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Slippage toughness measurement of soft interface between stiff thin films and elastomeric substrate

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Traditional interfacial toughness measurements for thin films on substrate are not appropriate to the structure composed of stiff films and soft substrate. This paper describes a new bending test system to measure the interfacial toughness for the soft interface between stiff films and elastomeric substrate. The experimental setup including the loading stages is easy to operate and scanning electron microscope is used to in situ monitor the interfacial slippage during loading. The proposed bending test is conducted for silicon film on poly(dimethylsiloxane) substrate. This method demonstrates the promising way to measure the slippage toughness of soft interface involving the flexible electronics and the bio-related fields. © 2011 American Institute of Physics. [doi:10.1063/1.3646461]

I. INTRODUCTION

Flexible electronics with innovative design, that releases the restricts of the rigid, brittle, and planar nature to enable new applications, will eventually change the traditional microelectronics and semiconductor industry. Organic electronic materials are promising for flexible electronics because of their excellent mechanical deformability.1 However, it is very difficult to obtain high performance such as electronic mobility, which restricts the range of their application.3 On the other hand, traditional inorganic electronic materials possess attractive performance, but cannot be subjected to large mechanical deformation. Therefore, the stretchable and flexible electronics with the structure of inorganic films on compliant substrate have recently attracted the increasing interest.5,6

Flexible electronics is inevitably subject to repeatedly bending and stretching deformation that may induce interfacial failure and material fracture. Therefore, it is very important to understand the failure mechanism of the system. There are a few techniques to measure the interfacial toughness for stiff thin films on stiff substrates.7 For instance, superlayer test based on residual stress-induced delamination gives accurate adhesion energy values.8,9 However, the stress-induced energy release rate is usually too small to debond. Indentation can be used to delaminate the brittle, weakly bonding thin film from stiff substrate and provides interfacial toughness measurements over a wide range of phase angles.10–12 Four- or three-point bending test has the initial lengths $L$ of substrate. The soft substrate buckles to an end-to-end length of $L - dL$ when it is subject to compression applied by the free baffle of the bending stage, shown in Fig. 2(a). Based on Dai’s theoretical results, we proposed a new method and the experimental setup to measure the slippage toughness for the soft interface between stiff films and elastomeric substrate. The setup with loading system can be put into the chamber of scanning electron microscope (SEM) and then the interfacial slippage displacement is in situ monitored during loading.

II. EXPERIMENTAL SETUP AND MEASUREMENT PRINCIPLE

Figure 1 schematically illustrates the proposed setup and method of bending test. The bending stage is composed of a flat base, a fixed baffle, and a free baffle in the horizontal direction. The sample is placed between two baffles. When the free baffle moves forward driven by the screw, the sample will be compressed and then buckle into upward deflection because of its extremely low critical buckling stress resulting from the very low modulus (~MPa or less) of substrate. Therefore, the top surface of elastomeric substrate subjected to tensile strain can drive the stiff films slipping on the interface. If slippage occurs, SEM will record the location of thin films and the slippage displacement while the loading is spontaneously recorded via the bending stage. A theoretical model can be developed to connect the loading and the slippage toughness.

Before applying the loading, the substrate with the ribbons has the initial lengths $L$. The soft substrate buckles to an end-to-end length of $L - dL$ when it is subject to compression applied by the free baffle of the bending stage, shown in Fig. 2(a). Based on the buckling theory, the profile of the buckled substrate shown in Fig. 2(b) can be given by

$\frac{d}{L} = \frac{\pi}{4}\sqrt{\frac{1}{4} - \left(\frac{d}{L}\right)^2} - \frac{1}{24}\left(\frac{d}{L}\right)^3$
FIG. 1. (Color online) Schematic illustration of the experimental setup. (a) The setup before applying loading; (b) the setup after applying loading; and (c) the slipping region can be in situ observed by SEM.

\[ w = w_0 \sin(\pi x/L) \] (Ref. 19) with

\[ w_0 = \frac{2}{\pi} L \sqrt{\frac{dL}{L} - \frac{\pi^2 h_s^2}{12L^2}}, \] (1)

where \( dL/L, h_s, w, \) and \( w_0 \) denote the applied strain, the substrate thickness, the deflection of the substrate in the \( z \) direction, and the deflection of the substrate at the center, respectively. The corresponding bent radius of the structure is \( R = L^2/\pi^2 w_0. \)

Actually in the slipping region shown in Fig. 2(c), the normal component of displacement is continuous but the stress is discontinuous at the interface. In the un-slipping region, the film and substrate perfectly bond together, therefore, both of the displacements and stresses are continuous. With the assumption of curvature continuity, the energy release rate can be calculated as\(^{18}\)

\[ G = \frac{1}{2E_s} N^2 \left[ \frac{t \eta}{1 + t \eta} + \frac{12}{1 + t \eta^3} \frac{w_0^2}{h_s^2} - \frac{12}{(1 + t \Sigma) \Delta_3} \right] \times \left( \frac{w_0}{h_s} + \frac{\Delta_1}{2} \right)^2, \] (2)

where \( \eta = h_f/h_s, \ t = \tilde{E}_f/\tilde{E}_s, \ N = \frac{\tilde{E}_s \pi^2 h_s^4}{12L^2}, \ \Delta_1 = \frac{t n(1 + n)}{1 + t \eta}, \ \Sigma = \Delta_2/\Delta_3, \ \Delta_2 = \eta \left[ \eta^2 + \frac{3}{(1 + t \eta)^3} \right], \ \Delta_3 = 1 + 3 \left( \frac{t n(1 + n)}{1 + t \eta} \right)^2, \ h_f \) is the thickness of films, and \( \tilde{E}_f \) and \( \tilde{E}_s \) are the plane-strain modulus of films and substrate, respectively. Therefore, the critical energy release rate can be obtained by substitution of Eq. (1) into Eq. (2), where \( dL/L \) is the input parameters measured by the bending stage. Therefore, slippage toughness can be measured by the critical energy rate with respect to interfacial slippage based on fracture mechanics. The slippage on the interface will occur when the energy release rate is larger than the critical value (e.g., slippage toughness).

III. THEORETICAL RESULTS AND FINITE ELEMENT METHOD (FEM) VALIDATION

The Si nanoribbons on poly(dimethylsiloxane) (PDMS) substrate is considered here, which represents the stiff film–soft substrate system widely used in inorganic flexible electronics. Figure 3 shows the energy release rate of such soft interface versus the applied strain \( dL/L \) for Si nanoribbons with different thickness. The analytical results (solid line) based on Eq. (2) are compared to the FEM simulation (dashed
line. In the calculation, specific parameters $E_f = 130$ GPa, $v_f = 0.27$, $E_s = 2$ MPa, $v_s = 0.45$, and $L = 12.80$ mm are used. It can be seen that the energy release rate increases approximately linearly with increasing of the applied strain $dL/L$. For FEM simulation, the un-slipping interface is set to tie together. The cohesive element is used to simulate the soft interface between Si ribbons and PDMS substrate. An interfacial crack is defined and energy release rate is output directly from the FEM results. It is found that the analytical results are consistent well with the FEM when the substrate thickness is 100 nm. However, the difference between theoretical results and FEM simulation will become significant as the substrate thickness and applied strain increase. Actually, the conventional FEM method is not proper to simulate the model even with cohesive element for soft interface because of the severe mismatch of stiffness and deformation between film and substrate. The FEM elements of the substrate with very low modulus near the crack tip will distort seriously and then the calculation loses its accuracy. However, the above mentioned theoretical model is based on the energy method, in other words, derived from the basic idea of Griffith energy criteria. Therefore, it can substantially avoid this problem and give accurate results for the fracture of soft interface.

IV. EXPERIMENT RESULTS AND DISCUSSION

The Si films are prepared by microfabrication and then are integrated with PDMS substrate by transfer printing method.\textsuperscript{5,20--22} Wet etching with KOH through a photoresist mask defines the Si film into dogbone-like ribbons with thickness of 100 nm, widths of 5 μm, and lengths of 200 μm as well as square pads (∼120×120 μm) at the ends of the ribbons. And then the structure is immersed into dilute hydrofluoric (HF) acid to completely etch off the SiO2 layer under the lengths of the ribbons but only partially removes this layer under the pads. The elastomer polydimethylsiloxane (PDMS; Dow Corning) stamp with 1 mm thickness and 12.8 mm length contacts with the ribbons and then is peeled away. The Si nanoribbons are removed from the substrate through fracture at points near their ends, and then to be integrated to the elastomer. Then the sample is placed on the designed bending stage within the SEM chamber for the measurement. After the loading is applied, the substrate buckles upward and then transfers the tensile stress to the Si ribbons. SEM is \textit{in situ} monitoring the slipping behavior of ribbons.

Figure 4 shows SEM image of silicon ribbons slipping on the PDMS substrate. When the applied strain reaches 4.69%, slippage occurs and the shadow of slipping region with the length of $dl$ can be observed, shown in Fig. 4(a). Therefore, the critical energy release rate can be calculated by Eq. (2) as $G = 0.31$ J/m$^2$. If the applied strain $dL/L$ increases, the ribbons continue to slip on the interface, and the length of slipping region accordingly increases, shown in Fig. 4(b). Through this way, we cannot only record the initial slipping but also the evolution of slipping region till the ribbons delaminate from the substrate. Therefore, the interfacial toughness can be obtained.

A series of samples are carried out to further reveal the slipping behavior of the interface and the corresponding results are presented in Fig. 5. The horizon axial is the applied strain, the right column is the slipping region length $dl$ (e.g., slipping distance), and the left column is the energy release rate $G$. The symbol line with square represents the relation of the average slipping distance $dl$ and the applied strain $dL/L$, and the symbol line with circle shows the energy release rate $G$ versus the applied strain $dL/L$. It can be seen that the slipping distance $dl$ increases with the increasing of the applied strain, and eventually approaches to the saturated value that means the ribbons delaminate from the substrate. The

FIG. 4. SEM image of Si ribbons slipping on PDMS substrate. (a) $dL/L = 4.69\%$, $dl = 1.41 \mu m$ and (b) $dL/L = 9.38\%$, $dl = 2.40 \mu m$.

FIG. 5. (Color online) Energy release rate and the slipping distance versus the applied strain. Symbol lines with square and circle represent the slipping distance and the corresponding energy release rate from experiments, respectively.
energy release rate also monotonously increases with the applied strain. We can find the soft interface can still resist the slippage although the slippage occurs. The interface toughness will increase until the ribbons totally debond from the substrate. In such way, the soft interface can adjust the deformation to increase the slippage resistance ability.

V. CONCLUSION

In this paper, a new method is proposed to measure the interfacial toughness for soft interface between stiff film and elastomeric substrate. The experimental setup including the loading stages is easy to operate and SEM is used to in situ monitor the interfacial slippage during loading. The test is conducted for Si ribbons on PDMS substrate and the slippage toughness is measured. The experimental results show the soft interface can still resist the slippage even if the slippage occurs and the soft interface can adjust the deformation to increase the slippage resistance ability, which is different from the case of elastic interface between stiff materials.

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