# Substrate Thickness: An Effective Control Parameter for Polymer Thin Film Buckling on PDMS Substrates

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Received 26 August 2008; accepted 2 December 2008
DOI 10.1002/app.29824
Published online 23 February 2009 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** Buckling patterns of polymer thin films on plasma-treated poly(dimethylsiloxane) (PDMS) substrates were sensitively affected by the thickness of the substrate in addition to the substrate modulus. On highly crosslinked PDMS substrates, the buckling wavelength of polymer thin films sharply increased as the thickness of the substrates were raised and approached a plateau value when the substrate was 2.5 mm-thick. On weakly crosslinked PDMS substrates, the wavelength still increased even when the substrate was thicker than 20 mm. The high dependence of the buckling on the substrate thickness has not been reported before and is unexpected from the current predictions. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 112: 2683–2690, 2009

**Key words:** buckling; polymer thin films; polymer patterning

## INTRODUCTION

Compressive stress created thermally or mechanically in stiff thin films on soft substrates results in out-of-plane deflection of the thin films. Such buckling of thin films is frequently encountered in nature and has been long interested. For instance, the wrinkling of human skin having a stiff epidermis on top of a soft dermis takes place when the skin is deformed due to muscle contraction or outside mechanical deformation. The surface of many dried fruits and the formation of mountain ranges are also the buckling phenomena. Metal films on top of relatively flexible substrates readily delaminate or peel off in repeated bending process. In past years, the transverse deformation of thin films has been a subject to avoid because it has been considered as a failure on the surface.[1–3]

Recently, Whitesides and coworkers have used the spontaneous phenomenon as a novel nonconventional strategy to fabricate microstructure patterns.[4] Compressive stress was generated by heating and cooling the substrates, creating isotropic buckling of metal films. Silica-like layers on poly(dimethylsiloxane) (PDMS) substrates generated by exerting plasma or UVO has also been exploited to fabricate a buckled surface pattern.[5,6] The buckling of elastic films on a viscous layer[7–9] and solid films residing on top of a liquid base[10] has also been demonstrated. Recent researches took advantage of the buckling to measure the modulus of polymer films on a PDMS substrate.[11–15] Now the robust patterning method using the buckling has increasingly found a wide range of applications such as biocompatible topographic matrices for cell alignment,[16] tunable diffraction gratings,[17] flexible electronics and devices,[18–20] microfluidic sieves,[21] modern metrology methods,[11–15] and optical devices.[22]

Buckling has been mainly studied on a thick foundation made of PDMS. Most works reported to date have used a two layer system comprising a thin stiff layer made of either metal or a silicon oxide and PDMS substrates. For the use of the PDMS substrate, it was exposed to oxygen plasma or UVO to form an oxidized layer at the surface of the PDMS substrate. The thickness of the oxidized layer was varied by adjusting the exposure time or the plasma power.[23,24] Chua et al. investigated the effect of plasma power and treatment time on buckling pattern.[6] The wavelength of the buckling increased as the thickness of oxidized layer was raised. Bowen et al. prepared buckling patterns by depositing thin layers of gold on thick PDMS at high temperature and cooling down the specimen. The volume shrinkage during the cooling built up a compressive stress and induced the buckling.[4] Ohzono et al. deposited a thin layer of platinum onto hexagonally organized arrays of holes in PDMS and showed the directional order of buckling patterns when the periodicity of the substrate pattern matched the intrinsic wavelength of the buckling.[25] The inorganic thin layers on PDMS usually generated large-wavelength patterns in a range of a few tens of micrometers. Very recently, polymers were spin-coated on the surface-

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Contract grant sponsor: KOSEF (MOST); contract grant number: RO1-2007-001-11281-0 and R11-2007-02004-0.

treated PDMS substrate and decreased the buckling wavelength a micrometer scale.[26] A polymer/nanoparticle composite on a PDMS substrate was suggested to prevent the buckling in polymeric systems.

The buckling patterns on PDMS have been generally analyzed by including the oxidized layer in the top surface layer. Such approach successfully explained the dependence of the buckling patterns on the thickness and modulus of the thin films. However, most studies have neglected the dependence of buckling patterns on substrate parameters. This is because current theories predict the buckling wavelength should be almost independent on the substrate thickness although the wavelength is inversely proportional to the modulus of the substrates. In this work, for the first time we will demonstrate that the thickness of PDMS substrate is an effective parameter to control the buckling patterns of polymer thin films. Such large dependence on substrate thickness and modulus are not explained with current analytical calculations. The model prediction is left for future study.

## EXPERIMENTAL

### Materials

The Sylgard 184 elastomer kit from Dow Corning was used to make PDMS substrate. Polystyrene (PS, $M_w = 230{,}000$) was purchased from Aldrich.

### Procedure involved in the buckling of polymer thin films

Flat PDMS substrates were prepared by mixing the siloxane prepolymer and its corsslinker. The crosslinking density of the PDMS substrates was adjusted by varying the mixing ratio of the crosslinker to prepolymer (1 : 40, 1 : 20, 1 : 10, 1 : 5, 1 : 2 (w/w)). The mixtures were hosted in glass Petri dishes (10 cm in diameter). They were left at room temperature to allow trapped air bubbles to escape and then cured at 80°C for 24 h. The PDMS substrates were further cured at 170°C in vacuum for 30 min because any additional crosslinking in the PDMS substrates during the thermal annealing for buckling may produce additional stress in addition to thermal expansion mismatch. We found that the PDMS substrates with and without curing at 170°C produced the same buckling patterns, which indicates the curing is completed by annealing at 80°C for 24 h. The cured PDMS with different thicknesses (0.5 mm, 1 mm, 2 mm, 5 mm, 10 mm, 20 mm) were prepared in the same way. Small substrates with dimension of 1.5 cm × 1.5 cm were cut from the cured PDMS elastomers and placed on clean glass slides. The PDMS substrates were exposed to oxygen plasma (Cute-100LF, Femto Science) of 30 W with a flow rate of 20 sccm and at a base pressure of 0.923 Torr. Usual duration of oxygen plasma was 30 s, whereas longer exposure was applied to investigate the plasma effect on the buckling patterns. PS polymers was dissolved in toluene (1 wt %) and spin-coated on the PDMS substrates at 3000 rpm for 30 s. The polymer-coated PDMS substrates were placed in a preheated vacuum oven (170°C) for 30 min. The samples were removed from the oven and cooled at room temperature in air.

### Characterization

Buckling patterns were observed using an optical microscope (OM, Olympus BX51) and atomic force microscope (AFM, Nanoscope IV[a], Digital Instrument) in tapping mode. AFM images were obtained in the modes of height and phase contrast. The wavelength of the buckling patterns was measured by taking an average of peak-to-peak distance of 10 parallel waves. For PDMS substrates, the Young's moduli were determined using a tensile stress tester (TST 350, Linkam Scientific Instruments). PDMS sheets with dimension of 3 cm ×1 cm × 0.1 cm were clamped with binder clips. The gauge length was 1.5 cm. The specimens were strained with constant tensile speed of 100 μm/s using load cell of 200*N*. The slope in the linear region of the stress versus strain curve over the range of 0–10% strain was used to calculate the moduli and the reported results were the average of the four specimens.

## RESULTS AND DISCUSSIONS

### Principles on the dimension of buckled patterns

Figure 1 shows the schematic illustration of the procedure involved in the buckling of this study. The cured PDMS substrates are hydrophobic due to their

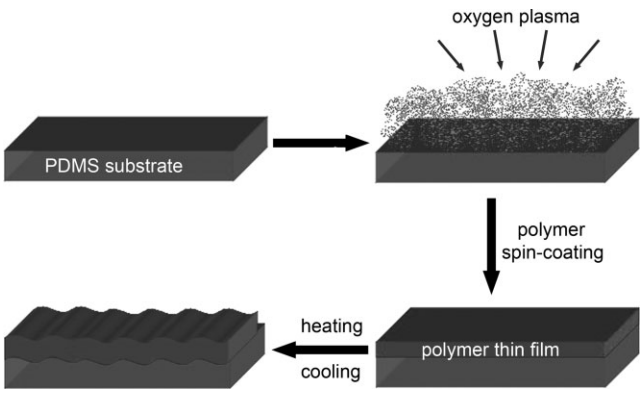


**Figure 1** Schematic illustration of the procedure involved in the buckling. PDMS substrates were lightly exposed to oxygen plasma and polymers were spin-coated on them. Thermal annealing at 170°C and cooling in air generated the buckling of polymer thin films.

hydrophobic repeating unit, —$Si(CH_3)_2O$—.[27] Their poor wettability to most organic solvents results in dewetting of polymer solutions during spin coating. Increasing the surface energy of the substrates enhances their wettability to organic solvents. Exposure to oxygen plasma oxidizes the surface of the PDMS substrates, helping the spin-coating of polymer solutions. Higher dose of plasma creates a ceramic thin layer in nanometer scale. The samples were annealed in a preheated vacuum oven (170°C) for 30 min and then allowed to be cooled. Thermal annealing expands the volume of both the PDMS substrate and the polymer thin layer. Although the polymer chains in the thin layer are relaxed during the annealing time, the stress is stored in the PDMS substrates. Volume shrinkage during the cooling process creates compressive stress because the difference in thermal expansion coefficients of the polymer film and PDMS substrate experiences different levels of contraction. When the modulus of the polymer layer becomes larger than a critical value ($\sigma_c$) at a certain temperature in the cooling process, buckling takes place at the interface to release the compressive stress.[28] The critical compressive stress is known to be determined by an equation,[26]

$$\sigma_c = \left( \frac{9}{64} \frac{E_s^2 E_f}{(1-v_s^2)^2 (1-v_f^2)} \right)^{1/3} \quad (1)$$

where $E$ and $v$ are the Young's modulus and the Poisson's ratio, respectively. The subscripts $s$ and $f$ denote the substrate and film. The film thickness of the substrate and polymer layer is known to be independent of the critical stress when the substrate is much thicker than the polymer layer. The corresponding wavelength ($\lambda_c$) of the buckling pattern is calculated as following,[29]

$$\lambda_c = 2\pi h_f \left[ \frac{(1-v_s^2)E_f}{3(1-v_f^2)E_s} \right]^{1/3} \quad (2)$$

where $h_f$ is the film thickness. From this equation, it is expected that the wavelength of buckling is independent of the thickness of the substrate and can be controlled only by adjusting the thickness of the polymer thin layer and the Young's moduli of the substrate and the polymer thin layer.

### Buckling patterns depending on the thickness of PDMS substrates

PDMS substrates (1.5 cm × 1.5 cm) were prepared by adjusting mixing ratio of the crosslinker to prepolymer (φ) at 0.1 (1 : 10, w/w), which is the most used for bucklings on PDMS substrates. The PDMS substrates were exposed to oxygen plasma for 30 s and then the PS solution (1 wt %) was spin-coated, followed by annealing at 170°C for 30 min and cooling in air. Figure 2 shows AFM images of the buckling patterns of PS thin films on PDMS substrates with different thickness: (A) 0.5 mm, (B) 2 mm, and (C) 20 mm. The wavelength of the buckling (λ) increased with the substrate thickness ($h_s$), which was 2.76 μm, 3.31 μm, and 3.52 μm for 0.5 mm, 2 mm, and 20 mm, respectively. The wavelength was obtained from an average of 10 peak-to-peak distances of parallel wavy patterns. The dependence of λ on $h_s$ is summarized in Figure 2(D). This dependence of wavelength on the substrate thickness was very unexpected because the current theories predict the thickness of the substrates can barely affect the buckling patterns. There is no variable relating the thickness of the substrate with the buckling wavelength in the eq. (2). Because the duration of plasma exposure to PDMS and the thickness of PS thin films are identical, the parameter to affect the buckling patterns is only the change of substrate thickness. The system in this work consists of three layers, PS/oxidized layer/PDMS substrate. The general analysis using a two-layer system incorporating the oxidized layer in the surface thin film cannot explain the effect of the substrate thickness.

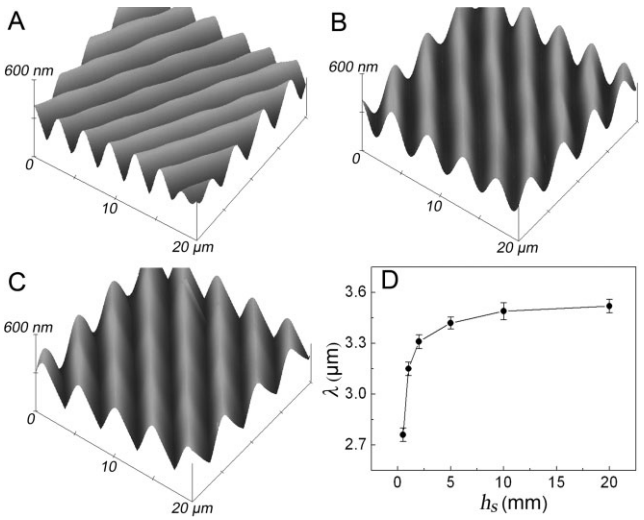


**Figure 2** AFM images showing the effect of substrate thickness ($h_s$) on the buckling wavelength (λ) of PS thin films (30 nm). The crosslinker-to-prepolymer ratio (φ) in the PDMS substrate was fixed at 0.1 (1 : 10, w/w). The thickness of the substrates ($h_s$) was varied: (A) 0.5 mm, (B) 2 mm, and (C) 20 mm. (D) The wavelength (λ) of the buckling sensitively increased and approached to a plateau value as the thickness of the substrate was raised.

To thoroughly investigate the effect of substrate thickness ($h_s$), we changed the crosslinker-to-prepolymer ratio (φ) to 0.025 (1 : 40, w/w). The samples were prepared in the same way performed for Figure 2. Figure 3 shows AFM images of the buckling

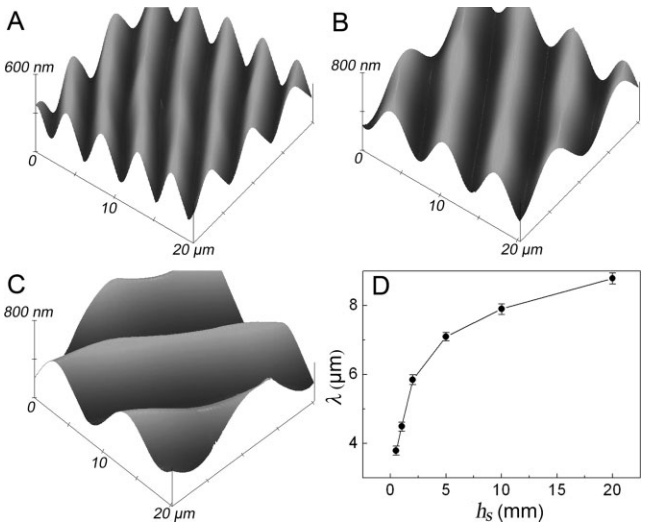


**Figure 3** AFM images showing the effect of substrate thickness ($h_s$) on the buckling wavelength (λ) of PS thin films (30 nm). The crosslinker-to-prepolymer ratio (φ) in the PDMS substrate was fixed at 0.025 (1 : 40, w/w). The thickness of the substrates ($h_s$) was varied: (A) 0.5 mm, (B) 2 mm, and (C) 20 mm. (D) The wavelength (λ) of the buckling continuously increased even when the substrate was 20 mm-thick.

patterns of PS thin films on PDMS substrates with (A) 0.5 mm, (B) 2 mm, and (C) 20 mm, respectively. The wavelength of the buckling (λ) was 3.78 μm, 5.85 μm, and 8.79 μm for 0.5 mm, 2 mm, and 20 mm, respectively. The change of λ versus $h_s$ is represented in Figure 3(D). The dependence on the substrate thickness was much more sensitive when φ was 0.025 than that of φ = 0.1. Comparing Figures 2 and 3, it is obvious that the buckling pattern can be varied by adjusting the thickness of the substrate and the dependency is more sensitive when the crosslinker-to-prepolymer ratio is small (low modulus).

Figure 4 shows the stress–strain curve for the Young's modulus ($E_s$) of the PDMS substrates. The experimental data symbols of (□), (○), (△), (▽), and (◇) correspond to the crosslinker-to-prepolymer ratio (φ) (w/w) of 0.025, 0.05, 0.1, 0.2, and 0.5, respectively. Figure 5 gives the value of Young's modulus ($E_s$) of the PDMS substrates according to the crosslinker-to-prepolymer ratio (φ) (w/w). PDMS sheets with dimension of 3 cm × 1 cm × 0.1 cm were prepared and clamped with binder clips. They were deformed with constant tensile speed of 100 μm/s using the load cell of 200*N* at room temperature. The moduli of PDMS were obtained from the linear slope of the stress ($F/A$) versus strain ($\Delta L/L$) curve in the range of 0–10% strain, where $F$ is force, $A$ is cross section area, $L$ is gauge length of 1.5 cm, and $\Delta L$ is change in length. The modulus increased with the relative amount of crosslinker involved in PDMS mixture, which was 0.08, 0.51, 1.81, 2.61, and 4.35 MPa for φ = 0.025 (1 : 40, w/w), 0.05 (1 : 20, w/w), 0.1 (1 : 10, w/w), 0.2 (1 : 5, w/w), and 0.5 (1 : 2, w/w), respectively.

Figure 6 shows AFM images of the buckling patterns of PS thin films on PDMS substrates with varying crosslinker-to-prepolymer ratio (φ) (w/w): (A) φ = 0.025, (B) φ = 0.05, (C) φ = 0.1, and (D) φ = 0.5. The substrate thickness ($h_s$) was fixed at 20 mm. The buckling wavelength decreased as the value of φ was raised, which was 8.79 μm, 5.48 μm, 3.52 μm, and 2.67 μm for φ of 0.025, 0.05, 0.1, and 0.5, in the order. The increased modulus of the substrate with larger value of φ led to corresponding increase

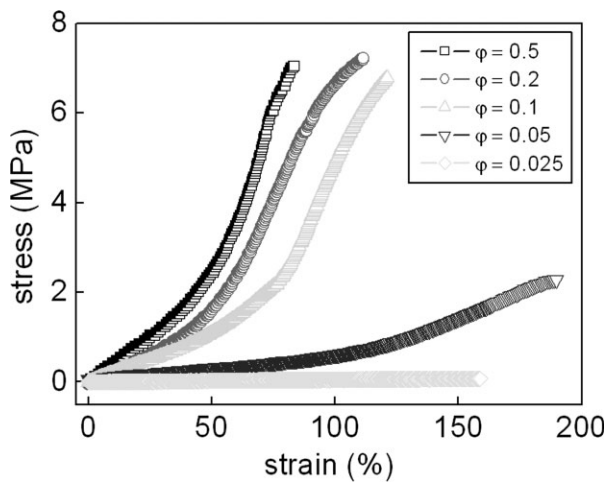


**Figure 4** Stress versus strain to evaluate Young's modulus ($E_s$) of PDMS substrate. The experimental data symbols of (□), (○), (△), (▽), and (◇) correspond to the crosslinker-to-prepolymer ratio (φ) (w/w) of 0.025, 0.05, 0.1, 0.2, and 0.5, respectively.

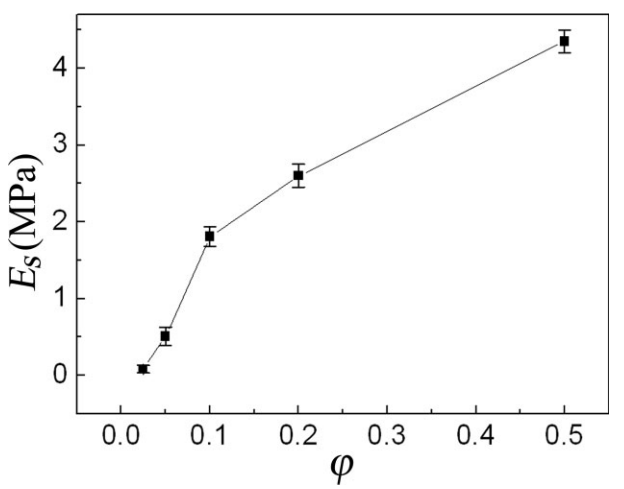


**Figure 5** Change in Young's modulus ($E_s$) of the PDMS substrates with different crosslinker-to-prepolymer ratio (φ) (w/w): 0.025 (1 : 40), 0.05 (1 : 20), 0.1 (1 : 10), 0.2 (1 : 5), and 0.5 (1 : 2).

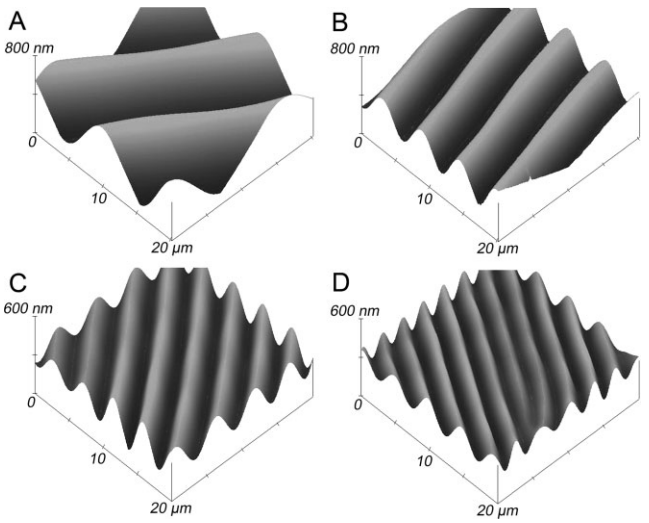


**Figure 6** Effect of the substrate modulus. AFM images show the buckling patterns of PS thin films on PDMS substrates with varying crosslinker-to-prepolymer ratio (φ) (w/w): (A) 0.025 (B) 0.05, (C) 0.1, and (D) 0.5.

in the modulus ($E_s$) of the substrate, therefore decrease in λ.

The optical microscope images of the buckling at various crosslinker-to-prepolymer ratio and different

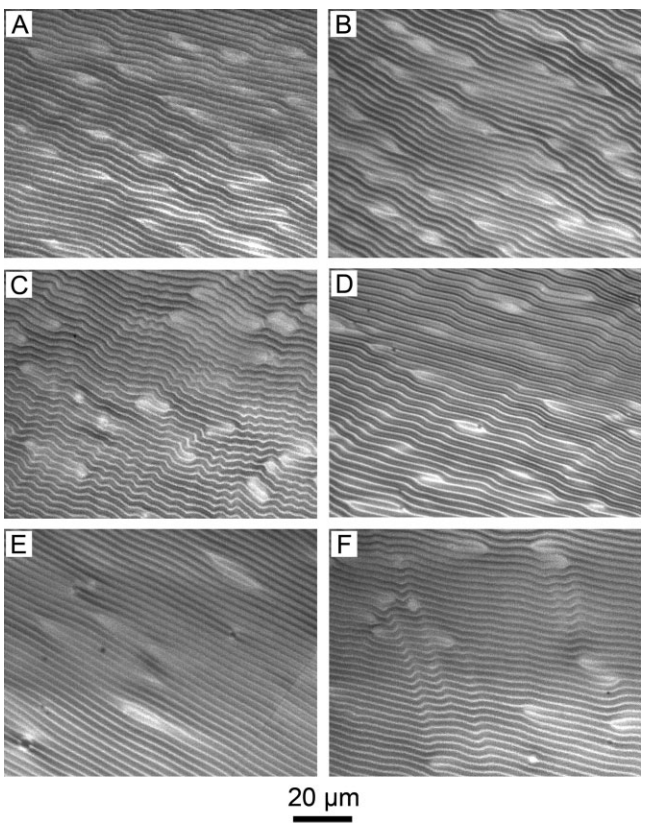


**Figure 7** Optical microscope images showing the buckling patterns of PS films on PDMS substrates with different thickness: (A) 0.5 mm, (B) 1 mm, (C) 2 mm, (D) 5 mm, (E) 10 mm, and (F) 20 mm. The crosslinker-to-prepolymer ratio (φ) (w/w) was fixed at 0.5.

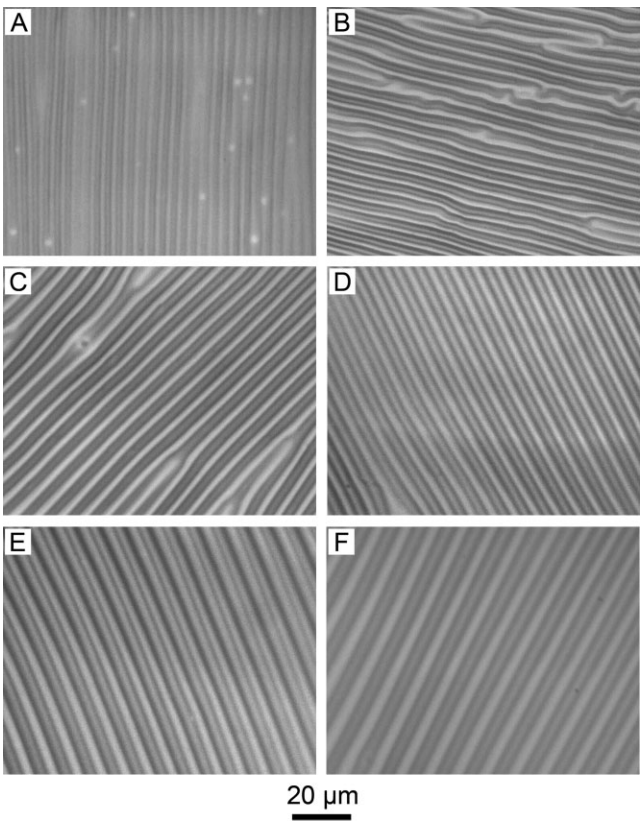


**Figure 8** Optical microscope images showing the buckling patterns of PS films on PDMS substrates with different thickness: (A) 0.5 mm, (B) 1 mm, (C) 2 mm, (D) 5 mm, (E) 10 mm, and (F) 20 mm. The crosslinker-to-prepolymer ratio (φ) (w/w) was fixed at 0.025.

substrate thickness are found in Figures 7 and 8. Each figure contains the buckling patterns of PS films on PDMS substrates with different thickness: (A) 0.5 mm, (B) 1 mm, (C) 2 mm, (D) 5 mm, (E) 10 mm, and (F) 20 mm. The crosslinker-to-prepolymer ratio (φ) was fixed at $\varphi = 0.5$ for Figure 7 and $\varphi = 0.025$ for Figure 8. Such buckling dependence on thickness and substrate modulus has been investigated for various thickness and modulus. Figure 9 summarizes the change in buckling wavelength (λ) of the PS thin films on PDMS substrates with varying modulus ($E_s$) and thickness ($h_s$) of the substrate. The symbols are the experimental data and the solid lines are guides to the eyes. The inset box indicates the thickness of the substrates. Three aspects should be noticed in the results. First, in any substrate thickness, the wavelength was inversely proportional to the modulus of the substrate and approached a nearly constant value. Second, the sensitivity of the wavelength on the substrate modulus was higher as the substrate became thicker. When the substrate was 20 mm-thick, the buckling wavelength was sharply decreased as the modulus of the substrate increased, whereas the dependence was not abrupt in the 0.5 mm-thick substrate. The minimum

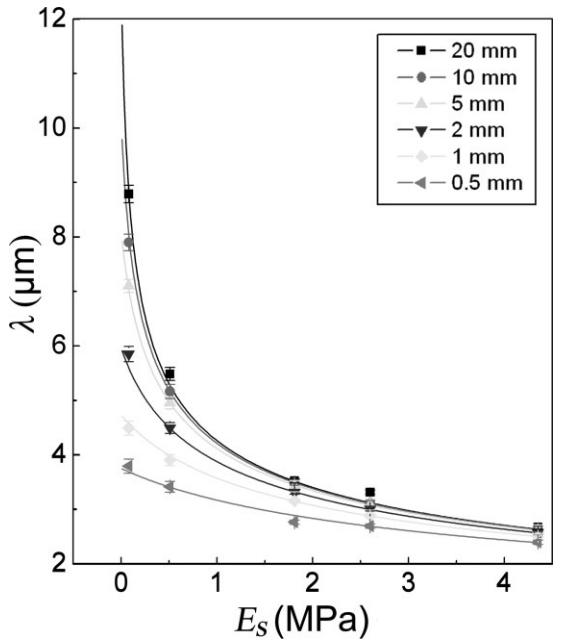


**Figure 9** Wavelength (λ) change in the buckling of the PS thin films on PDMS substrates with varying modulus ($E_s$) and thickness ($h_s$). The experimental data symbols of (■), (●), (▲), (▼), (♦), and (◀) correspond to the substrate thickness ($h_s$) of 20 mm, 10 mm, 5 mm, 2 mm, 1 mm, and 0.5 mm, respectively. The solid lines are guides to eyes.

wavelength from the above setup is considered to be around 2 μm regardless of the thickness and modulus of the substrates, whereas the wavelength can be increased by using a thick and soft substrate. Third, the buckling wavelength did not converge on one point, leaving offsets between different substrate thicknesses. The offset takes place because the contribution of the oxidized layer to the substrate modulus becomes less as the thickness of the substrate is increased, which results in larger wavelength.

The dependence on the substrate thickness with varying crosslinker-to-prepolymer ratio was displayed in Figure 10. The symbols are the experimental data and the solid lines are guides to eyes. The wavelength was more affected by the substrate thickness ($h_s$) when φ was small. For large φ (0.5 and 0.2) which are larger than the usual value (φ = 0.1), the wavelength sharply increased as the substrate thickness was raised up to ~ 2.5 mm and then gradually reached a plateau value. It deserves a note that the value 2.5 mm is not expectable because the film thickness was only 27 nm and the substrate is six order thicker than the polymer layer. Normally, when a substrate is two order thicker than a thin film, the dependence on the substrate thickness is expected to be negligible. For small φ = 0.025, the wavelength did not reach a plateau even on 20 mm-thick substrates. This behavior was not reported before and has not been estimated by theories. We expected a plateau value for φ = 0.025 on 100 mm-thick substrates. The results in Figures 9 and 10 are attributed to the large difference between the moduli of the substrate ($E_s$), the polymer thin film ($E_f$), and the oxidized layer ($E_o$). The oxidized layer (~ 10 nm) is six order smaller than the substrate thickness (20 mm), whereas its modulus is five order higher than the modulus of PDMS with φ = 0.025. Moreover, because the oxidized layer greatly increase the film modulus when the polymer was used for the thin film layer, small change in the substrate modulus can induce large change in the buckling wavelength. The results in Figures 9 and 10 reflect the substrate can play as a sensitive variable for the thin film buckling.

### The effect of oxidized layer on the buckling wavelength

Because the strong dependence of buckling wavelength on the substrate thickness shown in Figure 10 comes from the existence of the oxidized layer, thickness control of oxidized layer can result in large change in the buckling wavelength. According to the models[30,31] to explain the formation of oxidized layer on PDMS, carbon is removed in the form of volatile species in the first region with high oxidation rate. Oxidative crosslinking step via Si—O bridges is involved at first, leading to formation of barrier layer of $SiO_x$. After a certain thickness of $SiO_x$ barrier is formed, the diffusion of oxidative species governs the oxidation rate of PDMS network,

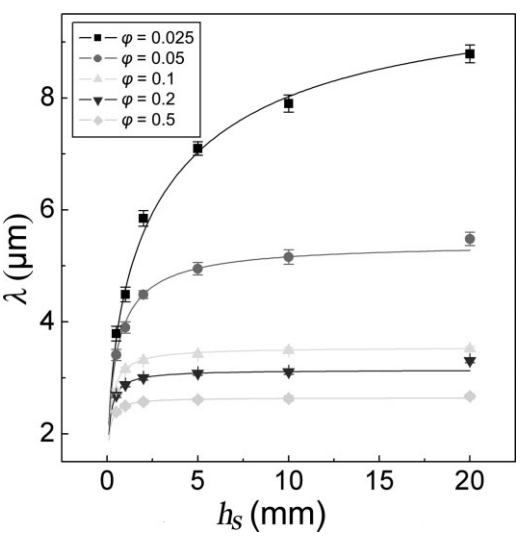


**Figure 10** Wavelength (λ) change in the buckling of the PS thin films on PDMS substrates with varying thickness ($h_s$). The experimental data symbols of (■), (●), (▲), (▼), and (♦) correspond to the crosslinker-to-prepolymer ratio (φ) (w/w) of 0.025, 0.05, 0.1, 0.2, and 0.5, respectively. The solid lines are guides to eyes.

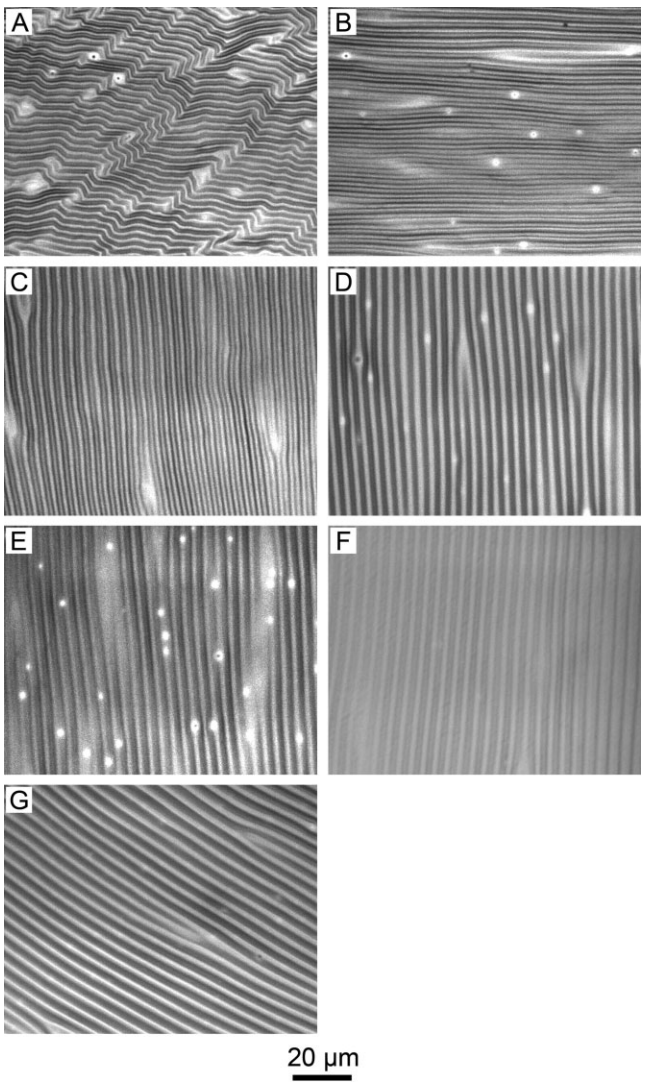


**Figure 11** Optical microscope images showing the buckling patterns of PS thin films on PDMS substrates with different exposure time to oxygen plasma: (A) 30 s, (B) 60 s, (C) 90 s, (D) 120 s, (E) 180 s, (F) 240 s, and (G) 300 s. The crosslinker-to-prepolymer ratio (φ) (w/w) and the thickness of PDMS substrate ($h_s$) were fixed at 0.5 and 5 mm, respectively.

therefore, the oxidation rate becomes low. Optical microscope images in Figures 11 and 12 show the buckling patterns for different exposure time (30, 60, 90, 120, 180, 240, and 300 s) to oxygen plasma on different crosslinker-to-polymer ratios: $\varphi = 0.5$ for Figure 11 and $\varphi = 0.025$ for Figure 12. The thickness of PDMS substrate ($h_s$) was fixed at 5 mm. Such buckling dependence on the plasma exposure time has been investigated for various crosslinker-to-polymer ratios. Figure 13 summarizes the change of wavelength (λ) of the buckling as a function of exposure time to oxygen plasma. The thickness of PDMS ($h_s$) was fixed at 5 mm for all data. The inset is the crosslinker-to-prepolymer ratio. The solid lines are guides to the eyes. Overall behavior of the wavelength was the same regardless of the substrate modulus. The buckling wavelength steeply increased in first 2 min and then linearly proportional with plasma exposure time. This behavior has been already observed in the literature.[5]

We presented the unexpected dependence of buckling wavelength on the variables of PDMS substrate, which was the thickness and modulus. Unfortunately, the analytical calculations cannot explain such results. The theoretical development is left for the future studies.

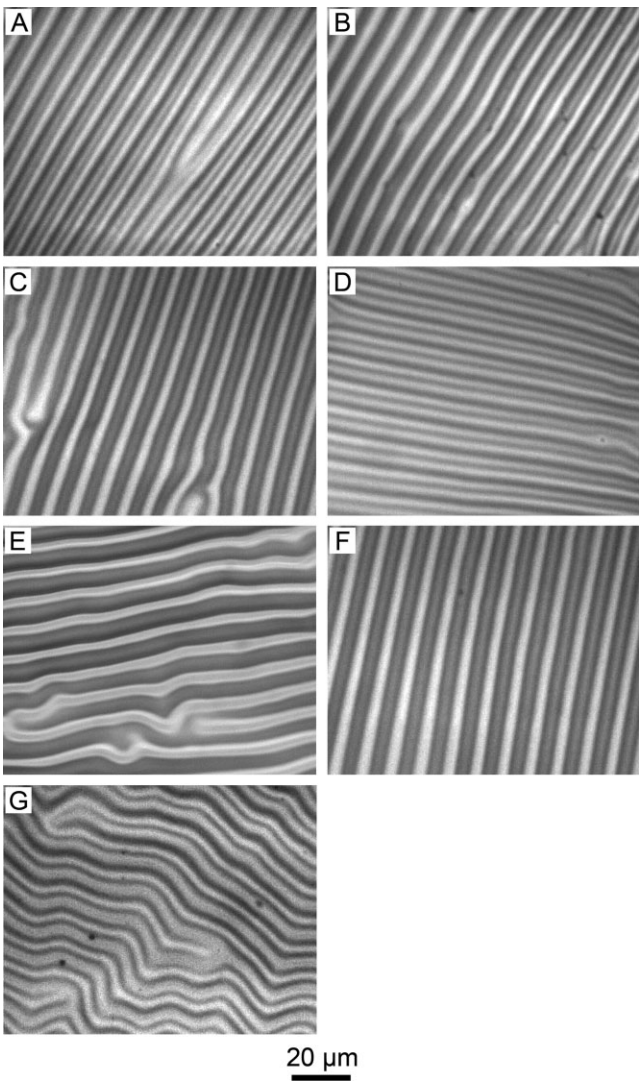


**Figure 12** Optical microscope images showing the buckling patterns of PS thin films on PDMS substrates with different exposure time to oxygen plasma: (A) 30 s, (B) 60 s, (C) 90 s, (D) 120 s, (E) 180 s, (F) 240 s, and (G) 300 s. The crosslinker-to-prepolymer ratio (φ) (w/w) and the thickness of PDMS substrate ($h_s$) were fixed at 0.025 and 5 mm, respectively.

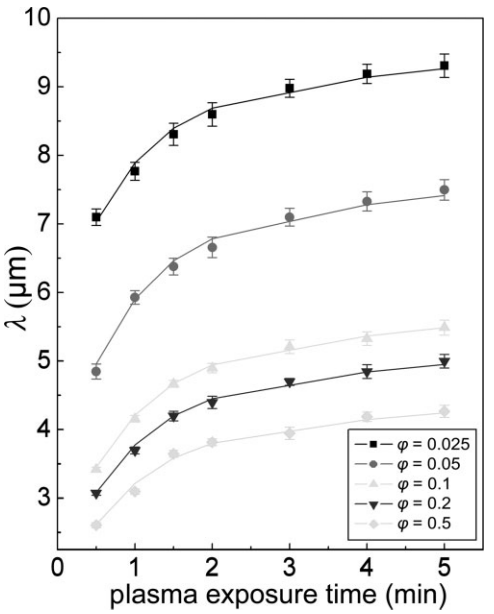


**Figure 13** The change of wavelength (λ) of the buckling as a function of exposure time to plasma. The thickness of PDMS substrate was fixed at 5 mm. The experimental data symbols of (■), (●), (▲), (▼), and (♦) correspond to the crosslinker-to-prepolymer ratio (φ) (w/w) of 0.025, 0.05, 0.1, 0.2, and 0.5, respectively. The solid lines are guides to eyes.

## CONCLUSIONS

For the first time, we have demonstrated that the thickness of a substrate is an effective parameter to control the buckling patterns. On PDMS substrates with high modulus, the buckling wavelength of polymer thin films rapidly increased as the thickness of the substrates were raised and approached a plateau value when the substrate was 2.5 mm-thick. Such high dependence on the substrate thickness has not been observed or predicted before. On PDMS substrates with low modulus, the wavelength still increased with the substrate thickness at even 20 mm-thick. The sensitive behavior on the substrate thickness results from the large difference between the modulus of the PDMS substrate and the oxidized layer.

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