Thin film properties by blister tests

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Abstract. With the measured geometry morphologies of telephone cord blisters (TCBs), that is, the local blister width and height, and the global wavelength, a mechanical model is built to determine the residual compressive stress in thin films and interface adhesion toughness. This model is based on the hypothesis that pockets of energy concentration (PECs) drive the nucleation and growth of TCBs in thin films under the constant residual stress. The model is validated with independent experimental measurements and shows the strong potential to guide the fabrication of micro-patterns on thin films.

Keywords: Telephone cord blisters, Pockets of energy concentration, Residual stress, Interface adhesion toughness, Spallation

1 Introduction

Thin solid films are built in engineering applications fulfilling various functions, such as confinement of electric charge in integrated electronic circuits, and in thermal insulation in thermal barrier coatings. Although these thin films are not primarily designed to withstand mechanical loads, their mechanical properties are usually major concerns since the desired functions may be lost as the result of the delamination of thin films driven by buckling [1–4] or pockets of energy concentration [5–9]. Under the residual stress, circular blisters, straight blisters, telephone cord blisters or more complex hexagonal blisters are observed in thin films. It is however, difficult to accurately measure the mechanical properties in experiments, such as the residual compressive stress before delamination and the interface adhesion toughness. The present work aims to develop a mechanical model to determine the residual compressive stress and fracture toughness in thin films with the measured TCB morphologies, that is, local blister width and height, and the global wavelength. The mechanical model is presented in Section 2, and the experimental validation is in Section3. Conclusions are drawn in Section 4.

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2 Mechanical models

The mechanical model to determine the film mechanical properties, i.e., residual compressive stress σ_0 and the interface adhesion toughness G_c is presented in this section. Fig. 1 shows a TCB in a film with the thickness h. The substrate thickness is assumed so large that its global deformations due to the residual stress in the film are neglected. The film material has Young's modulus E and Poisson's ratio ν and is assumed to be homogeneous and isotropic. In Fig. 1, the TCB geometry is described by the local sinusoidal blister width $2R$ and blister height A_x, and the global wavelength λ and transverse amplitude A_y. Note that in Fig. 1b, R is perpendicular to the sinusoidal centreline of the TBC whilst $\tilde{R}$ in Fig 1c is in the direction of the global x.

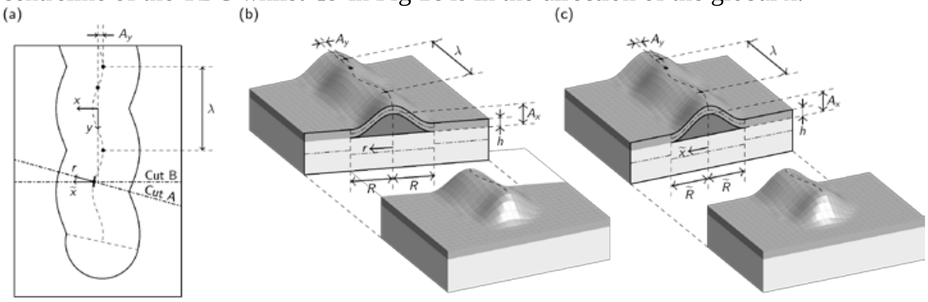

Fig. 1. A telephone cord blister. (a): Top view. (b): 3D view of cut A. (c): 3D view of cut B.

Based on the PECs theory in Ref. [5–7]. A TCB consists of a partial straight blister in fully-developed at some distance from the TCB tip, and a partial circular blister in non-fully-developed close to the TCB tip. The conditions for a TCB forms and propagates are: (1) $\Omega \geq 1.5$ where Ω represents the ratio between the in-plane residual strain energy density in films and the adhesion toughness at the film/substrate interface. The Ω determines the blister growth and is given by,

$$\Omega = \frac{1}{2}\bar{\varepsilon}_0\varphi_0 = \frac{hE\left(\bar{\varepsilon}_0\right)^2}{2\left(1-\nu^2\right)G_c} \geq \frac{3}{2} \tag{1}$$

where $\varphi_0 = h\sigma_0/G_c$ and $\bar{\varepsilon}_0 = \sigma_0\left(1-\nu^2\right)/E$ is the residual compressive plane strain in the plane strain condition. (2) For the partial circular blister, its blister energy denoting the net energy stored in the blister is required to reach the maximum level, called blister capacity before which is supplied by PECs. (3) For the partial straight blister, its half width is required to reach the level to trigger the secondary buckling, leading to the waviness of TCBs.

The present work aims to obtain the mechanical properties, i.e., the in-plane residual compressive stress σ_0 before delamination and the interface adhesion toughness G_c. Two methods are to calculate Ω first: (1) Based on the measured blister height A_x,

$$\Omega = 3\left(\frac{A_x}{8h} + \frac{h}{A_x}\right)^2 \tag{2}$$

(2) Based on the measured TCB wavelength-to-width ratio $r = \lambda/(2R)$,

$$\Omega = \frac{3}{2}\left[1 - \left(\frac{\nu - 1}{\nu + r^2}\right)^2\right]^{-1} \tag{3}$$

Then with the local TCB half width R, the ratio φ_0 can be obtained by

$$\varphi_0 = \frac{6R^2}{\pi^2 h^2}\left[1 + \left(1 - \frac{3}{2\Omega}\right)^{1/2}\right]^{-1} \tag{4}$$

Next, the residual strain $\overline{\varepsilon}_0 = 2\Omega/\varphi_0$, residual stress σ_0 and interface fracture toughness $G_c = hE(\overline{\varepsilon}_0)^2/\left[2(1-\nu^2)\Omega\right]$ are evident to work out from Eq. (1).

3 Experimental validations

The mechanical model is validated with two independent experimental measurements from Refs. [10,11]. The first comparison is against the measurements in Ref.[10] where the thin film is tantalum and the substrate is glass. The film thickness is $h = 0.225\,\mu\text{m}$. The Young's modulus and Poisson's ratio of the film are $E = 175\,\text{GPa}$ and $\nu = 0.3$, respectively. With the measured TCB width, height, and the wavelength, the quantity Ω is initially worked out from Eq. (2) then the mechanical properties are obtained from Eqs. (1) and (4). Comparisons between the predictions for the mechanical properties and the measurements are presented in Table 1, and the measurement locations are shown in Fig. 2.

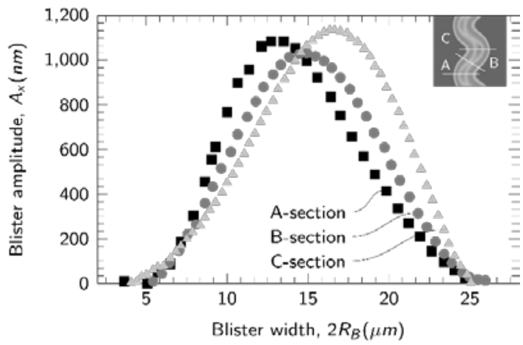

Fig. 2. Telephone cord blister morphology measurement [10].

Table 1. Comparisons between theoretical predictions and experimental measurements [10].

Experimental measurements			Theoretical predictions			
A_x [μm]	λ [μm]	R [μm]	Ω Eq. (2)	$\bar{\varepsilon}_0$ Eq. (1)	σ_0 [GPa]	G_c $[\mathrm{J\,m^{-2}}]$ Eq. (1)
Section A						
1.09	20.00	10.00	1.9779	0.49%	0.94	0.2641
Section B						
1.01	20.20	10.10	1.8434	0.43%	0.83	0.2179
Section C						
1.14	20.60	10.50	2.0702	0.48%	0.92	0.2376
Averaged						
1.08	20.40	10.20	1.9602	0.47%	0.90	0.2395

From Table 1, the Ω in all sections are larger than 1.5, and the predicted residual stresses have excellent agreement with the measurements, that is, 1 GPa reported in Ref. [10]. The measured interface fracture toughness is unavailable; however, the predicted values at three locations are consistent with each other which is more expected. Hereby, taking the same definitions for the mode I and II critical interface adhesion toughness in Ref [12], that is, $G_{\mathrm{Ic}} = 0.176u_0 = 0.1584\,\mathrm{N\,m^{-1}}$ and $G_{\mathrm{IIc}} = 28.5u_0 = 25.6500\,\mathrm{N\,m^{-1}}$, respectively, where $u_0 = (1-\nu)h\sigma_0^2 / E$ describes the residual train energy density. The fracture toughness G_{c} based on the mixed fracture mode partitions for classical plates, shear-deformable plates, and 2D elasticity theories are $0.1584\,\mathrm{N\,m^{-1}}$, $0.6221\,\mathrm{N\,m^{-1}}$ and $0.2534\,\mathrm{N\,m^{-1}}$. It is interesting to find the interface adhesion toughness from 2D partition theory agrees well with the results shown in Table 1 that based on the TCB morphologies. This demonstrates that the present PECs theory captures the real mechanics of TCBs.

The second comparison is against the experimental measurements in Ref.[11] where the thin film is tungsten and the substrate is silica. There are four groups of specimens with different film thicknesses. The Young's modulus and Poisson's ratio are taken to be $E = 411\mathrm{GPa}$ and $\nu = 0.28$, respectively. The quantity Ω is firstly worked out from Eq. (3) then the mechanical properties are obtained from Eqs. (1) and (4) based on the measured R and A_x, at three locations (see Fig. 3). The theoretical results and comparisons are recorded in Table 2.

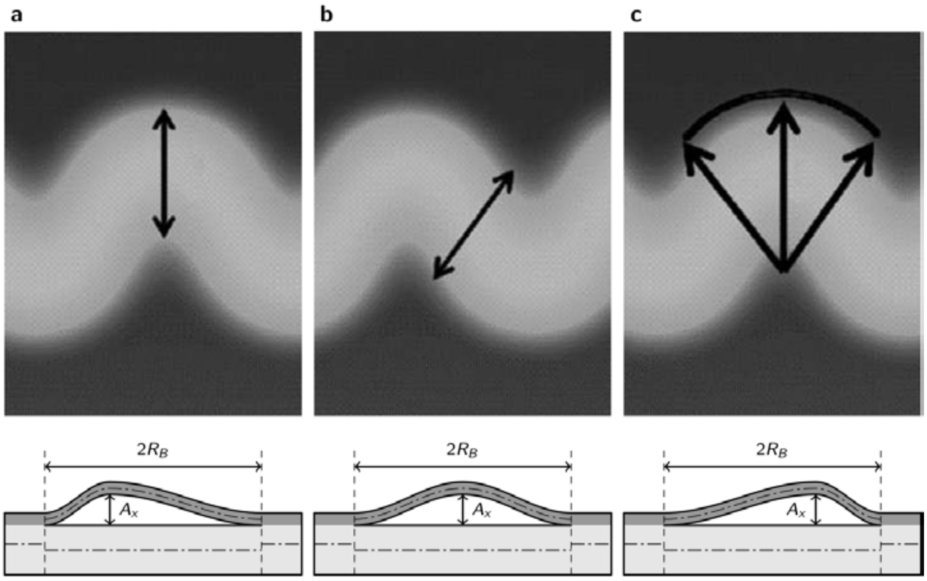

Fig. 3. Telephone cord blister morphology measurements at three different locations[11]. (a), Section A. (b), Section B, (c), Section C. A sketch of the cross-section is shown below each.

Table 2. Comparison between theoretical predictions and experimental measurements [11].

Experimental measurements [11]			Theoretical predictions			
h [nm]	R [μm]	A_x [μm]	Ω Eq. (3)	$\bar{\varepsilon}_0$ Eq. (1)	σ_0 [GPa]	G_c [N/m] Eq. (1)
100						
A	8.110	0.847	4.15	0.37%	1.67 [3.14]	0.08 [1.180±0.400]
B	7.400	0.787	3.70	0.39%	1.76 [3.07]	0.09 [1.130±0.250]
C	8.100	0.847	4.15	0.37%	1.67 [2.39]	0.08 [0.621±0.080]
AVG	7.870	0.827	4.00	0.38%	1.70 [2.87]	0.08 [0.980±0.280]
200						
A	5.400	0.670	1.54	0.75%	3.63 [4.75]	1.91 [6.010±0.900]
B	5.160	0.633	1.52	0.83%	3.72 [4.70]	2.05 [5.960±0.900]
C	5.400	0.670	1.54	0.81%	3.63 [3.74]	1.91 [2.930±0.420]
AVG	5.320	0.658	1.53	0.82%	3.66 [4.40]	1.96 [4.970±0.770]
225						
A	17.500	1.430	2.72	0.25%	1.10 [1.91]	0.11 [0.988±0.150]
B	17.200	1.360	2.54	0.24%	1.05 [1.82]	0.11 [0.897±0.100]
C	17.450	1.430	2.72	0.25%	1.11 [1.49]	0.11 [0.512±0.080]
AVG	17.383	1.407	2.66	0.24%	1.09 [1.74]	0.11 [0.799±0.110]
300						
A	20.000	1.730	2.40	0.29%	1.28 [2.14]	0.23 [1.670±0.200]
B	18.800	1.470	2.00	0.25%	1.12 [2.18]	0.21 [1.800±0.200]
C	20.000	1.730	2.40	0.29%	1.28 [1.64]	0.23 [0.862±0.120]
AVG	19.600	1.643	2.26	0.27%	1.22 [1.99]	0.22 [1.444±0.180]

It is observed that all Ω values in Table 2 are above 1.5, which gives the formation condition of TCBs, that is, $\Omega > 3/2$. It is worth noting the values of Ω for the second group of specimens rigorously obey the rule. The predictions for the residual stresses

σ_0 and the fracture toughness G_c shown in brackets are according to the traditional buckling approach [13], and the corresponding results in sections A and C in each group are completely different with each other; however, those from the current PECs theory are very close to each other, which is expected in reality. This convincingly demonstrates that the present theory captures the real mechanics of TCBs.

4 Conclusion

Telephone cord blisters are generated as an instability consequence under the constant residual stress in thin films. As per the hypothesis that pockets of energy concentration drive the nucleation and growth of TCBs, with the measured morphologies of TCBs, that is, the local blister width, height and the global wavelength, a mechanical model is built to effectively determine the mechanical properties of thin films, that is, the residual compressive stress before blistering and the interface adhesion toughness. For the TCBs growth, it is also verified Ω representing the ratio between the residual strain energy density and the interface adhesion toughness is required to be larger than 1.5.

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