Graphene-Based Bimorph Microactuators

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Supporting Information

ABSTRACT: A novel graphene-on-organic film fabrication method that is compatible with a batch microfabrication process was developed and used for electromechanically driven microactuators. A very thin layer of graphene sheets was monolithically integrated and the unique material characteristics of graphene including negative thermal expansion and high electrical conductivity were exploited to produce a bimorph actuation. A large displacement with rapid response was observed while maintaining the low power consumption. This enabled the successful demonstration of transparent graphene-based organic microactuators.

KEYWORDS: Graphene, actuator, organic, thermal, mechanical, sensor

Graphene, the thinnest elastic material, has superb electronic and mechanical properties that make it a promising host for various device applications.1–5 In particular, graphene has a distinctive negative coefficient of thermal expansion (CTE) (contraction upon heating),4,6 which is in contrast with the normal behavior of conventional materials. Therefore, in a case where a thin layer of graphene coupled to a different material member is heated, the asymmetric thermomechanical response may lead to large bimorph actuation.

Microactuators, which can convert electric energy directly to mechanical energy, have attracted attention for a range of biomimetic applications, including insect-like motion, flying robots, and fishlike manipulators.7–11 These microactuators were designed to reproduce the important features of natural muscle, such as response speed, displacement, and controllability. A range of inorganic materials including shape memory alloys and piezoelectric ceramics have been evaluated as actuators.12,13 However, the high operating temperature and voltage restrict their range of applications. On the other hand, polymer-based soft actuators including dielectric elastomers, conjugated polymers, and polymer gels have advantages, such as flexibility, lightweight, and optical transparency.10,14,15 Nevertheless, the slow response, short life cycle, and low-energy conversion efficiency limit the performance of polymer-based actuators. More importantly, there are difficulties in their fabrication method. The majority of the listed materials require postprocessing steps or assembly methods that are not compatible with conventional batch microfabrication steps. Hence, there are significant challenges in developing actuator materials for a large displacement and a rapid response at low voltages, as well as in developing a compatible fabrication method.

Figure 1 shows a schematic diagram of the fabrication process for a graphene/epoxy hybrid bimorph actuator. First, graphene films were grown on a 4 in. SiO2/Si wafer coated with Ni catalyst layer using the procedures described elsewhere.16,17 The transmittance of the graphene films was ∼78.8% at the 550 nm wavelength, which indicates that the average number of graphene layers is nine because the transmittance of an individual graphene layer is reduced by ∼2.3% (Supplementary Figure S1a, Supporting Information).18,19 The sheet resistance of graphene films was ∼200 Ω/sq measured using a four-probe Van der Pauw setup. In addition, the Raman spectra measured for graphene films on SiO2 substrates showed a strongly suppressed defect-related D-band peak (Supplementary Figure S1b, Supporting Information). The graphene-grown wafer was cut to a 2.5 × 2.5 cm² piece as a handling substrate. Gold contact pads and alignment markers were fabricated by photolithography. A serpentine microheater pattern of the graphene on Ni film was formed again by photolithography and O2 plasma reactive ion etching (RIE). Finally, a thin epoxy (SU-8) cantilever (L:W:H = 180 × 200 × 3.4 μm) with a transmittance of approximately 99% at 550 nm (Supplementary Figure S1a, Supporting Information) and a thick support body (50 μm measured by surface profilometer) were fabricated by two sequential photolithography steps.20,21 The graphene/epoxy hybrid cantilever microactuators

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were transferred from the wafer substrate using a bi-sacrificial-layer wet release process. First, a buffered oxide etchant (BOE) was used to remove the SiO2 layer (see also Figure 2a). The separated epoxy/graphene/Au/Ni layers were then transferred and soaked in an aqueous iron(III) chloride (FeCl3) solution (1 M) for 5 min to remove the Ni metal layers. After the Ni dissolution, the samples were set free in the solution (see also Figure 2b). This process generates a patterned stand-alone graphene-on-organic film structure that is detached from the carrier substrate without compromising the integrity of the graphene sheets. The resulting cantilever chips were cleaned with deionized (DI) water and made ready for the test. A rigid substrate (glass, quartz or silicon wafer) was used as a support during the measurement. The resistance of the graphene ranged from 50 to 60 kΩ. This process resulted in transfer yields approaching 99%.

Figure 2c shows a field emission scanning electron microscopy (FE-SEM) image of the four arrays of the graphene-epoxy hybrid microcantilevers. The inset micrograph clarifies the typical structure of a graphene serpentine pattern and a 3.4 μm thick cantilever beam at the oblique view. The conducting graphene films were directly heated, and the organic epoxy film was warmed up by the deposited heat upon applying the electric power. The compatibility of aromatic ring structure of graphene and epoxy is believed to provide a firm π−π interaction interface between these two layers, which can lead to an efficient bonding through the interface without delamination up to 0.4% strain. The asymmetrical thermal expansion between the graphene and epoxy films is expected to cause upward deflection of the actuator upon heating the setup (Figure 2d).

A model of the graphene actuator was created using the simulation package (ANSYS, Inc.) where the fixed support boundary condition was applied to the base of the epoxy beam. The free convection condition was applied to all surfaces in contact with air (including the top part of the graphene layer). The uniform internal heat generation in the graphene layer was derived from electrical power divided by the graphene volume. The mechanical and thermal properties that were used as constant values are listed in Table 1.

The maximum temperature ($T_{\text{max}}$) of the beam is a function of the convection heat transfer coefficient ($h$) and electrical power ($P$); therefore the simulation was done by systematically changing the values of $h$ and $P$. Summarizing the simulation results, a simple equation for $T_{\text{max}}$ as a function of $P$ (W) and $h$ (W/m$^2$ K) was obtained

$$T_{\text{max}}(h, P) = (Ah + B)P + C$$

where $A$, $B$, and $C$ are $-14$ $\text{°C}^2 \cdot \text{m}^2/\text{W}^2$, $29.3 \times 10^3$ °C/W/m, and $17.3$ °C, respectively. Equation 1 was implemented to fit the experimental data $T_{\text{max}} (P)$ by a chi-square minimization method following the Levenberg–Marquardt (L-M) algorithm (Figure 3a). The only fitting parameter was $h$, calculated to be $1027 \pm 23$ (W/m$^2$ K). In the next step the beam tip deflection ($d$) was investigated which is a function of CTE and $T_{\text{max}}$. Since the deflection...
measurement is independent of previous set of data, it could also be used to extract useful information about the system. Following the same procedure as in the previous part, a series of simulations were done for \(d(\mu m)\) at different \(T_{\text{max}}\) and CTE \((/C)\). The equation below was obtained for \(d\) as a function of CTE and \(T_{\text{max}}\):

\[
d(\text{CTE, } T_{\text{max}}) = (L \cdot \text{CTE} + M)(T_{\text{max}} + N)
\]

where \(L, M,\) and \(N\) are \(-3.1 \times 10^3 \mu m, 0.15 \mu m/\degree C,\) and \(-10.5 \degree C,\) respectively. Here the graphene CTE was used as the fitting parameter to fit the above equation to the measured data (Figure 3b) using the same method as the previous step, and CTE was found to be \((-6.9 \pm 0.6) \times 10^{-6} (/\degree C)\). This is in good agreement with the reported values.\(^4,6\) The small error bars obtained for \(h\) and CTE demonstrate the accuracy of the values obtained for the fitting parameters. The overall layout of the beam and the graphene layer, the steady state temperature distribution of the beam from a top view, the beam deflection from a side view, the equivalent (von-Mises) elastic strain and the thermal strain of the entire beam are shown for the maximum input power in supplementary Figure S2 in the Supporting Information. The deflection is not to scale and is exaggerated for clarity. The model was clipped in two directions to show the thermal and von-Mises strain contours inside the model.

Figure 3a shows the maximum temperature of the microcantilever as a function of the input power for the measured data and the simulation results. The cantilever was actuated by increasing the temperature. At the reference temperature (\(\sim 17 \degree C\)), the beam was bent down slightly (\(\sim 1.5 \mu m\)), which is in agreement with the resistance (supplementary Figure S3, Supporting Information). With the onset of power input (0.02 mW), the beam rapidly bent up and became flat. As the temperature was increased, the cantilever beam bent up smoothly in the positive direction. The temperature of the cantilever changed linearly to 36 \degree C with a supplied power up to 1.26 mW. The deflection of the cantilever increased linearly with temperature or the electrical input power. From these plots, the effective conversion factors of \(0.17 \mu m/\degree C\) and 2.58 \mu m/mW were calculated, which matches the theoretical quantitative analysis shown in Figure 3b. These conversion factors can be increased using a thinner epoxy layer. The nonsmooth transition through the zero point is an indication of the presence of initial strain in the material at rest. This can be the result of the fabrication process and the induced changes in graphene topology on the given substrate, which is consistent.
with the sudden uptake observed at the extremely low input power, as shown in Figure 3c. The tip displacement of the cantilever beam as a function of time was measured for eight repeated cycles of an input sweep voltage from 1 to 4 V for 1 s followed by off power period for 0.1 s in each cycle. (d) Resistance of graphene serpentine microheater as a function of the displacement of cantilever tip. The dashed line shows a polynomial fit (order of three) to the data.

In summary, a microactuator was developed based on a graphene-on-organic film hybrid bimorph. A new fabrication method allowed for the monolithic integration of a graphene layer as an active component. A photolithographically defined graphene sheet resistor could be used for both actuation and sensing. The extraordinary mechanical, optical, and electrical properties of graphene were implemented into a working device.

**ASSOCIATED CONTENT**

Supporting Information. Characterization of the sample, simulation data, and a movie file that shows actuation. These materials are available free of charge via the Internet at http://pubs.acs.org.

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