Chapter 4

REVERSIBLE SURFACE PATTERNING BY DYNAMIC STRESS GRADIENT INDUCED IN THE SUBSTRATE

4.1 CHAPTER OUTLINE

Utilizing the self-organization of soft materials to make systematic and ordered patterns can be very useful for optical-electronic and many other applications. The wrinkling of the thin-film provides captivating strategies to execute different applications. Despite several attempts to fabricate wrinkled surfaces, a simple, reversible ordered, and gradient pitch wrinkled surface is still proven to be elusive. In this chapter, we demonstrate to fabricate a mechanically self-organized linearly wrinkled smart optical pattern developed in polystyrene (PS) thin-film placed over an elastic Polydimethylsiloxane (PDMS) substrate. The developed substrate was demonstrated to be used as a smart optical filter giving different intensities of diffused light for different positions of the substrate. The origination of the pattern was due to stress induced in the PDMS substrate, which when removed, created localized buckling in the thin film placed over it. Control of the thin film topology was obtained by varying stress in the elastic substrate and thin-film thickness. Theoretical analysis and experimental demonstration of the results were done. A polystyrene film is coated on a glass substrate and floated over a gradient-stressed substrate of PDMS. Surface topography characterization using an optical microscope is done and Optical transmittance at different locations of the substrate was recorded for a constant intensity illumination using a lux meter. Application of the optical transmittance variation of the fabricated pattern was demonstrated for controlled diffusion of light and in continuation of our previous work we used it to illuminate samples for photomicrography. Potentially this arrangement can also be used to create microchannels for sorting biological samples etc.

4.2 INTRODUCTION

The wrinkling and buckling of thin films have been an interesting theme of research for a few decades.[1,2] When a thin film is coated over a thick substrate having different mechanical properties, the difference in material properties can lead to the formation of relief patterns or wrinkles. These wrinkles are generated due to a gradient of normal stress in the thin-film due to mechanical, osmotic, or thermal reasons.[3,4] Solvent-induced swelling can also trigger instability in thin films coated on rigid substrates causing wrinkling in the thin film.[5] Different methods of fabrication have their advantages and disadvantages, and suitability for different applications. Micro/nanoscale patterns with controllable geometries and dimensions have been used in biology, adhesives, electronics, and optics.[6-8]

Smart materials facilitating reversible switching from transparent to opaque have been explored for various applications.[9] Such smart materials can have potential applications as smart windows for energy-efficient buildings and vehicular applications.[9] Most of the development of such dynamic smart materials has mainly focused on chromogenic materials and devices such as electrochromic[10] and thermochromic[11,12] which are responsive to electrical field or temperature. However, these chromogenic materials are usually expensive, require complex fabrication processes, and pose problems of stability, durability, and functionality.[13,14]

Recently, there has been growing research interest in harnessing surface wrinkling to address some of these limitations. Wrinkling phenomena are often observed in our daily life such as wrinkles on human skin and dried fruits. Wrinkle pattern-based smart materials offer ease in fabrication, material handling, and pattern control.[15-18] Such facilitations provide an opportunity to fabricate a new class of smart optical materials. There are several methods studied to prepare wrinkle-based smart optical materials.[16-19] However, mechano-responsive optical wrinkled patterns have been proven to be more robust in tunability using simple mechanical strain.[19] Wrinkled light scattering surfaces having micro- and nano-topography have been demonstrated to reversibly switch between opaque to transparent using mechanical strain.[20] However, it often requires an additional electrical, thermal, or mechanical input to turn from opaque to transparent.

In this study wrinkling of thin polystyrene (PS) film over the elastomeric polydimethylsiloxane (PDMS) substrate is presented. The elastomeric substrate was bent by bringing the two opposite sides together, which generates a varying degree of strain that is maximum at the center. Then a strain-free PS thin film was transferred on the PDMS substrate and then the strain in the substrate was released allowing it to take a flat configuration. This produces compressive stresses in the PS film resulting in the wrinkling or buckling of the film. These wrinkled patterns were denser in the center and gradually sparse away from the center line due to gradient stress present in the substrate. Moreover, there was a preferential alignment of these wrinkles in the orthogonal direction of bending. Experimentally observed thin film buckling length was compared with finite deformation theory.[21] It is further demonstrated that a linearly aligned, wrinkled substrate with gradient pitch results in variable transmissivity and can be used effectively as a variable light diffuser. Such substrates can be used to change the transmittance of light through it, by simply changing the position against a point light source, obviating the need for any external mechanical stimuli to induce the wrinkle and change the transmittance.

4.3 MATERIALS AND METHOD

Fabrication of gradient wrinkled pattern and demonstration of its ability to diffuse the light in a controlled manner was demonstrated. The experimental methods are as follows.

4.3.1 Fabrication of Wrinkled Pattern

To make elastomer (PDMS) substrates of different thicknesses, Sylgard$^{\text{TM}}$ 184 (10:1 mix) was poured on a flat horizontal glass surface and heated at 120°C on a hot plate for 120 minutes. PDMS substrates of different thicknesses ranging from 1 to 4 mm were prepared and cut in the dimensions of 15×15 mm^2. A 4% (w/v) PS (M_w = 40 kg mol^{-1}) solution in toluene is used to prepare PS film of different thicknesses ranging from 50nm to 250 nm by spin coating on a clean glass substrate.[22] The PS film was floated on water from the glass substrate. The experimental procedure needs due care for the sake of reproducibility. Wrinkles in the thin film placed on the PDMS substrate are generated due to the gradient stress-induced. To control the gradient stress, the size of the PDMS substrate was kept at a fixed size during all the experiments. Also, the curvature induced was kept due to the natural inertia of the substrate while folding, and the two sides were just made to touch each other. With the help of a tweezer and having more precise control over the thickness of polystyrene thin film made the experiments more reproducible.

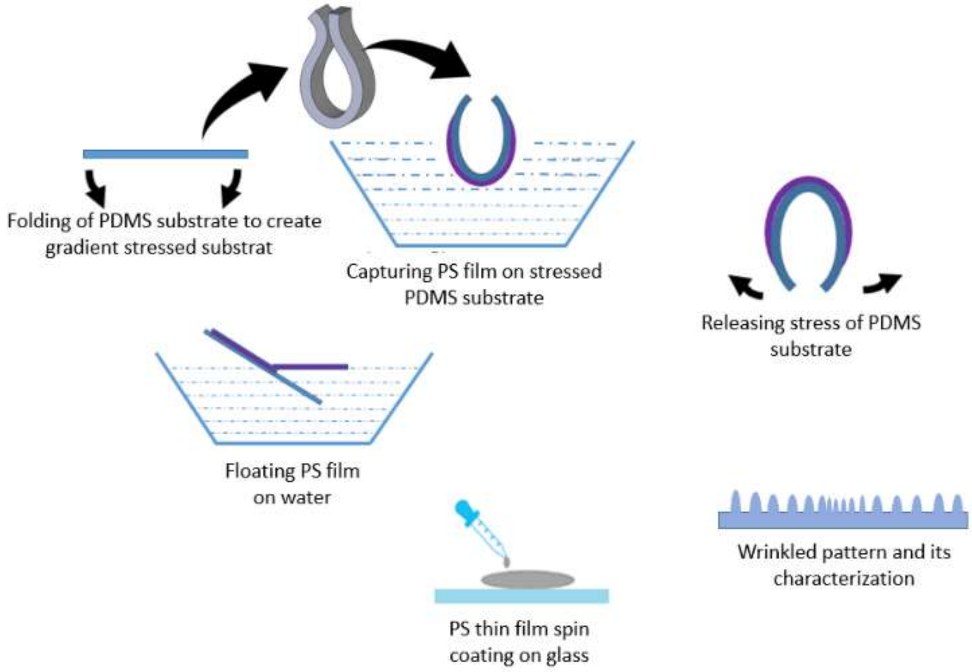

Figure 4.1 Fabrication process of the wrinkled substrate on a gradient-strained elastomeric substrate.

To produce variable strain in the PDMS substrate it was bent by joining two opposite sides of the substrate as shown in Figure 4.1 and used to capture the PS film. The substrate was kept in the bent configuration until the film get dried (in the air for about 10 minutes) and then slowly the strain was released in the substrate to generate the wrinkles over it. The center line parallel to the joined sides which is also the line joining the points with maximum strain in this case was defined as the bending axis. Wrinkles at different locations on the substrate were observed under an optical microscope and characteristic lengthscales of wrinkles are measured as a function of the thickness of the substrate and distance from the bending axis.

4.3.2 Diffusion of light through the substrate

A wrinkled substrate was used to diffuse the light based on the density and spacing between the wrinkles. A constant intensity illumination source of 120 Lux was kept in front of the lux meter. The wrinkled substrate is kept between the source and the lux meter. The distance between the illumination source and the wrinkled substrate was about 1cm, while the wrinkled substrate was kept just over the lux meter. The intensity graph of the lux meter is recorded while sliding the wrinkled substrate, to see the impact of wrinkles on the diffused light intensity. Such properties of the substrate are utilized in the photomicrography of micrometer-size objects as the setup demonstrated in Figure 4.2. A cellphone flashlight was used as an illumination source, and at the bottom, the lux meter was placed. Support provided at the middle is to support the wrinkled patterned substrate and slide it over it.

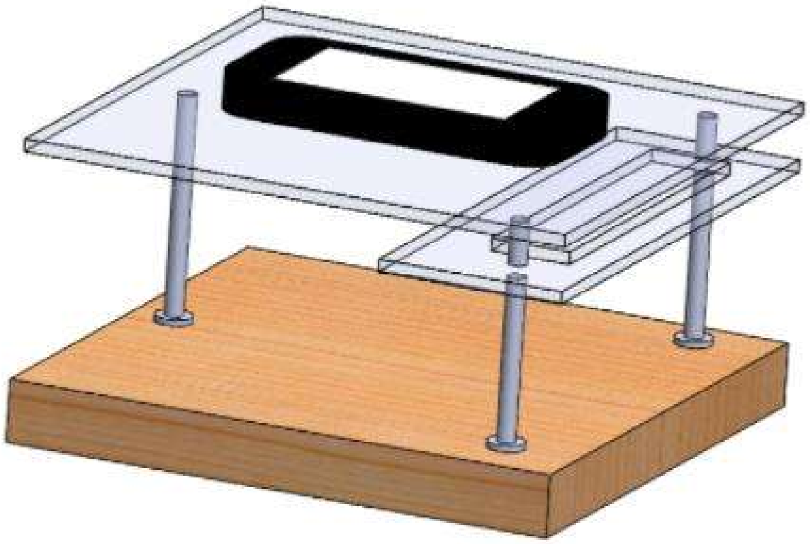

Figure 4.2 Schematic diagram of the application procedure of the wrinkled substrate. The wrinkled substrate or the cellphone camera can be moved to illuminate the sample with different intensities of diffused light.

4.4 RESULT AND DISCUSSION

Firstly, It was confirmed that delamination is not occurring along with theoretical analysis reported in previous studies. Then stress induced into the substrate and its effect on wrinkle formation was analyzed moving ahead with wrinkle characterization. Finally, the application of the substrate is discussed.

4.4.1 Lamination/Delamination of the Thin Film From the Substrate

Wrinkling or buckling of a hard thin film placed over a soft substrate is a well-known phenomenon in nanomaterials engineering.[2,23] When there is residual stress in the thin film due to thermal, mechanical, or any other reasons, the thin film may become unstable. When the residual stress reaches a critical level, the thin film surface buckles to release all or part of the residual stress and resulting in wrinkling, delamination, or cracking in the film.[1] If the lengthscales of the wrinkles on thin film can be controlled it can be very useful in various applications. The buckling of metallic thin films on an elastomeric substrate has been studied for its use in micro and nanoscale structures to generate well-defined structures in the 100nm to 100µm range.[24]

Buckling in the thin film may result due to compressive stress development which if exceeds a critical value may result in wrinkling or delamination. Delamination may further result in the cracking or peeling off of thin film over the substrate. There have been several mathematical models proposed to explain the buckling in a thin film placed on a flexible substrate.[25,26] Major theories proposed to explain the buckling and wrinkling phenomenon are small deformation theory[27,28] and finite deformation theory.[29,30] In this chapter, we will first see the compliance of the thin film with the viscoelastic substrate and delamination. Experimental observations of wrinkling will be explained with reference to deformation theories.[30]

Captured thin film on the PDMS substrate was cut from the backside of the surface where the film was deposited. The direction of the cut was kept perpendicular to the direction of the gradient stress applied as shown in figure 4.3a so that the compliance between substrate and film can be seen at different points of the substrate. Figure 4.3(b)(c)(d) shows the microscopic view of the cross-section of the thin film and substrate compliance junction, perpendicular to the direction of stress applied. It can be seen that the thin film and viscoelastic substrate boundaries comply throughout the substrate and no spacing between the two boundaries is visible. Also, it was observed that the stress-induced resulted in the tilt of the boundary layer of thin film and substrate towards the bending axis.

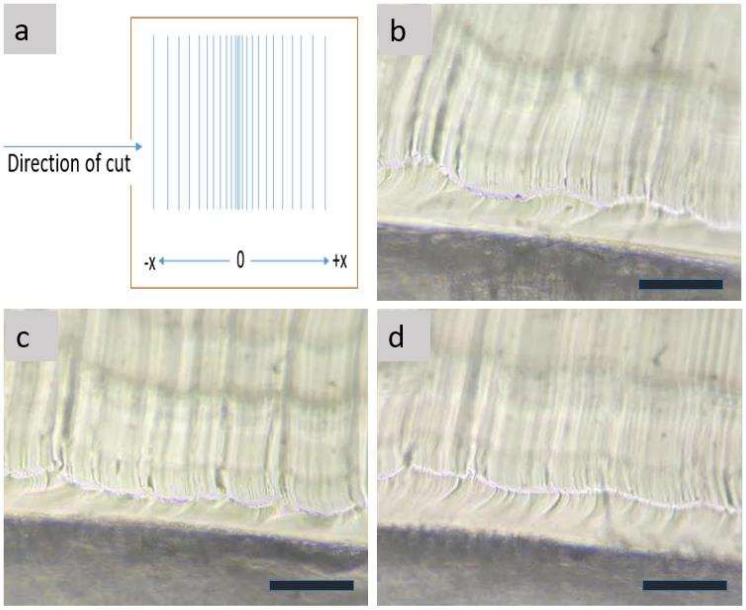

Figure 4.3 Cross-sectional cuts of the thin film and substrate boundary layer. (a) In the direction of the cross-section of the substrate, a cut was made from the opposite side of the PS film (b) (c) (d) microscopic view of the boundary layer at different places of the substrate. The scale bar is 5µm.

Microscopic visuals confirm that thin film and substrate comply throughout the axial length. Thus further analysis of the process was done with the wrinkling perspective using small and finite deformation theories. Since compressive stress results in delamination or wrinkling, the possibility of cracking is omitted. Later part of the chapter deal with the characteristic and application of the gradient wrinkled substrate.

4.4.2 Analysis of Wrinkling Phenomena with Deformation Theories

The wrinkling of the thin film on a strained substrate can be seen in the light of deformation theories. Small deformation theory is for a stiff film of thickness t_f and elastic modulus E_f placed on a stretched compliant substrate of elasticity modulus E_s ($E_s << E_f$) as shown in Figure 4.4.[27,28] Strain developed in the stretched film is defined as

$$\mathcal{E}_{pre} = \Delta L / L \tag{1}$$

Previous mechanics model based on small deformation theory gives a fixed wavelength of buckled film as

$$\lambda_o = 2\pi h_f \, [\bar{E}_f \,/\, (3\bar{E}_s)]^{1/3} \tag{2}$$

The amplitude of the wrinkles in the buckled film is given as

$$A_o = h_f \sqrt{\mathcal{E}_{pre}/\mathcal{E}_c - 1} \tag{3}$$

Here $\bar{E}_s$ and $\bar{E}_f$ are defined as $\bar{E}_s = \frac{E_s}{(1-\nu_s^2)}$ and $\bar{E}_f = \frac{E_f}{(1-\nu_f^2)}$, where ν_s and ν_f are Poisson's ratio of substrate and film. Critical strain is defined as the minimum strain required to start buckling in the thin film defined as

$$\mathcal{E}_c = \frac{1}{4}(3\bar{E}_s/\bar{E}_f)^{2/3} \tag{4}$$

The critical strain value for PDMS and PS film was calculated as 3.84% using values provided in the literature for PDMS and PS. Young's modulus of elasticity of PDMS and PS was taken as 1.8 and 3250 MPa respectively and Poisson's ratio as 0.48 and 0.34 respectively.[31,32]

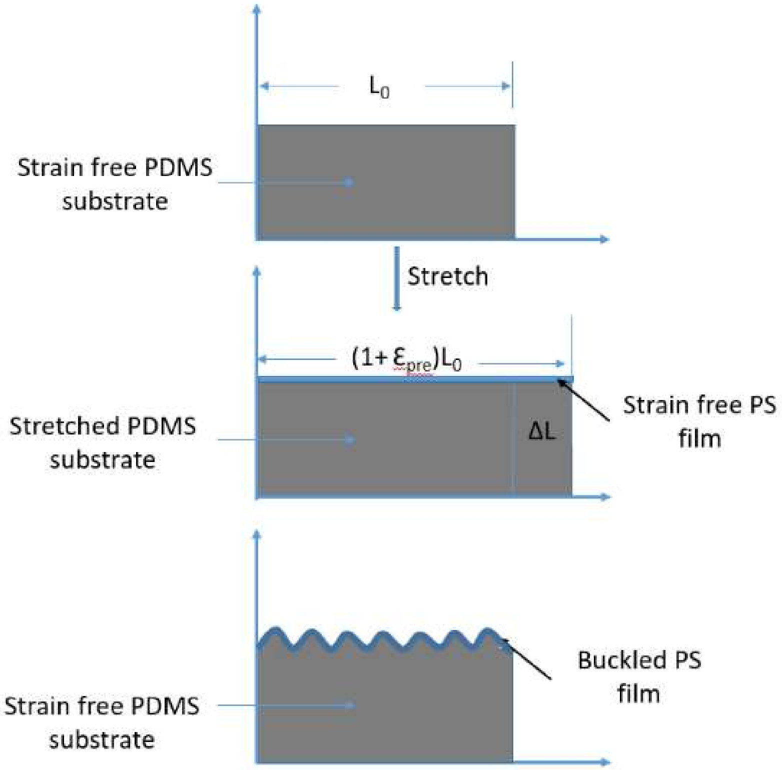

Figure 4.4 Sequential configurations of the buckling process of PS thin film on the PDMS substrate.

Small deformation theory gives preferentially aligned patterns locally but not in the long range. Also for the large strain deformations, a systematic and coherent decrease in wavelength was observed. Earlier studies pointed out that strain-dependent wavelength is due to the finite deformation in the compliant substrate and PS film on PDMS substrate demonstrated such large strain deformation behavior.[30]

Finite deformation theory states that the total energy in the thin film and substrate system consists of the membrane energy (U_m), bending energy (U_b) in the thin film, and strain energy (U_s)of the substrate giving $U_{tot} = U_m + U_b + U_s$.[29,30] Buckling occurs to rearrange the film to minimize the complied energy thus minimizing total energy i.e. $\frac{\delta U_{tot}}{\delta A} = \frac{\delta U_{tot}}{\delta \lambda} = 0$

Solving these equations gives buckling wavelength as $\lambda = \frac{\lambda_o}{(1+\varepsilon_{pre})(1+\xi)^{1/3}}$ and amplitude as $A = \frac{A_o}{\sqrt{(1+\varepsilon_{pre})}\,(1+\xi)^{1/3}}$, where λ_o and A_o are wavelength and amplitude based on small deformation theory and $\xi = \frac{5}{32}\frac{\varepsilon_{pre}}{(1+\varepsilon_{pre})}$.

It should be noted that $A = A_o$ *for* $\varepsilon_{pre} > \varepsilon_c$ as illustrated in earlier theories. However, the wavelength in finite deformation is less than that from the small deformation theory as strain increases. Theoretically calculated values of λ_o at the bending axis of the substrate, for different film thickness, shows that by increasing the film thickness from 50 to 250 nm wrinkle pitch increases from 2.94µm to 11.75µm.

Peak strain in the thin film, when $\varepsilon_{pre} < \varepsilon_c$, relaxing the prestrain does not induce buckling.[30] In such case film will sustain a small compressive strain $-\varepsilon_{pre}$ which is referred to as the membrane strain. The membrane strain as evaluated at the midpoint plane is perpendicular to the film thickness

$$\varepsilon_{mem} = -\frac{1+\xi/3}{(1+\xi)^{1/3}}\,\varepsilon_c \tag{5}$$

For prestrain up to 100%, this membrane strain essentially remains constant. Maximum strain in the film known as peak strain ε_{peak} is the sum of membrane strain (ε_{mem}) and bending strain induced by buckling. In most of the practical cases involved, the strain originated due to buckled geometry is much higher than membrane strain and this peak strain can be evaluated by

$$\varepsilon_{peak} = 2\sqrt{\varepsilon_{pre}\,\varepsilon_{c}}\,\frac{(1+\xi)^{1/3}}{\sqrt{1+\varepsilon_{pre}}} \tag{6}$$

The analysis made above can be used to predict the wrinkled pattern morphology with desired pitch gradient for a given Young's modulus and Poisson's ratio.

4.4.3 Wrinkled Surface Analysis

Tensile or compressive stress induced in a thin film placed over a substrate has been reported earlier by Gerald Stoney.[14] Stoney equation suggests that the stress induced in a thin film σ, bonded over a substrate is directly proportional to the curvature of the substrate and the second power of the substrate thickness as per the following equation

$$\sigma = \frac{E_s.t_s^2}{1-\nu_s 6t_f}k \tag{7}$$

Where E_s is Young's modulus of the substrate, v_s is the poisons ratio, $t_{s,}$ and t_f are the thickness of substrate and film respectively. k, the curvature of the substrate, due to which the stress is induced, is the most important parameter here. The Stoney equation gives the strain profile across the length, which can be used to find the critical strain across the length of the viscoelastic substrate. As shown in Figure 4.5, Strain developed in the substrate is proportional to the thickness of the substrate and can be defined as

$$\varepsilon = y/R \tag{8}$$

where R is the radius of curvature of the differential element and y is the thickness of the substrate from the neutral axis. The bending axis was the symmetric line, from where the distance from the force applied on the sides is maximum. The maximum curvature (minimum radius of curvature) of the substrate is at this bending axis, gradually decreasing towards the sides leading to the infinite radius of curvature at the sides. Since maximum curvature is at the bending axis, maximum pre-strain and hence maximum compressive stress in the thin film layer is at the bending axis. Strain and resulting compressive stress in the substrate gradually decreased towards the sides of the substrates as the curvature of the substrate and strain decreases. It is evident that the wrinkles organize themselves to minimize the stress in the thin film.[15]

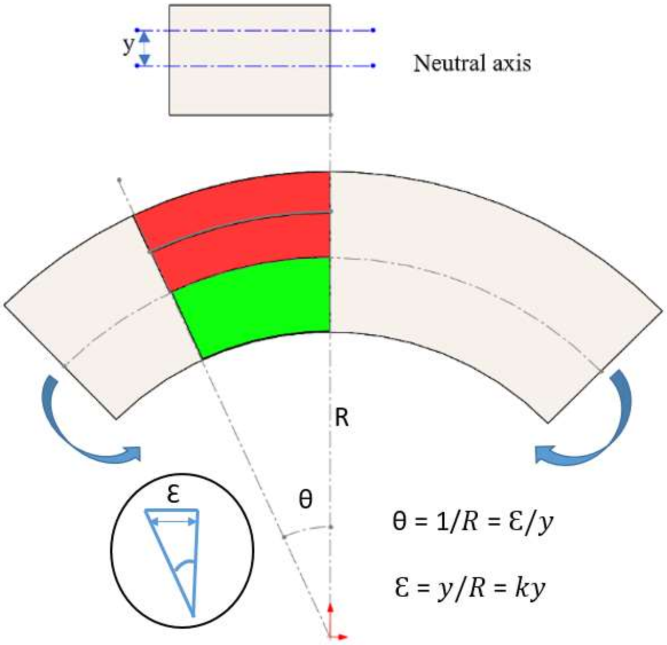

Figure 4.5 Bending analysis of the PDMS substrate. The radius of curvature R, beam curvature k, and strains in the different layers of the substrate thickness.

Thus the wrinkles are denser with a lower pitch in the middle of the substrate and gradually increasing the pitch towards the end of the substrate. Critical strain to induce the buckling in the thin film can be calculated from equations 4 and 8 and was found to be 3.84×10^{-2}. From equations 4 and 8, and experimentally measuring the curvature of the substrate we found that for a square substrate of 15 mm side, the minimum thickness required to induce more than critical strain Ɛc at the bending axis is nearly 400 µm. PDMS substrates of 1-4 mm thickness were used to create wrinkles and observed using a microscope.

A preferentially aligned pattern with increasing wavelength from the bending axis to both sides of the substrate was observed as shown in Figure 4.6 (a–d). Figure 4.6 represents wrinkles induced on a PDMS substrate of thickness 2 mm and PS film thickness of 100 nm. It is evident that wrinkles are denser at the bending axis of the substrate (Figure 4.6a) and the spacing between wrinkles gradually Increased towards the side of the substrate (Figure 4.6b-d)

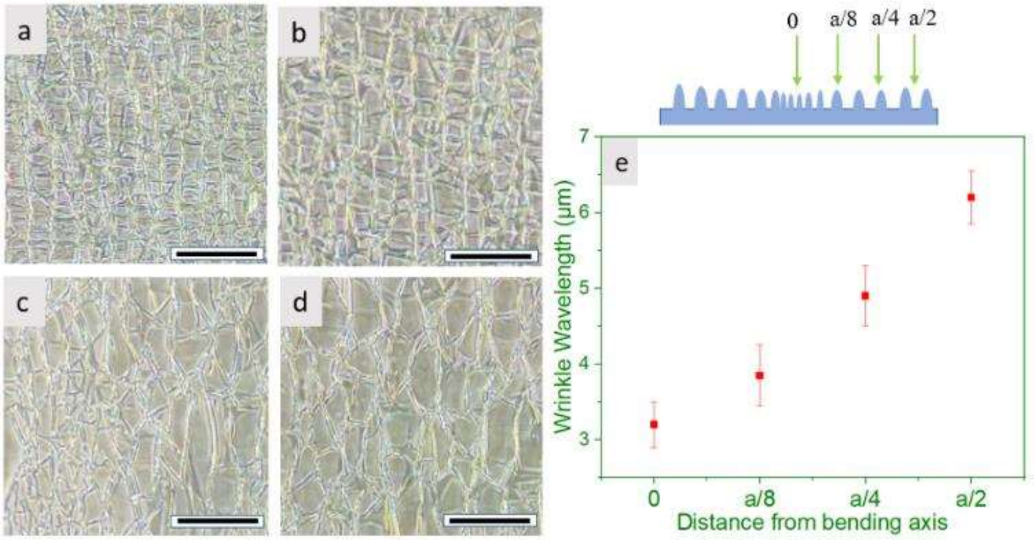

Figure 4.6 Wrinkled patterns corresponding to different positions on the substrate as depicted in the schematic diagram. (a) Denser wrinkled pattern corresponds to the middle part of the substrate. (b) and (c) Moving towards one end of the substrate, the pitch of the wrinkled pattern increase gradually. (d) At one end of the substrate wrinkles almost disappear. (e) Wrinkle wavelength corresponds to substrate position, where a is the half length of the substrate. The scale bar is 20µm.

4.4.4 Thickness Dependence of Wrinkles

As per the Stoney equation (Eq. 7) and bending analysis discussed above, the stress in the film bonded over a substrate is directly proportional to the second power of the thickness of the substrate. Strain $\mathcal{E}$ developed in the substrate is dependent on the substrate thickness and substrate curvature on bending. Bending and curvature are maximum at the bending axis and decrease towards the side of the substrate. Therefore the density of the wrinkled pattern is also dependent on the thickness of the substrate. Different substrates of the same size with varying thickness from 1mm to 3.5mm, were taken to capture the polystyrene thin film, and wrinkles spacing were characterized. Performing experiments for substrate thickness 1mm was difficult, as there was a very small region where wrinkling occurred. For each substrate thickness, five samples were made, and on each sample, six measurements were taken. Based on the data collected, the average value is reported and the standard deviation was used for calculating the error bar.

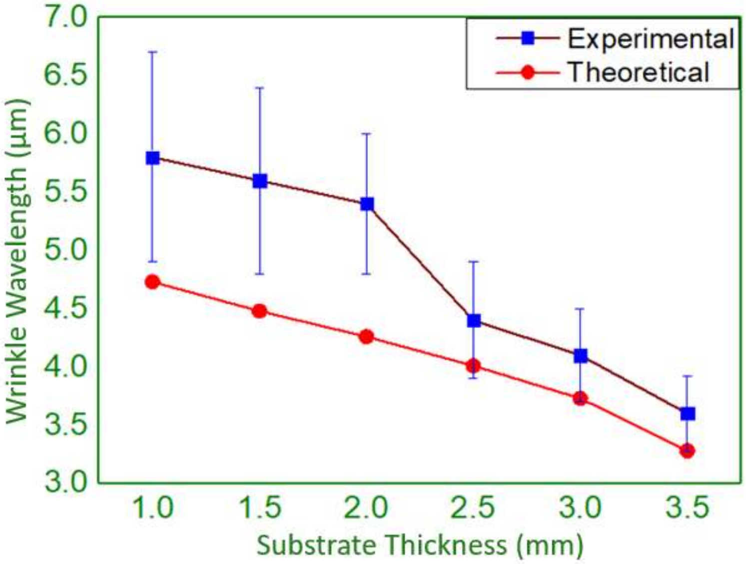

Figure 4.7 Relation between the wavelength of the wrinkles at the bending axis and thickness of the substrate. It is observed that increasing the thickness induces more stress and hence denser wrinkles with short wavelengths at the bending axis.

When the region around the bending axis of the substrate is examined by the optical microscope, an inverse relationship is observed in the substrate thickness vs wavelength of the wrinkled pattern. The theoretical value of wavelength at the bending axis for different substrate thicknesses is calculated and plotted against the experimentally observed values. These results are qualitatively in agreement with the theoretical values as shown in Figure 4.7. For substrate thickness up to 2 mm wavelength of the wrinkled pattern is almost constant, further increasing the substrate thickness reduces the wavelength spacing between wrinkles. This observation is in line with the theoretical understanding that for lower substrate thickness, wavelength spacing follows a small deformation model. For substrate thickness higher than 2.5 mm wavelength spacing follows the finite deformation theory of buckling.

4.4.5 Optical Diffusion Using Wrinkled Pattern

To demonstrate one possible application of the gradient wrinkled pattern we have placed it in front of a point light source and examined its light diffusion ability using a lux meter at different positions of the substrate. Figure 4.8 shows the end-to-end diffusion profile of the gradient wrinkled substrate, for an illumination source of intensity 120 lux.

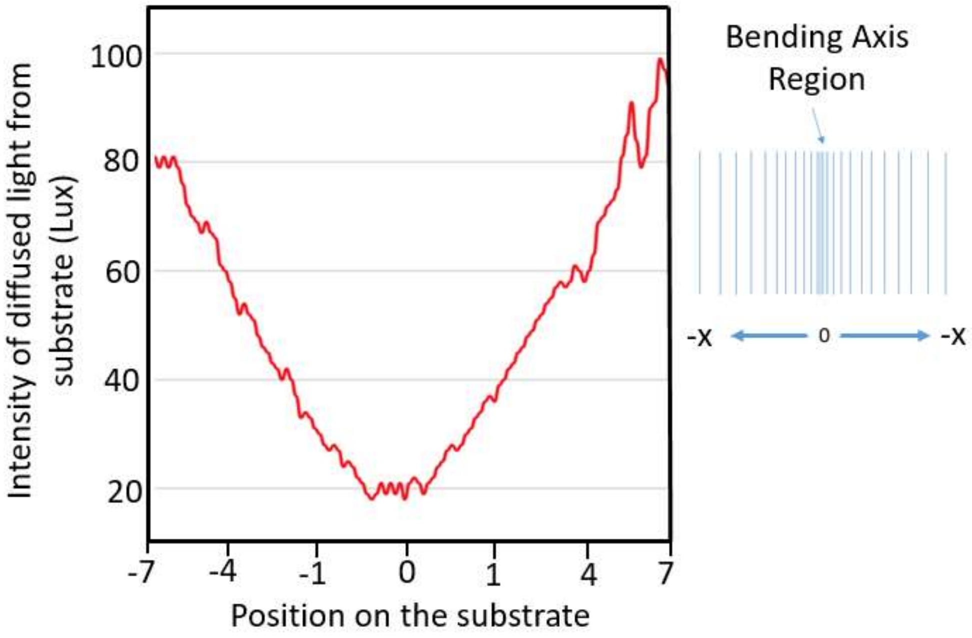

Figure 4.8 Diffusion of light and intensity modulation due to the wrinkles. Moving from one end to another end light scatters most at the bending axis of the substrate.

It can be seen in Figure 4.8 that intensity is maximum at one end and gradually decreases till the bending axis of the substrate, where wrinkle density is maximum (wavelength spacing is minimum). Moving away from the bending axis to either side of the substrate, the intensity of light increases as wrinkles appear increasingly at a greater distance and do not scatter the light to an earlier extent. This arrangement can facilitate the scattering of light in a controlled manner just by sliding the wrinkled substrate in front of the light source. There have been several attempts by researchers to image biological analytes of micrometer size using a cellphone camera.[22,33,34] Earlier reported methods to demonstrate diffusion of light through wrinkles required electrical or mechanical stimulation which is difficult to use in a point care setup.[35,36] Moreover, an important issue that needs to be addressed in photomicrography using a cellphone is the proper illumination of the object. Cellphone backlight can provide direct illumination with a constant intensity in reflection mode, however, it is not suitable for imaging in transmission mode. Stress-induced wrinkle patterned substrates using uniform stress throughout the substrate require additional mechanical arrangements to stretch the substrate for inducing the pattern which is not suitable for point-of-care cellphone photomicrography.[33] Whereas, a gradient surface wrinkled pattern developed in the present study provides a varied range of diffusion of light just by sliding the wrinkle patterned substrate. Such development can also be extended for Light filter and sensor applications.

4.5 CONCLUSION

In the present study, we used an existing understanding of thin film buckling and wrinkling over a complied substrate to fabricate a gradient pitch wrinkled pattern on an elastomeric substrate. A wrinkled pattern was demonstrated to facilitate diffusion of light in a wide range of illumination (80-20 lux) from a given point source of 120 lux. Fabrication of the pattern is relatively simple and does not require any additional mechanical arrangement to generate or release stress in the substrate. Since the substrate is subjected to folding, which developed gradient curvature, strain, and stress in the substrate, and hence wrinkles wavelength created are of gradient spacing. The wavelength of wrinkles increases from the bending axis of the substrate to the end side of the substrate in one direction. For a source illumination of 120lux, diffused light intensity of 100 to 20 lux was obtained using the gradient wrinkled substrate. Such wrinkled patterns can find potential applications as optical diffusers in photomicrography and smart windows etc. and further extension of the work can be directed towards the development of various optical filters and sensors.

4.6 REFERENCES

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