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ABSTRACT
Wrinkles, as a form of mechanical instability, can be employed in many different areas, including self-cleaning coating, flexible electronics, smart adhesion, channel for microfluidic devices, and mechanical property measurements. After two decades of research, the wavelength and amplitude of wrinkles can be well controlled by proper design of physical properties of thin films and substrates. However, it is still very difficult to change their orientations and complexity in a single piece of thin film, which is critically important for many practical applications. In previous studies, the orientation of all the wrinkles are either along the same direction or can only be controlled to some extent by the patterning of relief structures which rely on the complicated lithography techniques and cannot be adjusted for a given substrate. Here, we employed the shape memory polymer (SMP) as a smart substrate, combined with local/selective heating to control the boundary conditions for the strain field, and realized the gradual change in the orientation of wrinkles in a single piece of thin film. The wrinkles with a gradual change in orientations exhibited angle-dependent colors. Furthermore, by changing the sequence of thin film coating and partial triggering of an SMP, complex surface features with a sharp interface can be obtained. Finite element simulation investigations uncover the basic principles and requirements that need to be satisfied, as well as the limitations of our method, to generate wrinkles with controlled orientations in a single piece of thin film.

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I. INTRODUCTION

Wrinkles are ubiquitous in nature as a form of mechanical instability, occurring in vast different systems ranging from animal skin to lava flow.1–5 They were previously treated as a nuisance to be avoided, but in recent years, wrinkles have been taken advantage as exquisite patterns for advanced applications.3,5 For example, wrinkles can be employed to measure the properties of a thin film, enhance light extraction, separate small particles, endow brittle thin films with stretchability, and act as the channels for the solution flow in microfluidic devices, just to name a few.6–9 From the perspective of mechanics, wrinkling is essentially a type of structure instability, and wrinkles can be formed in a piece of thin film coated on prestretched rubber or a shape memory polymer (SMP) substrate. Upon releasing the prestrain or heating to trigger substrate shrinkage, compressive stress generated by the substrate buckles the coated thin film and results in features of wrinkles.10–15 As a periodical pattern, the main focus was on geometrical features (GFs) of wavelength and amplitude in previous studies [Fig. 1(a)]. Both of these two features are relatively easy to manipulate, since they are dependent on the prestrain, modulus of... 

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FIG. 1. (a) Schematic of characteristic geometrical features (amplitude, wavelength, and orientation) of wrinkles; (b) locally distributed wrinkles stamped out of shape memory polymers (b); (c)–(f) Buckling process of the 1/4 FE model in periphery heating case. The directions of the principal stresses (compressive stress) are indicated by arrows in the bottom of each image. The SMP substrate recovery strain = 0.90% (c), 1.65% (d), 2.15% (e), and final stage (f); (g)–(k) schematic of rigid boundary effects; (g) fundamental case, where straining along the boundary is restrained; (h) periphery recovery, in which additional compressive strains are introduced; (i) inner recovery with an open straight boundary, featuring no additional strains at a boundary; (j) inner recovery with a curved boundary, featuring additional shear strain at a boundary; (k) inner recovery with a closed boundary, with extra tensile strain.
the thin film, and the substrate, all of which can be properly controlled. However, the study of the orientation, which is another very important parameter for wrinkles, is still very limited. Indeed, for conventional strategy, the prestrain is released uniformly and completely, and thus the wrinkles are distributed across the whole thin film with an identical orientation. Developing a new strategy, by which the wrinkles can be patterned at any location as desired and their orientation can be controlled or changed at will in a single piece of thin film, is very important for practical applications, such as structure colors, microfluidic devices, and templates for nanowire growth.

As a substrate for the formation of surface features, an SMP has a number of advantages compared to prestretched rubber, such that it can be thermally triggered to shrink and, thus, avoid the complicated substrate prestretching process. The viscoelastic properties of an SMP also provide a new degree of freedom to control the GFs of wrinkles. However, the most distinguishing characteristic of an SMP is that it can be triggered locally, which has not been employed yet to form complex wrinkles as far as we know. In this study, we focus on the patterning and orientation control of the wrinkles by heating an SMP substrate locally. By properly designating the boundary between heated and unheated zones, shear stress can be introduced to change the direction of principal stress (PS), which is the key factor to determine the orientation of wrinkles. Instead of being distributed all over the thin film, wrinkles were only formed at locations that were heated. More interestingly, these wrinkles are highly parallel with the boundary, as proved both theoretically and experimentally. We further show that different sequences of SMP recovery and thin film deposition can even result in complex surface features with a sharp boundary. As a proof of concept, we illustrated that the wrinkles with a controlled orientation can be used as angle-dependent structure colors as well as angle-dependent flows. Basic principles and requirements that need to be satisfied, as well as the limitations of our method, to generate wrinkles with controlled orientations in a single piece of thin film are also investigated by a finite element method (FEM).

II. EXPERIMENTAL DETAILS

A. Deposition of a PEDOT thin film

To prepare PEDOT thin films on the SMP substrate, PEDOT dispersion with PSS [poly(3,4-ethylenedioxythiophene) polystyrene sulfonate] was first prepared. The solution was then deposited on the SMP substrate (Shrinky Dinks sheet), preheated to ~100 °C, by spray coating. This temperature was chosen to be high enough to quickly vaporize the water in the solution to enhance the uniformity of the thin film, yet not higher than the Tg of the SMP substrate to prevent its shape recovery. The above process was also employed to fabricate a thin film based on carbon nanotube (CNT) and graphene oxide (GO). The carbon nanotube (CNT) dispersion (0.9 wt. %) was prepared by tip-sonication at low power (48 W) for 7.5 h with the assistance of a surfactant (sodium dodecyl sulfate, 1.0 wt. %). Graphene oxide (GO) was synthesized by a modified Hummers method, as reported by Huang and Suo. After thorough purification by dialysis for two weeks, the obtained GO solution was sonicated by tip-sonication at a power of 120 W for 30 min to ensure homogenous dispersion. There is no sedimentation after 30 min of centrifugation. For copper deposition, a magnetron sputtering system was used. The thickness of copper can be controlled by the power and deposition duration, and the typical thickness used in our study is about 300 nm.

B. Pattern the wrinkles with the designed orientation

A stamp with protruded patterns, as shown in Fig. 1, was heated to ~180 °C, which is higher than the Tg of the SMP substrate. The preheated stamp was then manually pressed onto the thin film deposited on an SMP for 1 min. The patterns and orientations of the generated wrinkles were observed and studied by SEM.

C. FEM simulation

To study the underlying mechanism of wrinkle patterning, finite element (FE) models were developed using the commercial software package ABAQUS. Corresponding to specimens in the experimental session, the FE model consists of a square SMP substrate and a thin film coated on it, as shown in Fig. S1(a) in the supplementary material. Uniform heating and localized heating were simulated. Taking the localized heating case, for example, the inner 4 × 4 mm² unheated block of the substrate was modeled as a rigid part, and the surrounding portion was assigned elastic material properties of E = 10 MPa and v = 0.49. This very low Young’s modulus simulates the softened texture of the surrounding portion of an SMP at high temperature. In modeling the SMP contraction, a variation in the temperature field was defined in the heated portions, which resulted in the shrinkage of the material. The substrate was restrained over its bottom surface against out-of-plane deflection (i.e., only in-plane deformations of the substrate were allowed). The thin coating film was modeled with a layer of isotropic shell with elastic material properties of E = 1000 MPa and v = 0.3. The thickness was taken as 0.001 mm in this example. A layer of cohesive elements (of zero thickness) were used to model the bonding effect at the interface between the coating film and the substrate. Note that the thin coating film was actually deposited by directly spraying the PEDOT solution onto the SMP substrate. The coating film and the substrate were, therefore, bonded by the adhesive effect at their interface, without using an extra adhesive layer. Constitutive properties of the cohesive elements are E = 50 MPa and G1 = G2 = 30 MPa. Delamination of the cohesive layer is modeled with initiation criteria of maximum nominal stress and linear damage evolution criteria based on fracture energy. The maximum stresses for delamination initiation were taken as 50 MPa in all three principal directions, and the fracture energy was taken as 100 ml/m². Geometric nonlinearity was accounted for in all analysis.

III. RESULTS AND DISCUSSION

Micromechanical modeling using FEM was carried out first to simulate the formation of wrinkles, seeking for some basic rules for their orientation and distribution control. Two scenarios were simulated, namely, periphery heating and uniform heating [Fig. S1(b) in the supplementary material]. According to the symmetric
geometry of the specimen, 1/4 model was used as shown in Fig. S1(c) in the supplementary material.

In the case of the periphery heating model, the analysis terminated at a strain of about 2.15%. The out-of-plane buckling deformation of the thin film is shown in Figs. 1(c)–1(f). It can be seen that when the SMP recovery strain is small [Fig. 1(c)], the strain across the whole thin film varies dramatically. However, at a smaller scale [shown in the middle row images in Fig. 1(c)], the strain is quite uniform. The principal stresses (PSs) in both x and y directions are well aligned at all locations. Zone 1 has its PS along the y direction remarkably higher than that along the x direction. Such a gap between the x-axis and y-axis PSs are gradually alleviated for the zones further away from the boundary (zones 2–5). As the SMP recovery strain increased [Fig. 1(e)], the PS distributions in zones 2–4 became misaligned, viz., the originally parallel principal stresses are disturbed. Interestingly, round- or square-shaped pits are observed in this stage. This indicates the emergence of local crumpled, as a result of interference of buckling in two orthogonal directions. When the SMP substrate is recovered further [Fig. 1(f)], the strains in thin film are more concentrated locally at some isolated points in zones 2–4 and the two PSs are more severely disturbed. This can be explained by the fact that the formation of crumpled at those discrete positions releases the stored elastic energy in the film around them. Note that during the process of SMP recovery, the PSs in zone 1 remain well aligned, with the PS in the y direction always negligible compared to that in the x direction. This observation agrees with the experimental phenomenon of well aligned wrinkles, rather than crumpled in zone 1. The stable mode of deformation and always aligned PS in zone 5 is mainly due to its relatively small strain, which is lower than the critical buckling strain. More interestingly, at the corner of the inner boundary, the orientation of wrinkles has to be reconciled. This would compel a change of wrinkle orientations. As shown in Fig. S6 in the supplementary material, the wrinkles close to the corner can indeed adjust their orientations gradually without any significant discontinuity. Remarkably, even for the wrinkles at the corner but far away from the boundary, they are still capable of altering the orientations to some degree. These experimental results clearly demonstrated that the orientation of wrinkles can be controlled by boundary conditions, in single piece of thin film.

In the case of the uniform heating model, the analysis terminated at a strain of about 1.8%. Buckling process of the thin film is demonstrated in Fig. S2 in the supplementary material. Minor crumpling deformations instead of wrinkling are formed across the whole thin film (Fig. S2 in the supplementary material). Interestingly, for a given contraction strain of the substrate, relatively small buckling deformation is observed for the case of uniform buckling than that of periphery heating. This phenomenon is attributed to a possible counteracting effect of buckling deformations in the two mutually orthogonal directions. More specifically, the buckling deformation developed in one direction turns the flat plate into a corrugated profile, which increases the bending stiffness considerably, thereby suppressing buckling deformation in the orthogonal direction. When compressive stresses in the two directions are comparable, the counteracting effect might postpone but cannot prevent buckling in the two mutually orthogonal directions. Crumpled will still emerge in the final stage. On the other hand, when compressive stress in one direction dominates as in zone 1 of the periphery heating case (see Fig. 1), the buckling deformation tends to prevail in one direction and restrains buckling in the other direction, rendering a roughly aligned wrinkle pattern (see Fig. S6 in the supplementary material). This finding indicates that when the compressive strain in one direction is dominantly higher than the other, oriented wrinkles can still be expected.

The effects of boundary conditions on the stress/strain distribution are further investigated by analyzing several typical cases. An elementary case is shown in Fig. 1(g) where the whole SMP is heated with a rigid boundary line applied. In this case, the material at the vicinity of the rigid boundary is restrained from deforming in the direction along the boundary line. This effect gradually vanishes with the distance from the boundary, and the deformation pattern changes from “unidirectional shrink” at the rigid boundary to “isotropic shrink” at far locations. Based on this elementary case, more complicated cases where only part of the SMP substrate is heated can be well understood as shown in Figs. 1(h)–1(k). Figure 1(h) shows a situation where not only the strains parallel with the boundary were constrained but also the deformation compatibility and continuity requirements along the x and y directions tend to “squeeze” the inner part of the recovering material. Therefore, additional compressive strains are introduced in the direction normal to the inner boundary, which results in better aligned wrinkles along the boundary. Figure 1(i) shows the case where the heated region can be recovered in the horizontal direction with a rigid boundary normal to the recovering direction. Clearly, the morphology of the wrinkles in the middle part of the heated region is dependent on its width. Larger width will result in crumpled, while smaller width results in aligned wrinkles. Figure 1(j) shows the case where shear stress, in addition to compressive stress, is generated. This is mainly because of the nonperpendicularly with the recovering direction (horizontal) and the boundary line (inclined). Based on the theory of continuum mechanics, such shear stress can change the direction of principal stress/strain. In other words, shear stress could be introduced and controlled by proper design of boundary conditions for locally heated SMP and, eventually, realize the orientation control of the wrinkles. Figure 1(k) shows a case where the closed rigid boundary tends to “stretch” the heated SMP material, and no shrinking is allowed at all. This effect tends to prevent wrinkling in the coated film, and therefore is an undesirable case.

Keeping those simulation results in mind, we designed experiments to investigate our strategy to pattern wrinkles with controlled orientation in a single piece of thin film. The SMP used in this study is a polystyrene “shrink film.” Dynamic mechanical analyzer (DMA) results show that its recovery temperature is in the range of 100–110 °C, and the recovery ratio is ∼1/3 (Fig. S3 in the supplementary material). A thin film (~500 nm) of a rigid polymer, PEDOT, is deposited on the surface of the SMP via spraying. For the formation of local wrinkles, stamps with different patterns are preheated to ∼150 °C, after which the stamps are brought to contact with the thin film of PEDOT samples coated on the SMP with a load of ∼10 N for 30 s.

As a control case shown in Figs. 2(a)–2(c), when the whole SMP is uniformly heated above its recovery temperature, the coated thin film is under isotropic in-plane compressive stress in all directions and thus be buckled to form crumple features. On the other
hand, when only the periphery of the sample is heated as shown in Figs. 2(d)–2(f), the wrinkles form only in the areas that are heated. More importantly, the wrinkles close to the boundary between heated and unheated regions have a well-defined orientation, that is, aligning parallel to the boundary. However, such orientation is dependent on the distance from the boundary. The wrinkles lost their orientation ordering (parallel with the boundary) gradually when moving away from the boundary and eventually turn into crumple features [Fig. 2(h)]. Those experimental observations are consistent with the predictions obtained via FEM simulations. As shown in Fig. S3 in the supplementary material, the modulus of the SMP decreases by two orders of magnitude when heated above to its glass transition temperature. Therefore, the unheated zones will act as rigid boundaries for the heated zones to recover, which perfectly satisfies the assumption we made in the FEM simulation. Only the perpendicular direction to the boundary is free to develop significant strain and thus can strongly order the wrinkles. Such constrains exerted by the rigid unheated zone become weaker for the areas further away from the boundary and thus allows both directions (perpendicular and parallel with the boundary) to develop wrinkles, and as a consequence, crumple features. In order to quantify the continuity and ordering of the wrinkles, two dimensionless parameters, continuity index (CI) and ordering index (OI), are defined (Fig. S4 in the supplementary material). CI is the ratio of the length of the continuous ridge between adjacent branch point [Fig. S4(a) in the supplementary material] to the average wavelength (≈10 μm) of the wrinkles, whereas OI is the angle between the orientation direction of the wrinkle and the boundary (Fig. S4 in the supplementary material). For the case of periphery recovery, Fig. 2(e) shows that OI for the wrinkles close to the boundary is very small (less than 5), indicating the uniform parallel orientation of those wrinkles to the boundary. The OI increases gradually with the distance to the boundary, consistent with the qualitative description given above as well as the FEM results. Same as the OI, the CI is also dependent on the distance from the boundary, and the further away from the boundary, the smaller the value of CI [Fig. 2(f)]. The average length of continuous ridges close to the boundary is as large as ≈400 μm. The defects of the branch point that terminate the continuity of the ridges are indicated in Fig. S5(a) in the supplementary material, which are probably the result of the local defects of the thin film. It is pointed out that the fracture of the ridges of the wrinkles is rarely observed [Fig. S5(b) in the supplementary material], which suggests the structural integrity of the PEDOT thin film. Because of the intrinsic singularity characteristic of the wrinkles at the corner (the OI is defined based on tangent line, and there is no tangent line for singularity point), both the OI and CI for the areas that are near to the corner fluctuated much more significantly than those far away from the corner (Fig. S6 in the supplementary material). Obviously, the capability of our method to control the orientation of the wrinkles is limited by the slope of the boundary line. For example, upon further increasing the abruptness of the corner...
(using a “V” shaped stamp, as shown in Fig. S7 in the supplementary material), the oriented wrinkles at the two sides of the corner even lost connection with each other, and this is complete loss of continuity. Therefore, a singular point with a boundary orientation abrupt change of 90° is probably the limit for the continuity of wrinkles in our strategy. A third stamp, with "one-quarter of circle" bump, was designed to investigate the control of the orientation of wrinkles [Figs. 2(g)–2(i)]. It can be seen that the wrinkles are perfectly aligned with the boundary, even for the part that is far away from the boundary. After carefully checking the orientation of the wrinkles from bottom-left to top-right, we found that the wrinkles can gradually rotate its orientation to maintain their parallelism to the boundary in the whole heated region, which is also quantitatively proved by the small values of OI through the whole sample. This observation strongly proves the capability of stamping the SMP substrate to pattern the wrinkles as well as the orientation control.

Besides the strategy that is discussed above, in which the boundary condition is mainly played with, reversing the steps of heating and thin film coating can provide even more rich wrinkle features in a single piece of thin film. For example, before thin film coating, we heated the periphery zone of the SMP to eliminate its strain recovery capability that is perpendicular to the boundary, after which a thin film was coated on the sample, and then the whole sample was heated to trigger the complete recovery of the SMP. It can be seen in Fig. 3 that very different wrinkle features are developed in the central and periphery regions with a sharp interface. Instead of the parallelism of the wrinkles to the boundary in the periphery regions for the case as shown in Figs. 3(b) and 3(c), the wrinkles are oriented perpendicular to the boundary. This is because the strain recovery along the perpendicular direction has already been triggered and eliminated before the thin film coating, and only the parallel direction is still active that can generate compressive stress in the thin film during the second heating process. On the other hand, since the central zone of the thin film is subjected to isotropic compressive in-plane stress, crumples are formed accordingly. The alignment of wrinkles is also manifested by the Fourier transform image. The circle and highly elongated ellipse shown in the inset of Fig. 3(d) indicates the random (crumple) and aligned wrinkles in the left and right regions, respectively. Again, such complex wrinkles with patterned crumpling and oriented wrinkles generated in a single piece of thin film have never been reported, and combining SMP with local heating strategy might be able to provide a new way to synthesize multifunctional surfaces.

Other thin film materials, including carbon nanotube, graphene oxide, and copper, were also tested to investigate the material dependence of our strategy to pattern film with controlled orientation. As shown in Fig. S8 in the supplementary material, the orientation of the patterned wrinkles in the copper film has very similar features with that of PEDOT. For the case of carbon nanotube (CNT), the CNT thin film is still relatively smooth without the effective formation of wrinkles upon heating the substrate (Fig. S9 in the supplementary material). This is probably caused by the inferior interfacial bonding strength between the CNT film and the SMP substrate, and the strong integrity and mechanical properties of CNT thin film due to the entanglement of CNTs also prevent the formation of wrinkles. For the case of graphene oxide (GO), the compressive stress generated by the SMP substrate resulted in the fracture of GO film into small pieces, because of the weak integrity and low fracture strain of the GO film. Those experimental results, compared with that of the PEDOT thin film, indicate

![FIG. 3. The formation of complex surface features via the sequence of partial recovery–thin film deposition–secondary recovery. (a) Schematic of the process. (b)–(d) SEM images of the surface features, which consists of crumples and wrinkles with a sharp boundary. (d) Fourier transformation indicating the orientation of wrinkles.](image-url)
that the structural integrity, fracture resistance of the thin film, and interfacial bonding between the thin film and the SMP are the key properties to guarantee the formation of continuous patterned wrinkles with controlled orientation.

As proof of concept, we demonstrated several potential applications of the patterned wrinkles with controlled orientation (Fig. 4). Structure colors can be seen in various biological systems, such as butterflies, allowing them to change their appearance according to the environment. Structural color induced by wrinkles on an SMP substrate was previously reported by Xie et al., and they showed that the colors can be adjusted by the wavelength of the wrinkles. In contrast to their work, wrinkles with a controlled orientation developed in our work can further exhibit angle-dependent colors. Figures 4(a)–4(d) show that the optical reflections of the wrinkles [the samples as shown in Figs. 2(d)–2(f)] are sensitive to the polarizing angles, indicating a highly angle-dependent interaction between the regular patterns and optical waves.

IV. CONCLUSION

In summary, we reported a strategy to control the orientation of wrinkles in a single piece of thin film by stamping patterns on the SMP substrate. We control the alignment of wrinkles by the principal stress, and this can be adjusted by the shearing force, which was mainly regulated by boundary condition between heated and unheated zones. We further showed that changing the sequence of thin film coating and heating process can result in even more complex and interesting surface features. Wrinkles with a controlled orientation provide an opportunity to study new applications, such as structural colors and microfluidic devices. However, more precise and accurate control of the geometrical parameters, including wavelength, amplitude, and orientation of these wrinkles down to the nanoscale at the same time, is still very difficult. Further studies, in which materials optimization/design and nonlinear solid mechanics theory are combined, are conducted to push the limit of our strategy for the wrinkle orientation control.

SUPPLEMENTARY MATERIAL

The supplementary material includes some results of FEMs and SEM images of various wrinkle features.

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