Electrical and thermal effects on electromechanical performance of stretchable thin gold films on PDMS substrates for stretchable electronics

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ABSTRACT

Stretchable electrodes comprising thin gold films with initial nanocracks on elastic substrates of poly(dimethylsiloxane) are developed in this work, which can be stretched reversibly while maintaining conductivity to an applied uniaxial strain as large as 120%. Reliable electromechanical performance is essential for the application of stretchable electronics as bioelectronic interfaces under various working conditions; therefore, the electrical and thermal effects on the electromechanical performance of the stretchable gold film electrodes are investigated in this work. It is found that the stretchability deteriorates to some extent depending on the electrical and thermal treatments. Microstructures and stress analysis in the cracks are studied, along with crack quantification. It is believed that the decrease of the stretchability is ascribed to the combination of the crack widening by thermal mismatch stress and crack tip smoothening by the electromigration phenomenon. Therefore, the current density and temperature through the electrodes should be controlled within a certain range for wide applications in order to maintain a stable performance of the electrodes. This study also provides some guiding significance for the design of other stretchable electronic devices composed of two materials with large different physical properties.

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I. INTRODUCTION

Stretchable electronics has been the hot spot in the research community and, recently, it has been developed widely, which combines the advantages of the elastic substrates and conductors, making many exciting applications possible.^{1–4} Stretchable electronics, which can maintain electrical conductivity when suffering a large mechanical strain, has been developed for many applications, including biological signal detection,^{5,6} stretchable sensor skins,^{7,8} stretchable transistors, and wearable communication devices.^{9,10} Stretchable electrodes can be made of some advanced materials, such as conductive composite,¹¹ carbon nanotube,¹² and graphene films.¹³ Another kind of stretchable electronics is the

stretchable gold film electrodes that comprise thin gold films with characteristic micro/nanocracks on the elastic substrates. The stretchable electrodes based on the gold film deposited on polydimethylsiloxane (PDMS) have been studied extensively since they were developed.^{14–18} It was reported that the micro/nanocrack gold thin film has superior stretchability as the cracks in the gold film allow the film to elongate by deflecting and twisting out of plane so that a large applied strain induces only a small and elastic strain in the film. As a result, the energy release rate at the crack tip is minimized, inhibiting crack growth across the gold film.¹⁶ Recently, there have been many advancements in the field of stretchable gold film electrodes, such as the sensitive capacitive-type strain sensor

using wrinkled ultrathin gold films,¹⁹ and the helical gold nanotube film as a stretchable micro/nanoscale strain sensor prepared by magnetron sputtering using carbon nanocoils (CNCs) as the template.²⁰ Liu *et al.* reported a novel kind of stretchable gold film electrodes based on nanopile interlocking to improve the adhesion of the gold film significantly.²¹ Yan *et al.* even invented an interesting material combining the stretchable gold film with a self-healable polymer substrate complementary to commonly used PDMS-based thin film metal electrodes.²²

The stretchable micro/nanocrack gold film electrodes fabricated by the magnetic sputtering method can be stretched uniaxially to a strain as large as 120% while maintaining conductivity in our previous work.²³ Although the stretchability mechanism of the stretchable gold film electrodes has been fully investigated, few studies have been reported on the electromechanical performance of the stretchable electrodes under working conditions such as electrical and thermal treatments.²⁴ Since reliable electrical conductivity is essential for the stretchable electronics in various applications and circumstances, in this work, the performance of the stretchable gold film on a PDMS substrate is comprehensively investigated under working conditions. Stress analysis and crack quantification are discussed in detail, with some interesting phenomena observed and explained.

II. EXPERIMENTAL

The stretchable gold film electrodes were deposited on a PDMS substrate with about 100 μm thickness, which was prepared by spinning Dow Corning Sylgard 184 mixed in a 10:1 ratio with 600 rpm for 60 s on a silicon wafer substrate. After curing at 80 $^{\circ}\text{C}$ for 3 h and after plasma treatment, 8 mm long, 0.5 mm wide gold stripes with two $1.5 \times 1.5 \text{ mm}^2$ contact pads were defined by a stainless steel mask. The gold stripes were deposited of a chromium adhesion layer with about 1 nm thickness followed by a gold layer with around 30 nm thickness by direct current magnetic sputtering. The electrode was stretched in the direction of a longer dimension of

the gold stripe using a custom-designed stretcher and also a mechanical testing machine equipped with a resistance meter (AG-X Plus 100N, Shimadzu, Japan). The morphologies of the electrodes were characterized by a scanning electron microscope (SEM, NOVA NanoSEM 450, FEI), an optical microscope (NIKON eclipse 80i), and an atomic force microscope (AFM, BRUKER FastScan). The constant current density was the output between the two contact pads when the electrodes were electrified. During the thermal treatments, the electrodes were placed on a silicon wafer, which was then heated on a hot plate.

III. RESULTS AND DISCUSSIONS

A. Electromechanical performance

The electrodes are stretched in the direction of the longer dimension of the gold stripe and a sample is displayed in the inset of Fig. 1(a). The electrical resistance of the electrodes as a function of the applied uniaxial strain is shown in Fig. 1(a). The resistance increases along with the strain until it reaches the critical strain, i.e., the maximum strain at which the electrode maintains conductivity. The resistance out of the measuring range (beyond 40 M Ω) indicates a functional rupture of the electrode, which will recover after the strain is released. It can be seen that the average critical strain of the electrodes is about 120%. The resistance of the electrode before and after being stretched to 100% strain is about 500 and 50 000 Ω , respectively, so the surface resistivity can be estimated as 31 and 3100 Ω/sq , respectively.

Three combined features enable the reversible stretchability of thin gold films on PDMS: first, the elastic substrates are extremely compliant, and wrinkles will appear decreasing the stress concentration while the electrode is being stretched; second, initial nanocracks have the mean length of $90 \pm 10 \text{ nm}$ ($n = 100$) distributing with random orientations shown in Fig. 2(a), which are capable of expanding randomly into the microcracks, releasing strain energy

FIG. 1. Electromechanical performance of the stretchable electrodes. (a) Electrical resistance of the stretchable electrodes as a function of the applied uniaxial strain, where the value of O.R. represents that the resistance is beyond the measurement range, indicating a functional fracture. Inset: an optical image of an electrode sample. The black double arrow line shows the direction of the uniaxial stretching. (b) The critical strains of the original electrodes and the ones electrified or heated on a hot plate as a function of the duration. The electrodes electrified with $0.0 \times 10^5 \text{ A/cm}^2$ refer to the original ones. Electrodes were electrified with current densities of $0.8 \times 10^5 \text{ A/cm}^2$, $3.0 \times 10^5 \text{ A/cm}^2$, and $4.0 \times 10^5 \text{ A/cm}^2$ for 0 min, 1 s + 20 min, and 1 s + 60 min. The asterisks in the figure indicate the diversity factor of the critical strains between the treated electrodes and the original ones.

FIG. 2. SEM images of the original and treated electrodes. (a) An SEM image of the original electrode with initial nanocracks of about 90 nm in length. (b) and (c) SEM images showing the electrodes after being heated on the hot plate at 80 °C and 200 °C for 20 min. (d) and (e) SEM images showing the electrodes electrified with $0.8 \times 10^5 \text{ A/cm}^2$ and $3.0 \times 10^5 \text{ A/cm}^2$ for 1 s + 20 min. The nanocracks in (b) and (d) are not significantly different from that in (a), while the widths of the nanocracks in (c) and (e) are remarkably wider than that in (a). (f) An SEM image showing the failure area of the electrode electrified with $4.0 \times 10^5 \text{ A/cm}^2$. There are many small gold particles dispersing around the gully.

while being stretched; third, the nanocrack pattern in the gold film allows the film to elongate by deflecting and twisting out of plane, so that a large applied elongation only induces a small elastic strain in the film. Furthermore, since the properties and performance of the electrodes strongly depend on the fabrication process, the gold film can show three different morphologies with different gold film thicknesses, namely, smooth, microcracked, and buckled.²⁵ Therefore, the electrodes with different gold film thickness are fabricated and studied in this work. When the gold film is thinner than 30 nm, the stretchability does not improve but the adhesion between the gold film and polymer substrate decreases. When the gold films are much thinner, the gold films become discontinuous and the conductivity deteriorates. When the gold film is thicker than 30 nm, the stretchability of the electrodes decreases with gradually increasing thickness, until vanishing completely when the micro/nanocrack morphology cannot form. Therefore, we have optimized the fabrication process and the gold film thickness to be 30 nm on the basis of performance and film adhesion (sample robustness).

The critical strains, or termed as the stretchability, of the electrodes will change after the electrification, depending on the applied current density. In order to determine if there is an instantaneous effect at the nanocracks of the gold film, the electrodes are electrified for 1 s first. From Fig. 1(b), it can be seen that the critical strain barely changes after the instantaneous power (1 s) whether with a low current density of $0.8 \times 10^5 \text{ A/cm}^2$ (LCD) or a median current density of $3 \times 10^5 \text{ A/cm}^2$ (MCD), indicating that there is no instantaneous effect on the gold film surface. Meanwhile, the average critical strain of the original electrodes is 120% and drops slightly to 110% when the electrodes are electrified with LCD for 20 min. The value barely changes when the electrodes are electrified for another 40 min, which implies that the electrification with LCD has little effect on the critical strain of the electrodes. However, the critical strain reduces markedly, decreasing to 80% when the electrodes are electrified with MCD for 20 min. Furthermore, the critical strain falls to zero just at the moment of the electrification with the high current density of $4 \times 10^5 \text{ A/cm}^2$ (HCD), representing that the electrodes rupture functionally. In addition, the critical strains are almost the

same for the electrodes electrified for 20 min and 60 min whether with LCD or MCD. This indicates that the microstructures of the electrodes become stable after electrification for a period of time and will no longer be affected by the increase of the duration.

To study the thermal effects on the stretchable gold film electrodes compared to the electrical effects, the electrodes are heated on a hot plate at a low temperature (80 °C) and a high temperature (200 °C) for 20 min. The temperatures are chosen because the curing temperature of PDMS is 80 °C, and the application temperature of PDMS 184 is not higher than 200 °C. Then, the critical strains of the electrodes after thermal treatments are measured. As shown in Fig. 1(b), the average critical strain of the electrodes heated at 80 °C for 20 min decreases from 120% to 100%, while the value falls to 65% when the electrodes are heated at 200 °C for 20 min. The results are similar to the situation of the electrodes with electrical treatments, which indicates that the thermal mismatch stress plays a key role in affecting the performance of the stretchable gold film electrodes whether in the electrical or thermal treatment process. The stress produced in the electrification with LCD or heating at 80 °C is relatively small so that the critical strain of the electrodes decreases slightly, whereas the critical strain of the electrodes electrified with MCD or heated at 200 °C decreases significantly due to the higher thermal mismatch leading to larger and wider cracks. When the gold film reaches thermal equilibrium in the electrical or thermal treatments after a period of time, the microstructure of the film becomes stable and the critical strain of the electrodes is no longer affected by the increase of the duration.

Moreover, the stretchable gold film electrode is stable and can still work even after 1000 stretching cycles tested by a mechanical testing machine, which is custom-designed for the flexible and stretchable materials. The resistance of the electrodes as a function of the applied strain shows a hysteresis nature, and the resistance decreases with increasing the cycle number and tends to stabilize after extensive cycles. After the electrical and thermal treatments, the microcrack microstructure retains in the gold film electrode, and only the cracks grow and widen to some extent, so the electrodes still work stably.

B. Microstructure characterization

The surfaces of the electrified and heated electrodes are observed by SEM, as shown in Fig. 2. The microstructures of the electrodes heated at 80 °C or electrified with LCD shown in Figs. 2(b) and 2(d) are not significantly different from that of the original ones shown in Fig. 2(a), while the surface morphology of the electrodes heated at 200 °C or electrified with MCD in Figs. 2(c) and 2(e) changes obviously. The previous irregularly distributed nanocracks become wider significantly, and the average crack width of the treated electrodes is about twice as large as that of the original ones. When electrified with HCD, the gold film shows a penetrating gully perpendicular to the direction of the current, resulting in a breakage of the electrode shown in Fig. 2(f). In addition, there are many small gold particles dispersing around the gully, which is due to the electromigration (EM) phenomenon at the applied current density. The change in the microstructure of the electrodes electrified with MCD is ascribed to the EM phenomenon and the thermal mismatch stress resulted from the heat

generated in the process of electrification, during which the gold film cannot detach from the substrate due to the adhesion between the gold film and the PDMS substrate, so the nanocracks in the gold film propagate and widen in order to release the stress. Once the stress is beyond the yield point, the nanocracks cannot recover to the original state after the power-off, resulting in wider cracks. Meanwhile, the crack tips become smoothened by the EM phenomenon. The smoothened crack tips and wider cracks deteriorate the ability of electrodes to release stress. Therefore, the electrodes are more prone to fracture when stretched, i.e., the stretchability decreases.

C. Stress analysis

The stretchable electrodes are composite materials, made of a metal film and elastic polymer substrate, which are two materials with entirely different physical properties. Take the gold as an example; its Young's modulus is about 82 GPa,²⁶ while that of PDMS is around 1 MPa.²⁷ Besides, the thermal expansion coefficient of the PDMS is about 69 times larger than that of bulk gold ($\alpha_{Au} = 14 \times 10^{-6}/K$, $\alpha_{PDMS} = 960 \times 10^{-6}/K$).¹¹ It is worth mentioning that the property of a thin gold film is different from that of bulk gold. Here, we take the value of bulk gold for estimation. The thermal mismatch stress can be estimated as

$$\sigma = \Delta T \cdot \Delta \alpha \cdot E. \quad (1)$$

Take the parameters above into the equation and the temperature difference is hypothesized as 50 °C (the difference between the room temperature and that when the gold films are being deposited), then the stress is calculated as 3.9 GPa, which is so large that the energy release rate motivates the nanocracks in the stretchable gold film propagate. Electrical treatments can generate heat in a local area through the resistance and cause the thermal mismatch stress similar to the thermal effects.

The decrease in the stretchability of the electrodes is due to the crack propagation and widening after the electrification; however, the wrinkle morphology of the gold film appears after the electrification and causes the thermal mismatch stress to redistribute. Hence, it is necessary to assess the stress redistribution of the wrinkled surface. As discussed above, the thermal mismatch stress σ of about 3.9 GPa is taken, with the temperature change ΔT as 50 °C from Eq. (1). For a flat surface, the stress can be assumed to be biaxial, i.e., $\sigma_{xx} = \sigma_{yy} = \sigma$ and $\sigma_{zz} = 0$. However, for a wrinkled surface, the component σ_{zz} normal to the gold film surface is no longer zero and the in-plane components σ_{xx} and σ_{yy} vary from place to place depending on the geometry of film. For a cylindrically symmetric case, an analytical solution for the thermal mismatch stress is obtained as²⁸

$$\sigma_r(r=R) = -\frac{E^g(\alpha^p - \alpha^g)\Delta T \left[\left(\frac{R+H}{R} \right)^2 - 1 \right]}{1 - 2\nu^g + \left(\frac{R+H}{R} \right)^2 + \frac{E^g}{E^p} \left(\frac{1-\nu^p}{1+\nu^g} \right) \left[\left(\frac{R+H}{R} \right)^2 - 1 \right]}, \quad (2)$$

$$\sigma_\phi(r=R) = \frac{E^g(\alpha^p - \alpha^g)\Delta T \left[1 - \left(\frac{R-H}{R} \right)^2 \right]}{1 - 2\nu^g + \left(\frac{R-H}{R} \right)^2 + \frac{E^g}{E^p} \left(\frac{1+\nu^p}{1+\nu^g} \right) \left[1 - \left(\frac{R-H}{R} \right)^2 \right]}, \quad (3)$$

where H is the gold film thickness, R is the radius of the wrinkled

FIG. 3. The calculation of stress redistribution on a wrinkled surface. (a) The schematic and definition of the stresses on a wrinkled surface based on a cylindrical model. (b) The normalized tangential stress σ_{xx} , σ_{yy} , and normal stress σ_{zz} and the average stress σ as a function of the radius and gold film thickness from the analytical solution.

surface, α is the thermal expansion coefficient, ν is the Poisson ratio, and ΔT is the temperature change. The superscripts P and g stand for the PDMS and gold film, respectively. From Eqs. (2) and (3), the redistribution of the thermal mismatch stress can be estimated. The schematic illustration and calculation of the stresses are summarized in Fig. 3(a). Normalized tangential stress σ_{xx} , σ_{yy} , and normal stress σ_{zz} as a function of the radius and gold film thickness ratio are shown in Fig. 3(b), where σ_0 is the stress at a flat surface, σ_{zz} decreases but σ_{xx} increases with increasing radius. The average stress $\sigma = (\sigma_{xx} + \sigma_{yy} + \sigma_{zz})/3$ follows the same trend as the tangential stress; hence, it increases with increasing radius, as shown in Fig. 3(b). The gold film thickness is about 30 nm. The wavelength of the wrinkle is round $2\ \mu\text{m}$ measured by the SEM and optical microscopy images. The height of the peak estimated from AFM measurement is about $1\ \mu\text{m}$, so the radius is

in the magnitude of $1\ \mu\text{m}$. The radius and gold film thickness ratio is calculated as 33. Taking these parameters into Eqs. (2) and (3), it is obtained that $\sigma = 0.5\sigma_0 = 1.9\ \text{GPa}$. With this wrinkled morphology surface, although the thermal mismatch stress is released to some extent, the energy release rate is still large enough to motivate the crack growth, leading to the crack propagation and widening.

D. Crack quantification

The cracks propagate and widen by the thermal mismatch stress and the extent depends on the current density of electrification or thermal treatments. The quantification of the cracks is shown in Fig. 4. First, the high resolution images of the nano-cracks are taken by SEM, followed by image analysis to extract

FIG. 4. The quantification of the crack density in the original and treated electrodes. (a) An SEM image of the electrodes electrified with $4.0 \times 10^5\ \text{A/cm}^2$ for 20 min. (b) The quantification of the crack percentage of image (a) by the image analysis method. (c) The crack area percentage of the original and treated electrodes vs current density and temperature. (d) The crack width and crack length estimated by the division between the crack area and width as a function of the current density.

the cracks from the images with the binarization method, as shown in Figs. 4(a) and 4(b). Unsurprisingly, the crack percentage increases with increasing current density and temperature, as shown in Fig. 4(c). It is found that the crack percentage of the samples treated with LCD and MCD is slightly smaller than that of the samples heated at 80 °C and 200 °C, respectively, which is corresponding to the stretchability shown in Fig. 1(b). There is no obvious increase in the crack length with increasing current density estimated by the division between the crack area and the crack width, as shown in Fig. 4(d). The cracks play a much more important role in affecting the stretchability than the wrinkled morphology. After the electrification, the cracks propagate and widen, accompanied with the occurrence of wrinkles. The propagation and widening of the cracks deteriorate the stretchability despite of the benefits of stress release from the wrinkled morphology.

E. Further discussion and interesting phenomenon

It is reported that electromigration (EM) can happen in the thin film when the film is electrified at the current density in the range of 10^4 – 10^6 A/cm².²⁹ The current density in this work is in the range and there are signs of the EM phenomenon shown in the electrodes after the electrification. The tips of the cracks become smoothened by the EM phenomenon, which decreases the ability of the cracks to propagate and release the stress during stretching shown clearly in Fig. 4(a). The crack widening caused by the thermal mismatch stress and crack tip smoothing by the EM deteriorates the stretchability of the electrodes

electrified with LCD or MCD. For the HCD situation, the penetrating crack causing the fracture of the electrodes may initiate at the area with high local resistance, where great heat is generated. To confirm this hypothesis, a point of the gold strip is chosen and a tiny area at the edge is scratched to make its local resistance higher, and then the electrode is electrified with HCD. A video shown in Figs. 5(a) and 5(b) (Multimedia view) is taken with an optical microscope, and the edge of the photographed area has been artificially scratched earlier. The video is taken at the moment of power-on when the electrode is electrified with the HCD. Figures 5(a) and 5(b) (Multimedia view) show the status of a location before and after the electrification, respectively, and a penetrating crack perpendicular to the direction of the current is clearly shown in the location after the electrification. It is certain that the experimental results are in good reproducibility. The results indicate that the thermal effect is generated during the electrification and causes the thermal mismatch stress in the electrodes. The gold film having a much smaller thermal expansion coefficient than the PDMS substrate is under a large thermal mismatch stress as analyzed in details above and the cracks propagate and coalesce, leading to a fracture. Besides, the crack propagation occurs easily in the area having the high local resistance due to the heat generated through the electrification.

There is an interesting phenomenon. The wrinkles will appear immediately on the power-off of electrification and disappear immediately on the power-on, as shown in Fig. 5(c) with video illustration (Multimedia view). The wrinkle phenomenon is reversible with the electrification process; however, there is no such phenomenon during the thermal treatment process. No wrinkles appear during

FIG. 5. Optical micrographs of the original and treated electrodes. Optical micrographs showing the electrode before (a) and after (b) the electrification with a current density of 4.0×10^5 A/cm². After the electrification, there is a penetrating gully perpendicular to the direction of the current appearing on the surface, and there are many wrinkles appearing parallel to the direction of the current. (c) An optical micrograph showing the surface of the electrode after the electrification with a current density of 3.0×10^5 A/cm², the wrinkles appear immediately on the power-off of electrification and disappear instantaneously on the power-on of electrification. (d) An optical micrograph showing the surface of the electrode after being heated at 200 °C for 20 min. There are no wrinkles on the surface. Multimedia views: <https://doi.org/10.1063/1.5079435.1>; <https://doi.org/10.1063/1.5079435.2>; <https://doi.org/10.1063/1.5079435.3>

cooling, as shown in Fig. 5(d). The main reason for the distinction between the two processes is the difference of heating source and resultant thermal stress. The heating source during the electrification is the fragile gold film, which transfers the heat to a partial PDMS substrate just below the gold film, and the distribution of the thermal field in the gold film is anisotropic, so the wrinkles appear after the power-off. On the contrary, the heating source in the process of heating is the hot plate, which transfers the heat to the whole PDMS substrate and then to the gold film; as a result, the distribution of the thermal field in the gold film is isotropic and there are no wrinkles appearing after heating.

IV. CONCLUSIONS

In summary, this study investigates the electromechanical performance of the stretchable thin gold film on an elastic substrate with electrical and thermal treatments. The stretchability of the stretchable gold film electrodes is slightly impaired, decreasing from 120% to 110% when the electrodes are electrified with a current density of $0.8 \times 10^5 \text{ A/cm}^2$, while it reduces significantly to 80% when the electrodes are electrified with a current density of $3 \times 10^5 \text{ A/cm}^2$. Furthermore, the electrodes functionally rupture, i.e., losing the conductivity just at the moment when the applied current density is beyond a critical value of $4 \times 10^5 \text{ A/cm}^2$ in this study. The thermal treatments have similar effects compared with the electrification process. The stretchability of the electrodes heated at 80°C and 200°C for 20 min drops to 100% and 65%, respectively. The decrease of the stretchability is ascribed to both the crack propagation and widening by thermal mismatch stress and crack tip smoothening by the electromigration phenomenon, reducing the ability of the cracks to release stress during stretching. Reversible wrinkles appear and disappear during the electrification process but not the thermal treatment process, which is due to the different heating source resulting in a different stress distribution in the electrodes. Stretchable electronics with reliable electromechanical performance is necessary for its application in bioelectrical interfaces. The work provides suitable working conditions of applied current density and temperature for the stretchable gold film electrodes and some perspectives for the design of other stretchable electronics.

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