bars were previously measured for a Ni/DLC microcage with $L = 200 \mu$m [7], and the line with triangles is the FEA modelling results for the same bilayer structure with $L = 160 \mu$m. The measured temperature for an opening angle of ~90° is 700 K for this Ni/DLC microcage, and is consistent with the simulation results. This indicates the FEA model used for the simulation is accurate and applicable to the trilayer device.

For the polymer/Al/DLC trilayer microcage with the same device dimensions as for Fig. 3(b), the curvature angle of the

\begin{table}[ht]
\centering
\caption{Physical material properties employed in the FEA simulation \cite{10,12,13}}
\begin{tabular}{|c|c|c|c|c|}
\hline
Material & $\alpha (\times 10^{-6} \text{K}^{-1})$ & $\rho \, (\Omega \text{m})$ & $\kappa \, (\text{W m} \text{K}^{-1})$ & $E \, (\text{GPa})$ & $\xi \, (\times 10^{-3} \text{K}^{-1})$ \\
\hline
Ni & 12.7 & $2 \times 10^{-7}$ & 83 & 210 & 3.0 \\
Al & 21.3 & $2 \times 10^{-8}$ & 237 & 70 & 3.5 \\
DLC & $\sim 1$ & $>10^7$ & 450 & 600 & n/a \\
SU8 & 52 & $>10^7$ & 0.2 & 4 & n/a \\
\hline
\end{tabular}
\end{table}

\textsuperscript{1} The MathWork Inc., 3 Apple Hill Drive, Natick, MA 01760-2090, USA.
trilayer, initially 180°, decreases rapidly with increasing temperature, and reaches ~90° at a temperature of 400 K, and ~12° at a temperature of 500 K—a much lower temperature than that used for the bilayer structure. In combination with the pulse current operation, the opening temperature of 400 K is more suitable for practical biological applications.

In order to obtain a microcage with small dimensions, the thickness of each layer has to be reduced accordingly. But to obtain a reasonable resistance for Joule heating, the Al thickness should remain at a similar level, e.g. 40 nm. The relatively thick Al layer will affect the curvature and operation temperature of the trilayer structure. The analytical model of Eq. (1) for a simple bilayer is no longer suitable for this structure, and the curvature and performance of the trilayer structure can only be assessed by FEA modelling. Fig. 6(a) shows the curvature of a microcage...
with a device finger length of 70 μm and SU8/Al/DLC thicknesses of 250/40/40 nm. The structure is also in the closed state of 180° at room temperature. Note that without the Al layer, the angular deflection of the same bilayer structure is ∼220°—much larger than 180°—but it does not have sufficient force to open the bilayer structure. Fig. 6(b) is the curvature of the same device at 400 K, showing an opening of the structure by ∼90°. The curvature of this device as a function of temperature is also shown in Fig. 5, in a similar way to that of the trilayer structure with different layer thickness, further indicating that the opening temperature is determined by the TEC of the materials rather than the finger length.

4. Fabrication and characterisation

The fabrication of the microcage devices was divided into two parts. The first part was to confirm whether an SU8/DLC bilayer can form an effective closed microcage, and the second part was to investigate the trilayer structure with the Al electrode included for the heater. The first simplified SU8/DLC bilayer devices were made using a single mask process detailed previously in Ref. [7]. The process flow is as follows: the DLC was grown on a bare crystalline silicon substrate using an S-bend filtered cathodic vacuum arc (FCVA) which produces a highly pure C⁺ ion beam. The typical base pressure prior to deposition was ∼1.5 × 10⁻⁶ Torr, and the growth rate was ∼10 nm/min [12–14]. The DLC film is very smooth with a surface roughness less than 2 nm with no visible macro-particles. A 1.5 μm thick
SU8-2 was then spin coated onto the DLC layer at a speed of 1500 rpm, and patterned using standard photolithography. The exposed DLC was etched in an oxygen plasma with the SU8 as an etch mask. The etch gas was then switched to SF6 to reactively etch the silicon from under the fingers. This step was time controlled etch to ensure release of the fingers only, leaving the middle parts of the microcage still attached to the silicon substrate. Fig. 7 shows scanning electron microscope (SEM) images of the SU8/DLC (∼500/40 nm) microcages after release from the substrate with finger lengths between 20 and 50 μm. Since the radius of curvature of the fingers is fixed (R ≈ 22 μm) for all devices with the same thickness ratio, the microcage with L = 20 μm is only half-closed, while that with L = 40 μm is fully closed, and that with L = 50 μm is ‘over curled’. The bilayer structures show that polymers can be used together with DLC to form fully closed microcages with small dimensions.

For a trilayer microcage with a heating element, an extra mask step is required to form the electrode bond pads. The two-mask, self-alignment process flow is highlighted in Fig. 8. Al (100 nm) and Cr (10 nm) were deposited on a 3″ diameter crystalline silicon wafer by thermal evaporation. The Cr layer was used as an etch mask for the Al bond pads. Patterns were formed by photolithography, and followed by Cr and Al etch in Cr- and Al-etchant, respectively, with the positive photoresist AZ5214E as the soft etching mask. The Cr layer on top of the Al bond pads was then removed by proprietary Cr etchant without affecting the other layers. The remaining finger release etch process is the same as for the simplified SU8/DLC structures. Fig. 9 shows an optical microscope image of a six-finger microcage made by this two-mask process before release etch.

Fig. 10 is the SEM image of the released devices. The microcage with L = 30 μm is nearly closed, while that with L = 40 μm is curled more than 180°. These devices have again shown that a polymer with a low Young’s modulus can be used together with DLC to make fully closed microcages with small dimensions.

The SU8 has thus been used as both a structural element and also as an etch mask to remove the DLC on the outside of the active area. The key issues for these processes are the hardening of the SU8 layer and the etch selectivity of the DLC relative to the SU8 as the latter material can also be removed partially by an O2 plasma. The DLC has a higher etch rate in the O2 plasma than that the SU8, and the etch time is typically <60 s only. As the SU8 is relatively thick, a short time etching in the O2 plasma only removes a small part of the SU8 layer, and the remaining SU8 becomes part of the trilayer structure. The
plasma etch rate of SU8 can be modified by the SU8 curing conditions. Two extra process steps were added to enhance the SU8 hardness: an extra UV light exposure of up to 30 s after development, and post-development curing at >150 °C for 20 min. These additional process steps effectively reduced the SU8 etch rate in the O₂ plasma from ~200 to ~60 nm/min. It was found that SU8 can be etched in an SF₆ plasma at a rate of ~100 nm/min, which leads to a thinning of the SU8 layer to ~500 nm after the releasing process. This is entirely suitable for the polymer/DLC structure.

The fabricated microcages have been electrically tested on wafer using a probe station with a CCD camera to measure the displacement of the devices. Current was applied to the device from a Keithly source (model 240), and the voltage was monitored. The initial test showed that the microcages can be opened by ~90° at an average temperature of 100–150 °C, which was estimated from the resistance change. Fig. 11(a) is an optical photograph of a six-finger microcage before applying the current. When a current of 2.5 mA (the measured voltage was 3.2 V) was applied to the device, the fingers opened with an angular deflection of ~80°. The estimated average temperature is ~150 °C, in agreement with the theoretical analysis. A relatively large device with a finger length of 110 μm was used for the test due to the limited magnification of the microscope. However, the opening temperature of the microcage is independent of the finger length as discussed above.

5. Summary

Multi-finger, ‘normally closed’ microcages consisting of a metal/DLC bilayer or a polymer/metal/DLC trilayer structure were modelled and fabricated. Finite element analysis-based modelling revealed that an optimal thickness ratio exists for a DLC layer stress of 6 GPa, causing the fingers of the microcage to curl by 180°, forming a normally closed structure with small dimensions. The temperature to open the fingers of a polymer/DLC bilayer structure by 90° is only ~400 K, which is much lower than the 700 K required to open a Ni/DLC bilayer structure previously reported. The opening temperature is determined by the relative thermal expansion coefficient of the materials used, and is independent of the geometrical dimensions of the devices. This low opening temperature makes the polymer devices more suitable for practical biological applications. Microcages with trilayer structure have been fabricated and initial electrical tests have showed these devices open by more than 90° at a temperature lower than 150 °C, in agreement with the theoretical analysis.
Acknowledgement

This project was sponsored by the Cambridge-MIT Institute under grant number 059/P, and partially by EU FP6 scheme under the project PROMENADE.

References


Biographies

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