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The Buckling of Metal Thin Films on Soft Elastomers: Use in Flexible Electronics

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Abstract

The deposition of a metal thin film over soft elastomers like PDMS is associated with the distribution of random buckles over its surface. The effective utilization of these sinusoidal buckles in the bending mode of the entire composite structure finds lot of applications as a flexible MEMS sensor. The present article deals with the effective control of these buckles in the successful realization of flexible sensors. The deposition of a nichrome thin film over a non-planar elastomeric surface induces highly ordered buckle structures over the elastomeric ridge. These buckles, aligned parallel to the ridge width, can be used in effective bending of the sensor along its length without resulting in any cracking of the thin film. The fabrication of a non-planar PDMS surface using the soft lithography technique, the deposition of a crack-free nichrome film, and the generation of highly ordered buckles over the non-planar geometry have been discussed in the present paper. The deposition time has also been varied to optimize the buckle wavelength needed for effective bending and the results have been compared with the theoretical values.

1. Introduction

Flexible and stretchable electronic sensors find numerous applications in robotic skin [Lumelsky *et al.*, 2001], flexible displays [Lacour *et al.*, 2004] and various implantable/non-implantable biomedical devices [Hsu *et al.*, 2002]. The major need in all such applications is an elastomeric substrate for the conformable fixation of the fabricated sensor/ electronic device. One such elastomer that is widely used in the development of MEMS sensors for biomedical applications is polydimethylsiloxane (PDMS) due to its unique properties like high flexibility, biocompatibility, chemical and thermal stability, optical transparency and ease of fabrication [Sia *et al.*, 2003]. However the major concern in the fabrication of metallic interconnecting circuits or sensing elements is the successful deposition of metal thin films like nichrome, gold, aluminum, etc., over such polymer surfaces, the successful realization of the desired pattern over the same, and the implantation of the polymer substrate-metal sensor assembly in an effective bending or stretchable condition.

In general, the temperature of the substrate increases to some extent during the thin film deposition process [Bowden *et al.*, 1998, Chua *et al.*, 2000] by the thermal/ e-beam/ plasma/

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sputtering technique due to the accumulation of high energy/ thermally excited cluster of molecules on any substrate surface. This is advantageous for the proper adherence of a thin film over any rigid substrate. However, for a polymer substrate, as the film/substrate system is cooled after the deposition is over, a large mismatch in thermal expansion between the metal and elastomer induces an equi-biaxial compressive stress in the film, resulting in the buckling of the film below a critical temperature [Bowden *et al.*, 1998, Chua *et al.*, 2000, Volynskii *et al.*, 2000, Genzer *et al.*, 2006, Manocha *et al.*, 2010]. These sinusoidal buckles are typically of a few microns in length and are randomly oriented over the flat elastomer surface [Bowden *et al.*, 1998, Chua *et al.*, 2000]. Such a formation of buckles might prove to be useful for flexible electronics applications wherein the buckles would allow for the bending of the metallic sensor over the elastomer without the formation of cracks. However, not only the formation of buckles but also their orientation along the direction of bend is important in realizing their flexible nature. Attempts to realize ordered buckles of deposited metal thin films over elastomeric surfaces have been made possible by the incorporation of a differential stress distribution over the elastomeric surface. It has been shown that the development of non-planar topology over the elastomeric surface using soft lithography, like the formation of ridges [Bowden *et al.*, 1998, Chua *et al.*, 2000], formation of holes [Ohzono *et al.*, 2005] or crosslinking of specific areas [Huck *et al.*, 2000] induce an ordered buckling of the deposited metal thin film over the elastomeric surface. In the present paper, the deposition of a nichrome thin film using the DC sputtering technique and an initial study about the formation of ordered buckling over small ridges of PDMS elastomer are presented. The degree of bend would determine the wavelength and amplitude of the buckle needed which, in turn, can be controlled by varying the thickness of the deposited thin metal film and/or controlling the substrate thickness. Different deposition conditions are being explored to optimize the buckling wavelength needed to realize the flexible sensors.

2. Experiment

Non-planar PDMS molds were prepared using soft lithography over SU8 masters. A nichrome film was deposited using the DC sputtering technique. Details about the generation of PDMS non-planar topology and the formation of buckles under different deposition conditions have also been presented below.

2.1 SU8 Master fabrication

In order to make a non planar topography (square/ rectangular ridges) over the PDMS surface using soft lithography, SU8 master templates having rectangular wells of 40 µm and 100 µm depth with a varying surface area were initially fabricated. Silicon wafers having a resistivity of 1-10 Ù-cm were used as substrates for fabricating the masters. SU8 50 from Microchem was spin-coated over glass substrate in two steps: first at 500 rpm for 10 sec followed by 3000 rpm for 30 sec to obtain 40 µm wells, and at 500 rpm for 10 sec followed by 1000 rpm for 30 sec to obtain 100 µm wells. SU8-coated samples were then prebaked at 65°C for 5 min and 10 min, respectively, followed by softbaking at 95°C for 15 min and 30 min, respectively. Subsequently, the samples were exposed to a UV source for 25 sec and 30 sec for the 40 µm and 100 µm wells, respectively. After developing, the depth of the resulting SU8 wells was measured using a surface profiler (Dektak 150) and the wells were used as master mold for subsequent process to obtain PDMS ridges by soft lithography [Xia and Whitesides, 1998].

2.2 Non-planar topology over PDMS

PDMS samples were prepared by mixing the elastomer (Sylgard 184 from Dow-Corning Corporation, USA) and the curing agent in 10:1 (w/ w) ratio. This was followed by degassing the mix in vacuum for 15 min to remove any bubbles formed during mixing of the two parts. The PDMS mixture was spin-coated over SU8 wells at 500 rpm to obtain a planar surface with SU8 wells filled with PDMS and cured at 120 °C for 2 hrs in a convection oven to ensure complete polymerization. The thickness of the resulting coplanar PDMS layer above the SU8 wells was about 250 ìm as measured by a surface profiler. The PDMS mold was peeled off from the master and placed over a silicon wafer with the ridge side on top. The SU8 wells thus reproduced themselves as PDMS ridges having ridge height of 40 µm and 100 µm with a base PDMS layer of 250 µm thicknesses. Subsequently, a nichrome thin film was deposited over the ridge surface for buckling studies.

2.3 Nichrome Sputtering

Nichrome targets used for the sputtering process, with Ni/Cr - 80/20 wt. % of Ni/Cr composition and 99.95% purity, were obtained from Leico Industries, Inc. The PDMS ridge samples were treated with oxygen plasm2a for 30 sec using (Harrick Plasma, model PDC-32G-2) at 18 W power before being loaded into the sputtering chamber. This ensured better adhesion of the sputtered nichrome thin film [Morent *et al.*, 2007]. Various sputtering conditions like deposition time, deposition power, base vacuum, and working pressure were standardized to obtain good quality, crack-free deposited NiCr thin film over PDMS surface, as discussed elsewhere [Maji *et al.*, 2010]. The initial deposition at a base vacuum of 5×10^{-5} mbar and a working pressure of 0.1 mbar resulted in cracks over the deposited film even for deposition at low power of 60 W for 5 min. Finally, the base pressure was reduced to 5×10^{-6} mbar and deposition was carried out at a working pressure of 0.009 mbar, with the argon gas yielding light purple-colored plasma. Thus, high quality, crack-free nichrome films of thickness up to 0.2 μm for 5 min deposition at plasma power of 60 W over the planar and ridge structure of PDMS layer were effectively produced.

2.4 Buckle formation over planar and non-planar PDMS surface

The thin metal film deposited over compliant surfaces of PDMS causes the development of residual stress due to mismatch of strain between the two materials. As the temperature of the film/substrate falls, the stress increases and above a critical stress, the thin film undergoes buckling into a family of modes with short wavelengths known as buckle wavelength [Bowden *et al.*, 1998]. The deposited nichrome thin film produced randomly oriented buckles over the planar PDMS surface and highly ordered buckles over the ridges, as discussed below and shown in Fig. 1. The theoretical modeling o f the formation of buckles has been discussed elsewhere [Bowden *et al.*, 1998, Volynskii *et al.*, 2000, Manocha *et al.*, 2010]. This model predicts the ordered orientation of the buckles perpendicular to the ridges, unlike the random orientation of buckles on a planar PDMS surface. Moreover, variations of stress profiles along the axial as well as perpendicular directions of ridges have been studied, along with their variation for different ridge widths, and have been reported in the theoretical analysis by Manocha *et al.*, 2010.

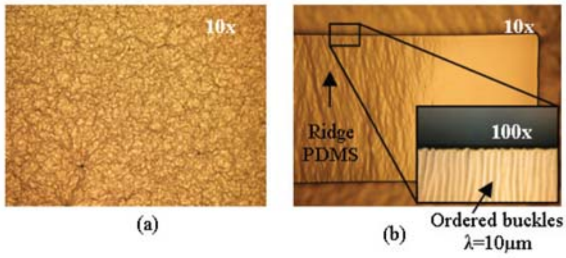

Figure 1. Microphotographs of sputtered NiCr over PDMS surface: (a) Random orientation of buckles over planar PDMS, (b) Highly oriented buckles over PDMS ridges parallel to the ridge width.

The sputtering of nichrome thin films over the PDMS elastomer raises its temperature, causing thermal expansion of the film/substrate assembly. As the temperature of the system drops to room temperature after sputtering, equi-biaxial compressive stress in the film increases, which results in the formation of a random arrangement of buckles measuring few microns, at a critical temperature, s_{crit} given by [Bowden et al., 1998]:

where E_m, E_p, v_m and v_p are the Young's modulus

$$S_{crit} = 0.52\left(E_m/(1-V_m{}^2)\right)^{1/3}\left(E_p/(1-V_p{}^2)\right)^{2/3} \quad (1)$$

and Poisson's ratio for nichrome and PDMS, respectively.

The stress distribution in the film becomes discontinuous when steps are present in a non-planar substrate topology. Instead of equi-biaxial stress, the distribution will be oriented preferably along the direction of the steps, resulting in formation of sinusoidal wave pattern having crests aligned perpendicular to the direction of maximum compressive stress [Bowden *et al.*, 1998], as depicted in Fig. 1. The presence of a ridge prevents the compressive stress acting along the direction of the step, whereas the stress in the direction perpendicular to the step remains almost the same. This exerts a compressive stress along that direction, i.e., perpendicular to the steps, and the associated sinusoidal wave pattern has a buckle wavelength L (m) given by [Bowden *et al.*, 1998]:

$$L = 4.36t\left[E_m/(1-V_m{}^2) * (1-V_p{}^2)/E_p\right]^{1/3} \quad (2)$$

where t (m) is the film thickness. The equation above shows that the buckle wavelength increases with an increase in the thickness of the deposited thin metal film. In order to test the above observation , we fabricated small ridge structures of the PDMS elastomer and deposited nichrome films over the structures for varying time durations, and studied the buckling wavelength.

At the edge of a ridge, the stress starts from zero and gradually increases as one move towards its centre [Bowden *et al.*, 1998]. As the stress gradually increases beyond the critical stress, buckling reappears. This distance, from the step edge to the point where the buckles reappear again over the ridge area, is known as the transition length or buckle-free length.

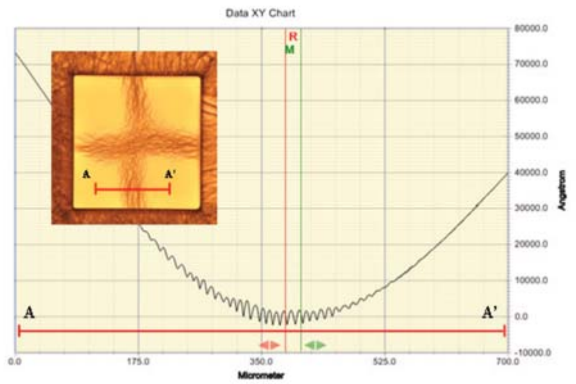

Figure 2. Variations in buckle wavelength and amplitude from the centre of the ridge to the edges, as obtained from a surface profiler.

The transition length l (m) characterizing the distribution of stress from the step is given by [Bowden *et al.*, 1998]:

$$l = 0.3t * \left[E_m/E_p\right] \quad (3)$$

The transition length for the deposited thin film has also been calculated and measured in the present study. It is observed from Eq. (3) that the transition length also increases with the thickness of the deposited metal film which, in turn, depends upon the deposition time. Small square ridges with the edge length varying from 1100 µm to 2300 µm and height *(h)* 40 µm and 100 µm were fabricated investigate the variation of transition length with film thickness. The nichrome sputtering time was varied from 2 to 10 min for ridges with a step height of 40 µm and from 2 to 15 min for ridges with a step height of 100 µm. The buckle wavelengths *(L)* as well as the transition length *(l)* obtained for the square ridges are shown in Table 1. The variations in buckle wavelength from the centre of the ridge to the edges have been plotted using a surface profiler (Dektak 150 with a stylus force of 0.5 mg) and a typical buckle profile over the ridge structure is shown in Fig. 2.

The same experiment was repeated for rectangular ridges having a width varying from 500 µm to 1500 µm and ridge length varying from 3 mm to 12 mm for two different ridge heights (40 µm and 100 µm). The ridge length was varied for effective bending of the samples along its length. The sputtering time for nichrome was also varied from 2 min to 10 min for ridges with step height of 40 µm, and from 2 to 15 min for ridges with a step height of 100 µm. Fig. 3 shows the formation of buckles and the transition length for the deposited nichrome film over samples with a ridge height of 100 µm. The measured buckle wavelengths as well as the transition buckle length are shown in Table

Table 1: Measured buckle wavelength (*L*) and transition region length (*l*) of NiCr thin film for different deposition times.

Deposition time (min)	NiCr Thickness (µm)	Square ridge (h=40µm)		Rectangular ridge (h=40 µm)		Square ridge (h=100µm)		Rectangular ridge (h=100µm)	
		L (µm)	*l* (µm)	*L* (µm)	*l* (µm)	*L* (µm)	*l* (µm)	*L* (µm)	*l* (µm)
2	0.06	6.5	130	7	130	1.5	200	1.5	200
5	0.1	7.5	280	6.7	250	3.5	320	3.5-4	300
10	0.19	9	450	8.5	350	5-7	550	5-6	500
15	0.25	-	-	-	-	11-14	700	8-10	650

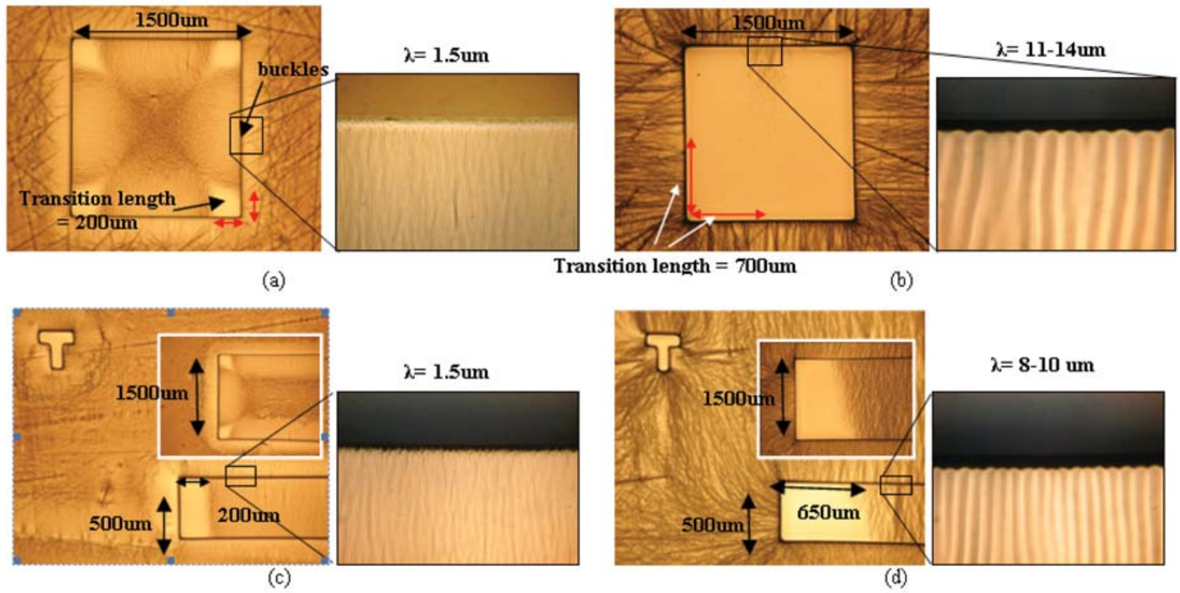

Figure 3. Microphotographs showing variation in buckle wavelength and transition length for the deposition of nichrome over 100 μm height square ridges for (a) 2 min and (b)15 min and, rectangular ridges for (c) 2 min and (d) 15 min. The inset of (c) and (d) shows corresponding rectangular ridges of width 1500 μm.

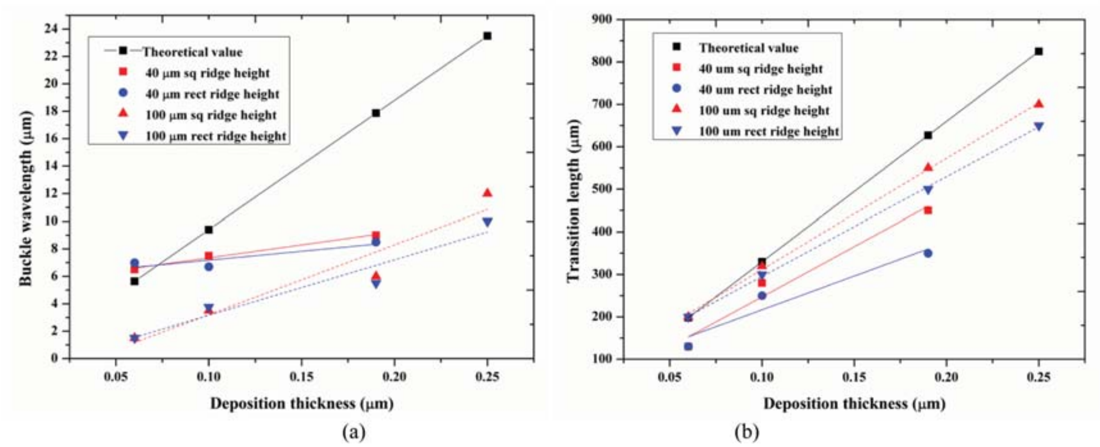

Figure 4. Variation of measured and calculated (a) buckle wavelength and (b) transition length for a film of thickness for 40 μm and ridge height 100 μm, for both square and rectangular ridges.

1. The variation of measured buckle wavelength and transition length with deposited film thickness in Fig.4 and compared with theoretical results.

3. Results and Discussion

It may be observed from Fig.1 that a nichrome thin film deposited over non-planar PDMS ridges showed highly ordered buckle patterns, unlike disordered buckles over planar and unconstrained PDMS surface. It is also observed that the buckles so formed over ridges, after deposition, were highly regular and parallel to each other. They were almost perfectly oriented perpendicular to the ridge length and parallel to the width of the ridge, as shown in Fig. 1. The buckle wavelength varies from 10 μm at the centre of the ridge and increases gradually to 13 μm as it goes towards the edges and finally disappears near the edges. The amplitude of the waves also decreases gradually from about 4700 Å at the centre to 900 Å near the edges, as shown in Fig. 2.

Table 1 shows the measured buckle wavelengths and transition length of the nichrome film deposited over the 40 μm and 100 μm ridge structures. As nichrome sputtering time increased from 2 min to 5 min and finally up to 10 min at 60

W power and working pressure of 0.008 mbar, the transition length, i.e. ,the buckle free region at the edges increased from 130 µm to 280 µm and finally to about 450 µm, thus covering the entire square ridge of dimension 1100 µm. It was also observed for the rectangular ridges that an increase in deposition time from 2 min to 5 min resulted in an increase of the buckle-free region along the edges from 130 µm to 250 µm, respectively. Thus the width of the rectangular ridge (500 µm) was completely buckle-free for a deposition time of 5 min, as the transition length from both the sides covered the entire ridge width. Further increase of deposition time beyond 10 min resulted in complete buckle-free region along the width of the ridge; however, the transition length remains constant at nearly 350 µm along the ridge length.

Fig. 3 shows the microphotographs of nichrome films of two different thicknesses on the square (3a, 3b) and rectangular (3c, 3d) ridge structures of 100 µm step height deposited to study the influence of the structural geometry of the ridge on the buckle wavelength and transition length. It may be observed, by comparing the measured buckle wavelength between Fig 3a and 3b for square ridges as well as for Fig 3c and 3d for rectangular ridge structures, that the buckle wavelength increases with film thickness. The corresponding measured wavelength has been given in Table 1. The results confirm that the buckle wavelength depends mainly on film thickness and not on the ridge structure. Similarly, the measured transition length was about 200 µm and 700 µm as observed from Fig. 3a and 3b, respectively. Since stress distribution at the edge is zero, no buckles are present near the edges. Fig. 3a also indicates that, for a small film thickness, square ridges had buckles distributed over their central portion with gradually increasing wavelength towards the edges, similar to Fig. 2. As the film thickness increases, the transition length also increases from the opposite edges, thus making the entire square ridge of dimension 1500 µm a buckle-free region, as shown in Fig. 3b. Fig. 3c shows a transition length of 200 µm which is sufficient to render the 500 µm rectangular width buckle-free, but not the 1500 µm width (inset Fig. 3c). However, with the increase of film thickness transition length increases to about 650 µm, thus converting the 1500 µm rectangular width into a buckle-free zone, as shown in Fig. 3d. It was observed that the buckle-free region expands not only due to the presence of edges but also due to presence of corners as in the case of squares ridges. This is because corners allow a decrease in stress in both the directions, unlike the presence of an edge which allows a decrease in stress along only one direction, i.e., perpendicular to the edge. The above experimental results show that the buckle wavelength and transition length increase with the increase of film thickness, as predicted by Eq. 2 and 3.

Fig. 4 shows the variation of measured buckle wavelength and transition length with nichrome thickness and compares the results with theoretically calculated results considering the Young's modulus and Poisson's ratio for the nichrome film and PDMS as: E_m = 220 GPa, E_p= 20 MPa [Bowden *et al.*, 1998], v_m = 0.325 and v_p = 0.48, respectively. The results show a consistent increase in buckle wavelength and transition length with increasing film thickness. It may be observed from Fig. 4 that the values of the measured and calculated buckle wavelength and transition length are almost equal at low film thickness, but deviate considerably as film thickness increases. Thus might be attributed due to an error in the Young's modulus and Poisson's ratio considered for calculations.

The above theoretical and experimental observations predict not only the regular orientation of the spontaneously formed parallel buckles, but also helps in estimating the variation in buckle wavelength with film thickness and ridge height. The results would be useful in flexible electronics/ sensor applications, where such ordered buckles would help in bending along the direction of ridge, by allowing for the stretching incorporated during bending [Maji *et al.*, 2012]. The proposed sensor structure in this case can be fabricated over the buckles ordered in parallel atop the ridge structure. Further research is currently being carried out to utilize this concept in the development of a catheter tip-mounted flow sensor for healthcare delivery [Maji *et al.*, 2013].

4. Conclusion

Non-planar structures over the PDMS elastomer were successfully fabricated by the soft lithography technique using SU8 master templates containing square/ rectangular wells. The SU8 wells of 40 µm and 100 µm depth reproduced themselves as ridges over the PDMS mold after soft lithography. Nichrome was e deposited at a base vacuum of 5×10^{-6} mbar and working pressure of 0.009 mbar to obtain high quality, crack-free films over the ridge structures of PDMS. Successful orientation and manipulation of ordered buckles in nichrome thin films over non-planar PDMS film has been achieved in this study. An increase in buckle wavelength and transition length with the increase of film thickness has been successfully observed in the experimental results, as predicted by the theoretical equations. Further studies and experiments are being conducted to optimize the proper buckling wavelength needed for the effective bending/ wrapping of the sensor for flexible electronics applications.

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