Determination of Mechanical Properties and Actuation Behaviors of Polypyrrole–Copper Bimorph Nanoactuators

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Abstract—This paper reports on a polypyrrole (ppy) based bimorph nanoactuator. The nanoactuator consists of ppy nanowire and Cu thin film. To create a nanoactuator, a (10 ± 5)-nm-thick Cu film was precisely deposited on one side of a (13.7 ± 1.0)-μm-long and 270 ± 20 nm diameter ppy nanowire. The bimorph was thermally actuated using the mismatch of the coefficients of thermal expansion (CTEs) between Cu and ppy. The CTE of ppy nanowire was obtained by measuring the deflection of the bimorph. The measured CTE of ppy was $(12 \pm 1 \times 10^{-6})$/K, which was found to be much less than CTE of a typical polymer. Further, the amount of the force generated by the ppy/Cu bimorph was measured by lateral force microscopy (LFM). The measured force from the nanoactuator was approximately 1 nN at $\Delta T = 100$ K.

Index Terms—Coefficient of thermal expansion (CTE), lateral force microscopy (LFM), nanoactuator, polypyrrole.

I. INTRODUCTION

POLYPYRROLE (ppy) is a conducting polymer whose electrochemical and mechanical properties have been actively investigated. Since the availability of conducting ppy film via electrochemical oxidation of pyrrole was reported [1]–[3], there have been many reports describing the use of this material in electrode applications [4]. The interest in this material arises from its versatility, including adherence to supporting electrodes, electroactive behavior, and stability from chemical reactions [5]. This material is also well known for its expansion and contraction through redox reactions with ions in the liquid environments [6], [7]. Such geometrical variation with insertion and depletion of ions was used for the developments of electrochemical actuators [8]–[15]. Currently, ppy can be produced in thin film [16], nanotube [17], [18], or nanowire [8], [18] forms. The nanotubes and nanowires can be fabricated by electrochemical deposition. Because of their size, the electrical and mechanical properties of such nanomaterials are different from the bulk values, where their mechanical properties depend on the dimension [15]. Recently, novel nanoactuators using ppy have been demonstrated inside an electrolyte through electrochemical actuation [18], showing its potential usage for some future miniaturized actuator.

The nanowire is an ideal template for a nanoscale actuator, attributed to its length and geometrical shape. The first nanowire-based actuator was demonstrated using multiwalled carbon nanotubes [19]. The mismatch of coefficients of thermal expansion (CTE) between the carbon nanotube and the deposited oxide materials (i.e., bimorph configuration) induced the deflection of the bimorph under temperature variation. Recently, the author's group demonstrated a technique to measure force generation from the tip of such a carbon-nanotube-based bimorph nanoactuator by lateral force microscopy (LFM) [20], [21]. However, due to very high stiffness of carbon nanotubes, the bimorph can only be used at very high temperature changes (i.e., $\Delta T \leq 800$ K) [19]. A polymer-based nanoactuator would have the advantage of a low elastic modulus and can be used for relatively low-temperature operation compared to the carbon-nanotube-based nanoactuator. Typically, polymers are known to have much larger thermal expansion rate than metals, but the coefficient of ppy nanowire has not been reported yet.

In this paper, a thermally actuated bimorph nanoactuator based on ppy nanowire and Cu film is demonstrated. This nanoactuator does not require a liquid environment, which is needed for electrochemical actuation [18]. The measurement of the tip movement of the bimorph nanoactuator yielded the CTE of ppy nanowires (i.e., the ppy material in the form of nanowire used for measurement was 13.7 ± 1.0 μm long and 270 ± 20 nm diameter). Further, the force generated at the bimorph tip was measured using the LFM.

II. THERMAL DEFLECTION AND COEFFICIENT OF THERMAL EXPANSION OF PPY

To investigate the CTE of a ppy nanowire, a ppy–Cu bimorph structure (i.e., nanoactuator) was fabricated by depositing a 10-nm-thick Cu layer on one side of a ppy nanowire. The ppy nanowires were grown inside commercially available anodized aluminum oxide (AAO) (Whatman Ltd.) membranes by electrochemical deposition. First, an Ag film was coated on one side of the AAO membrane and used as the metal electrode for electroplating. The ppy electrolyte was composed of 1-M pyrrole monomer (Alfa Aesar) and 1-M sodium chloride (NaCl) (Alfa Aesar) in deionized water. The membranes with nominal
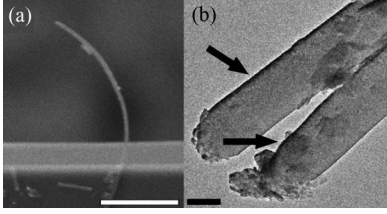
Fig. 1.   (a) SEM picture on the initial curvature of the Cu–ppy nanobimorph. The copper film is inside of the curvature (left side of the bimorph). The scale bar is 5 μm. (b) TEM picture showing the deposition of Cu films (black arrow, 20 nm thick) on top of ppy nanowires, which were prepared separately to monitor the film deposition. The scale bar is 200 nm.
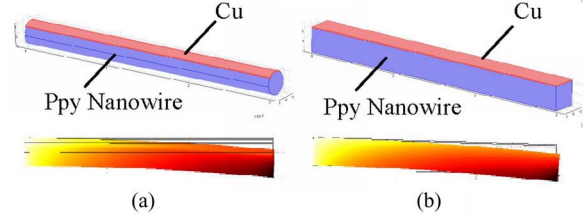

Fig. 2.   (a) Geometrical model assuming circular cross section and its simulation result in a side view. Cu film of 10-nm thickness was modeled on top of the ppy nanowire. The tip deflection (26 nm) is exaggerated four times from the simulation result. (b) Geometry and simulation result (22 nm) from rectangular cross-sectional model. The diameter in model (a) and the height and width in model (b) of the ppy bars are 270 nm. The thicknesses of Cu films are 10 nm, the lengths of the two bimorphs are assumed to be 3 μm, and the temperature variation was 160 K from room temperature.

pore diameters of 200 nm were used to grow ppy nanowires. Applied current of 1–2 mA/cm^2 through a platinized titanium anode (Stan Rubinstein Assoc. Inc) was kept constant using a galvanostat (263A-1, Princeton Applied Research, AMETEK Inc.) during the electrochemical deposition process. The growth rate and length of ppy nanowires were controlled by both applied current and deposition time, which was confirmed by an optical microscope (Hi-scope advanced KH3000, Hirox) and a scanning electron microscope (SEM) (XL-40, Phillips). After deposition, the AAO membrane was dissolved in 5-M sodium hydroxide (NaOH) and cleaned with deionized water several times. The effect of NaOH on the mechanical property of ppy is miniscule, since the dissolving process took only a few minutes. After the solution was dried, the substrate was broken into small pieces to create cantilevered ppy nanowires. The circular cross section of individual ppy nanowires was verified using SEM. For accurate measurement of deflection and force, the Cu e-beam evaporation was done in the vertical direction from the top view [see from the left direction in the pictures of Fig. 1(a)]. The bimorph had an initial curvature due to the CTE mismatch between ppy and Cu due to the cooling after deposition of the Cu thin film (see Fig. 1). The double-layer interface was verified by a transmission electron microscope image (TEM) of a ppy nanowire, which was dispersed on a TEM grid. The thickness of the Cu film matched the nominal thickness when the metal was evaporated [see Fig. 1(b)].

From [22], the thermal deflection of a bimorph can be calculated by equation, assuming rectangular cross sections of the two constituting materials

$$\Delta = \frac{3L^2(\alpha_{\mathrm{Cu}} - \alpha_{\mathrm{ppy}})\Delta T(d_{\mathrm{Cu}} + d_{\mathrm{ppy}})}{d_{\mathrm{Cu}}d_{\mathrm{ppy}}}$$

$$\times \left[\left(\frac{d_{\mathrm{Cu}}}{d_{\mathrm{ppy}}}\right)^2 \frac{Y_{\mathrm{Cu}}}{Y_{\mathrm{ppy}}} + 4\frac{d_{\mathrm{Cu}}}{d_{\mathrm{ppy}}} + 6 + 4\frac{d_{\mathrm{ppy}}}{d_{\mathrm{Cu}}} + \left(\frac{d_{\mathrm{ppy}}}{d_{\mathrm{Cu}}}\right)^2 \frac{Y_{\mathrm{ppy}}}{Y_{\mathrm{Cu}}}\right]^{-1} \tag{1}$$

where ΔT is the differential temperature, L is the length of the bimorph and d is the thickness (diameter) of each layer, α is the CTE of each layer, and Y is the modified Young's modulus, $E/(1 - \nu)$ of each layer, where E is the Young's modulus and ν is the Poisson ratio. Because the CTE of ppy is not known, it is not possible to predict the amount of deflection. However, one can estimate α_{ppy} by measuring the amount of deflection Δ.

Equation (1) gives a bimorph deflection based on a rectangular cross section, while the bimorphs used in the experiments have a circular cross section. Therefore, we modeled deflection behaviors of bimorphs with a circular cross section and a rectangular cross section, respectively, using finite-element analysis software (Comsol 3.5a, Comsol Labs), in order to provide a correct prediction of the actuation behavior. As shown in Fig. 2(a), the deflection from the bimorph with a circular cross section was larger than that with a rectangular one by 1.18 throughout the temperature range ($\Delta T = 60$, 110, and 160 K). Therefore, the calculated deflection using the (1) was scaled by the ratio 1.18 in (2). By applying the same principle, the force prediction in (4) can also be scaled using the force simulation results. The factor Δ from (1) can be scaled to Δs by the ratio of the two simulation results as follows:

$$\Delta s = \Delta[\text{from (1)}] \times \frac{\text{simulated deflection (circular)}}{\text{simulated deflection (rectangular)}}. \tag{2}$$

Thus, this scaled deflection, Δs from (2), was compared with measurements.

To measure the thickness of the Cu film, a separate substrate was prepared. The Cu film was deposited on both the ppy nanowires and the separate substrate at the same time. The measured thickness of the Cu film using atomic force microscopy (AFM) was 10 ± 5 nm. In the calculation, the thickness of the ppy nanowire was set identical to the diameter, which was measured as 270 ± 20 nm using high-magnification SEM. The measured length of the bimorph (see Fig. 1) was 13.7 ± 1.0 μm. The Young's modulus and Poisson ratio of Cu are 100 GPa and 0.34, respectively [23]. The Young's modulus of ppy nanowire is approximately 2 GPa from [17], [24], and [25]–[27]. Especially, temperature dependence of Young's modulus of ppy film reported in the [25] has been used for the simulation. The Poisson ratio of ppy was set to 0.4 from the earlier reported value [28].

A custom-built thermal stage was set up inside an SEM chamber (XL-40, Phillips) to control temperature of the nanoactuator stage. The repeatability of the bimorph actuation was tested by multiple thermal cycles between room temperature (293 K) and 403 K. In the first thermal cycle, there was the largest deflection observed that could not be reproduced in the following cycles
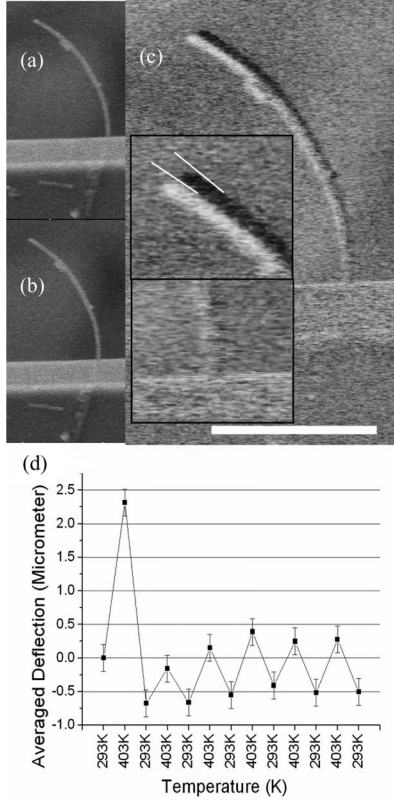
Fig. 3. (a) SEM image of the Cu–ppy bimorph nanoactuator at room temperature (after five thermal cycles). (b) Same bimorph at 403 K (110 K above room temperature) after the image (a) was taken. (c) Overlapped image from (a) and (b). The scale bar is 5 μm. The inset shows a magnified view at the tip. (d) Consecutive record of the tip movements of bimorphs. The initial location of the tip was set as zero. Positive (negative) amount means the tip moved toward right (left), implying stretching (curving) under heating. The pictures (a)–(c) are from the last thermal cycle of an actuator.

[see Fig. 3(d)]. The reason for such large movements at the initial cycles is not from thermal stress, but from the partial release of the residual film stress in a Cu film [29], [30]. In a thin Cu film, at the initial thermal cycle within $\Delta T = 100$ K, the change of film stress is ∼0.15 GPa [30]. The deflection of a bimorph cantilever by a film-stress change can be described by the Stoney equation [31] as follows:

$$\sigma_{\text{Cu}} = \frac{\delta Y_{\text{ppy}} d_{\text{ppy}}^2}{3L^2 d_{\text{Cu}}(1 - \nu)} \qquad (3)$$

where δ is the initial deflection, scaled by (2). From the deflection of first thermal cycle in Fig. 3(d), and (3), the calculated film stress change is 0.04 GPa, which agrees well with the reported values of thermal stress of metal films [29], [30].

The directions of the tip movement under temperature change were such that the bimorph was stretched when heated and curved when cooled. In the following thermal cycles (second and third thermal cycles), there were unpredictable tip movements. But, after fourth thermal cycle, there were repeatable stretching and curving from the tip movements, and the deflections were similar in each cycle. Therefore, only the thermal cycles after the fourth cycle were used for the estimation of the ppy CTE.
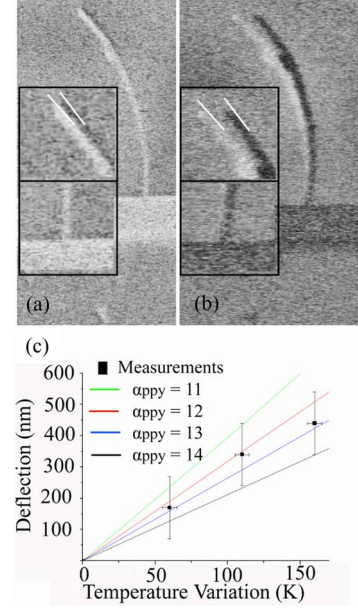

Fig. 4. (a) Overlap image of the identical bimorph in Fig. 3(a) with the temperature change by 60 K (inset is a magnified view at the tip), and (b) with the temperature variation by 160 K (inset shows the largest tip deflection at the tip). (c) Comparison between deflection measurements and temperature change. The lines are the predictions adjusted by (2).

Fig. 4 shows the thermal deflections at chosen temperature changes 60, 110, and 160 K. As expected from (1), a higher temperature difference gives larger deflection at the tip of the bimorph. The amounts of the deflections were measured directly from SEM pictures and, when those numbers are given as inputs to the left-hand side of (2), the CTE of the ppy was obtained to be $(12 \pm 1 \times 10^{-6})$/K, which is close to the reported value $[(10.179 \times 10^{-6})$/K] for ppy matrix composite [32]. However, it is much lower than the typical plastic materials and the reported value $[(44 \times 10^{-6})$/K] [33]. Even negative CTE $[(-1579.70$ and $-6248.50 \times 10^{-6})$/K)] of ppy coated on carbon fiber has been reported [34]. Therefore, CTE of ppy could be strongly dependent on the fabrication process and the specimen dimensions. Unlike all of the reported CTE of ppy measured in millimeter-scaled specimens, we reported CTE of ppy in a nano-scaled specimen for the first time. Thermal actuations were done multiple times (at least three times) at each temperature elevation, with three temperature elevations, 60, 110, and 160 K.

III. FORCE MEASUREMENT

The maximum force generated at the tip of a rectangular bimorph nanoactuator can be calculated, assuming that the bimorph was forced to be stretched straight from natural deflection [20], [22] as follows:

$$F = \frac{w(\alpha_{\text{Cu}} - \alpha_{\text{ppy}})\Delta T}{L} \frac{Y_{\text{Cu}} Y_{\text{ppy}} d_{\text{Cu}} d_{\text{ppy}} (d_{\text{Cu}} + d_{\text{ppy}})}{d_{\text{Cu}} Y_{\text{Cu}} + d_{\text{ppy}} Y_{\text{ppy}}}. \qquad (4)$$

Using the same simulation models shown in Fig. 2, the force generated from the bimorph with a circular cross section was larger than rectangular cross section by 1.3. Therefore, the numbers
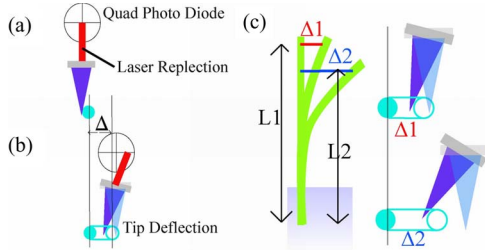
Fig. 5. (a) and (b) When the AFM cantilever is under torsion, the laser reflection falls on an off-center location, and its location can be recorded by the voltage difference between the left and right half of the quad-photodiode. Here, the deflection of the bimorph is assumed to be identical to the lateral movement of the scanner because the amount of torsion is nanometers, while the deflections are microns in scale. (c) When the AFM scans deeper location (such as L2), the amount of torsion gets larger as recorded in the increased voltage.

calculated from (4) were scaled by 1.3 for the correct theoretical prediction. From this calculation, the predicted maximum force at the tip of the bimorph was 1.3 nN at $\Delta T = 100$ K, for a bimorph with 10-nm-thick Cu film and 270-nm-diameter ppy nanowire.

The LFM technique was used to measure the actuation force generated from the nanoactuator. When an AFM tip is engaged at the tip of a bimorph, the AFM tip experiences a reaction force from the bimorph, making the AFM cantilever twist [see Fig. 5(a) and (b)]. The amount of twist or torsion is recorded by the voltage difference (I_{L-R}) generated between the left and right half of the AFM quad-photodiode depending on the location of the reflected laser light beam. If the correlation between the torsion and I_{L-R} is calibrated, one can obtain information on the amount of force applied to the AFM cantilever by recording I_{L-R}. Prior to the force measurement, the calibration was performed using an AFM cantilever (ContPt, Nanoworld) in the AFM system (Model Nano-I$_2$ AFM, Pacific Nanotechnology). From this calibration, the coefficient was found to be $c_L = 0.20 \pm 0.09$ nN/mV [35]. The recorded force (reaction with thermal actuation) is calculated by the following:

$$f = c_L I_{L-R}. \tag{5}$$

The amount of the reactive force experienced by the AFM depends on the location along the bimorph; it becomes larger as the scan location moves toward the mounting edge of the bimorph [see Fig. 5(c)]. The amount of the force experienced by the AFM cantilever at room temperature is given by

$$f = \frac{3\pi r^4 E_{\text{ppy}} \Delta}{4L^3} \tag{6}$$

where r is the diameter of ppy nanowire, and Δ is the deflection of bimorph until the AFM tip disengages the bimorph. It is expected that the force f will increase, as the AFM cantilever in the LFM mode scans along the length of the bimorph; the shorter the length L is, the larger the recorded f is. The LFM measurements in Fig. 6(a)–(c) can be used to estimate Δ, which was found to be ~0.5 μm increases per LFM scan step (i.e., 150 nm in L direction). When the AFM tip is scanning again at higher temperatures, the thermally generated force from the bimorph
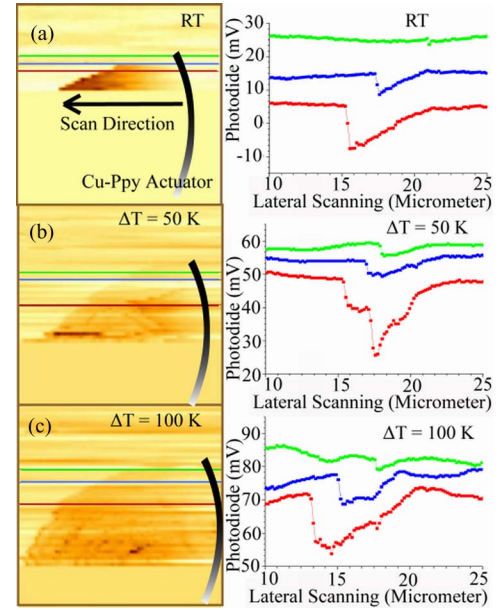

Fig. 6. (a) LFM measurement in the arrow direction in each scan at room temperature. The displayed area is 10 by 10 μm and one scan is 150 nm apart from each other. From each scan area, three scans at different locations (colors) are plotted in the same color to show the gradual increase of the reaction from the nanoactuator along its length. A square area (in 40 by 40 μm) was chosen to include the location of the tip of bimorph. The resolution of the scan area was 256 pixels. Therefore, one step of neighboring scans has 150 nm distances. (b) Same scan at 50 K above the room temperature. (c) Same scan at 100 K above the room temperature.
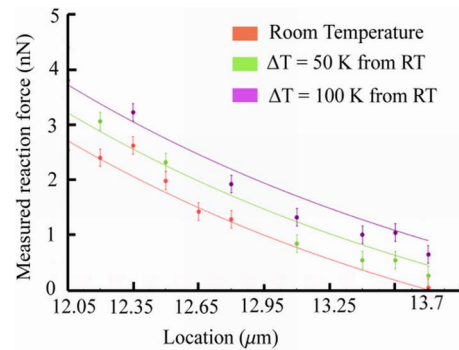

Fig. 7. Measured I_{L-R} in the room temperature was converted into reaction force using (5), and they were plotted against their measurement locations (red dots). The red solid line is the expectation to (6). The measurement at the tip location (13.7 μm) was set as zero of the vertical scale. The measurements of elevated temperatures (green and purple dots) were plotted with (green and purple) solid curves, which are expectations from the sum of the (4) and (6) at each temperature variation. The net thermally generated force is the difference between the green and red dots (curves) in case of 50-K elevation and between purple and red dots (curves) in case of 100-K elevation.

pushes the AFM cantilever further. This additional amount of torsion in the AFM cantilever appeared as an increase in I_{L-R}, which can be calculated from (4). Therefore, at an elevated temperature, the expected total reaction experienced by the AFM cantilever is the sum of amounts from (4) and (6) at any location along the length of bimorph.

In the first step of the force measurement, the reaction from the bimorph at the room temperature was recorded [see Fig. 6(a)]. The scanning started at a location away from the actuator tip; therefore, there was no response when the AFM tip was positioned slightly offset from the bimorph tip. As the AFM tip scanned laterally for a step of 150 nm, the expected saw-tooth-shaped I_{L-R} appeared and its size became large, as the AFM tip scanned deeper toward the bimorph nanoactuator support. In the next step of the force measurement, the thermal stage was set at predetermined temperature elevations (50 K up and 100 K up from room temperature), and the lateral scans were repeated in the same manner [see Fig. 6(b) and (c)]. The net actuation force was obtained by subtracting the force measurement at room temperature from that at higher temperature (see Fig. 7). As clearly seen in Fig. 7, the force becomes larger at higher temperatures, and the amount is about 1 nN at 100 K above room temperature, as expected from (4).

IV. CONCLUSION

A ppy nanowire-based bimorph nanoactuator has been fabricated, modeled, and tested. The nanoactuator was fabricated by depositing a thin Cu film on one side of a ppy nanowire. While there was a nonrepeatable deflection in the first actuation step due to the change in film stress, the thermal deflection over a finite-temperature cycle was found fairly repeatable in the following cycles. For the first time, the CTE of ppy nanowire was measured by characterizing the thermal deflection behavior of a nanoactuator. The measured CTE was $\alpha_{\text{ppy}} = (12 \pm 1 \times 10^{-6})$/K. Further, the amount of force generated at the tip of the nanoactuator was measured using the LFM technique. The ppy-/Cu-based nanoactuator generated 1 nN force at $\Delta T = 100$ K.

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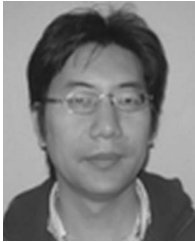
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