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Controlling Au electrode patterns for simultaneously monitoring electrical actuation and shape recovery in shape memory polymer

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This paper presents an effective approach to achieve efficient electrical actuation and monitoring of shape recovery based on patterned Au electrodes on shape memory polymer (SMP). The electrically responsive shape recovery behavior was characterized and monitored by the evolution change in electrical resistance of patterned Au electrode. Both electrical actuation and temperature distribution in the SMP have been improved by optimizing the Au electrode patterns. The electrically actuated shape recovery behavior and temperature evolution during the actuation were monitored and characterized. The resistance changes could be used to detect beginning/finishing points of the shape recovery. Therefore, the Au electrode not only significantly enhances the electrical actuation performance to achieve a fast electrical actuation, but also enables the resistance signal to detect the free recovery process.
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1. Introduction

As one of the most important shape memory materials, shape memory polymers (SMPs) have been rapidly developed in the last three decades [1–7]. The SMPs have the capability of memorizing a permanent shape and can be programmed for one or many temporary shapes upon exposure to an external stimulus [8–12]. Having a stable polymer network and a reversible switching transition of the polymers are two pre-requisites for the shape memory effect (SME) in the SMPs [13–16]. A specific thermal or other type of stimulus is generally required to trigger the SME in SMPs [17–21]. Apart from conventional thermal heating, thermally responsive SMPs can also be triggered using light (thermally conductive heating) [16], magnetic field (inductive heating) [22,23], or electric current (Joule heating) [24–34]. To achieve electrically resistive Joule heating, various strategies have been studied to improve the electrical conductivity of SMP matrix by incorporating it with conductive ingredients, such as particles, hybrid filler, mat and nanopaper [24–34]. The internal resistive joule heating method has many advantages such as fast speed, convenience, uniform heating, and potential for remote control [33–36]. Electrical actuation of the SMP can be achieved at a low voltage of 10.0 V [25,28,37,38]. The conductive ingredients could also improve the thermal conductivity of the SMP matrix to achieve a fast response [26,31,39,40]. This electrically induced SME is especially useful for applications where direct heating is impossible such as in self-deployable aerospace structures [41], implanted biomedical devices [42,43], micro-actuators and sensors [44–46].

Gold (Au) film is widely used as a flexible electrode on an elastomeric substrate, because of its good electrical conductivity, good ductility, reliable performance, easy fabrication and a low price [47,48]. In addition to these advantages, the Au film could be used to detect the strain of the substrate from the variation of resistance of the Au films, which was due to the change on the Au surface morphology [49]. Previous studies showed that there was a distinct relationship between the resistance of gold and strain of the elastomeric substrate at room temperature [47,49], which is mainly due to generation of two microscopic structures of Au film, i.e., creases and cracks [50]. The modulus of a common epoxy-based SMP is of 10–50 MPa at 20 °C above T_g, which means that there are some micro-cracks in the Au film on the epoxy substrate [50]. The Au film could provide two functions, i.e., driving the substrate to recovery its original shape by Joule heating, and also detecting

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the recovery process [51,52]. During the free recovery process, the temperature gradually rises leading to the increase of resistance [35], whereas the strain was continuously released leading to the decrease of resistance by closing of micro-cracks. Utilizing these two opposite effects on the resistance, both the beginning and the finishing points of free recovery process could be monitored. In previous work, we briefly reported the sensing effect tested in an oven [35]. However, the sensing effect in Joule heated free recovery process has not been explored.

In the present study, we present an effective approach to significantly improve the electrical properties and shape recovery performance of SMP triggered by Joule heating using the patterned Au electrode. Both electrical conductivity and temperature distribution in the SMP were significantly improved by controlling the Au electrode pattern. Temperature dependence of electrical properties and electrically actuated shape recovery performance of SMP nanocomposite have been systematically studied, and electrically actuated shape recovery behavior and the temperature profile during the actuation were characterized. During the free recovery process, the resistance of Au coating experiences an "S"-shape change due to the increase of temperature and closure of micro-cracks, which could be used to detect recovery ratio. Therefore, the Au coating is expected not only to enhance the electrical properties (thus resulting in an improved recovery behavior to provide high speed electrical actuation of the SMP), but also to monitor the free recovery process.

2. Experimental details

The SMP used in this study was epoxy-based thermoset SMP resin with a glass transition temperature (T_g) of 110 °C. The resin was cured at a ramp rate of approximately 1 °C·min^{-1} from 22 °C to 80 °C and kept for 3 h before being ramped to 100 °C for 3 h, after which it was ramped to 150 °C and kept for 6 h to produce the final samples (with a dimension of 40 × 25 × 2 mm^3). To ensure the durability of the Au coating and improve Au layer adhesion, the SMP samples were polished using an aqueous solution containing Al_2O_3 particles at room temperature of 22 °C and a speed of 20 μm h^{-1} to obtain a smooth surface. They were then cleaned using distilled water to remove Al_2O_3 particles and heated up to 130 °C for 5 mins.

The Au electrode was deposited using a magnetron sputter (EMITECH K575X). To improve the binding capacity between the Au layer with the polymer substrate, a chromium (Cr) layer of 15 nm was initially deposited, followed by a 50 nm Au layer. The patterns of the electrodes were controlled using shadow masks. The epoxy based Au nanocomposites were then annealed at 130 °C for 30 min to release the internal stress between Au coating and epoxy substrate.

Considering the thickness of Au/Cr coating, (about 75 nm in total) is far less than the other two in-plane scales, the differences in the electrical currents normal to the surface of the coating could be neglected. Therefore, the electrode could be treated as a 2-D device, of which resistance could be measured as a sheet resistance as expressed using Equation (1), in which ρ presents the resistivity, and t, W and L presents thickness, width and length of thin film, respectively.

$$R_s = \frac{\rho}{t} = R\frac{W}{L} \tag{1}$$

Meanwhile the sheet resistance of the Au coating could be considered as a parallel connection between Cr layer of 15 nm and Au layer of 50 nm. Using the resistivity values of Cr and Au at 25 °C (i.e., 125 nΩ m and 22 nΩ m), the sheet resistance of Cr/Au coating is estimated to be 0.42 Ω.

In this study, two types of Au electrode patterns were fabricated as listed in Table 1. The electrodes were designed according to the estimated sheet resistance of Cr/Au coating (approximate to 0.42 Ω). In reality, there were errors between the designed resistance and the actual resistance, which is due to the deviation of surface roughness and errors from the shadow mask patterning.

The sample I was used to determine the electrical properties of Cr/Au coating and its thermal response in an oven. The electrical resistance was determined using the van daur Pauw four-point probe instrument (Keithley 2400), and the temperature range was from 25 °C to 130 °C at a heating rate of 5 °C/min. The sample II was used to determine the relationship between electrode resistance and the temperature of the sample in a Joule heated process, and to study the relationship between electrical properties of coating and the recovery ratio in the free recovery process driven by a DC power source ZPS-305D with fixed voltages of 11.5 V, 13.3 V and 14.9 V, respectively.

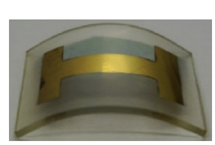
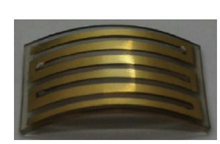
In the free recovery process, a pre-deformation was applied to the sample. Before the free recovery process, the sample was heated to 130 °C, deformed into a "U" shape under an external force, then cooled down to room temperature, thus resulting in a bending shape. The electrically responsive shape recovery process was recorded and monitored using a video camera and infrared video camera (VarioCAM HiRessl and JENOPTIK Infra Tec., respectively).

3. Results and discussion

3.1. The electrical properties of Au electrode enabled SMP

Electrical resistivity of the Au electrode enabled SMP was determined using a Keithley 2400 resistivity tester and a Keithley 2000 picoammeter/voltage source. When a Kelvin connection was

Table 1
The images and designed/experimental data of three samples fabricated in this study.

Sample		
Label	I	II
Number of stripe electrodes	N = 1	N = 6
Size of electrodes	20 × 5 mm^2	35 × 2.5 mm^2
Designed resistance in 25 °C	1.68 Ω	35.28 Ω
Actual resistance In 25 °C	1.67 Ω	40.25 Ω
Error	−0.6%	+14.1%

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