COMBINATORIAL APPROACH TO THE EDGE DELAMINATION TEST FOR THIN FILM RELIABILITY --- CONCEPT, RESULTS AND VARIABILITY

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Introduction

In this study, a high-throughput combinatorial approach to edge delamination test is proposed to map the failure of adhesion as a function of both temperature and film thickness in a single step. In this approach, a single specimen of a thin film bonded to a substrate with orthogonal thickness and temperature gradients is subdivided into separate samples. This approach can be adopted to measure the adhesion reliability for films with thickness in the sub-micron range by the addition of an overlayer. Also, through the study of the surface energy effect on the adhesion of a film/substrate system, the variability of the approach for constructing the reliability distribution of the adhesion (as a function of other material parameters) has been demonstrated.

Combinatorial Approach to the Edge-Delamination Test

In the proposed combinatorial edge delamination test, a film is coated onto a relatively rigid substrate in such a way that the film has a thickness gradient in one direction (Fig. 1a). The film is diced into a square grid pattern to form an array of individual edge delamination samples on the substrate (Fig. 1b). The cut penetrates some distance into the substrate also. The edges are at 90° to the interface of the film/substrate. The depth (d) and width (w) of the cut (Fig. 1c) are the design parameters that need to be optimized and have been discussed in a previous publication [1]. Due to the existence of residual biaxial stresses during the solidification of the film and the stress-free edges after dicing in a bi-material system, stress concentrations arise at the interface near the free edges. These stress concentrations are sufficient to create small initial interfacial flaws at the film/substrate boundary. This is the well-known free-edge effect that is unique to bi-material systems [2-4]. Coupled with an interface having finite adhesion strength, these initial flaws are the nucleation sites for interfacial debonding after a further loading. To introduce the further loading, the specimen is cooled with a temperature gradient applied in the direction orthogonal to the thickness gradient (Fig. 1a). Interfacial debonding events will be observed for those samples having critical stresses that depend on the combination of local temperature and film thickness. Consequently, a failure map as a function of temperature and film thickness can be constructed with one step, as shown in Fig. 1a. In principal, if the adhesion of a film to a substrate is independent of temperature, the adhesion can be deduced from this failure map as long as the thermo-mechanical property (the stress-temperature relation) of the test film is well characterized [5,6].

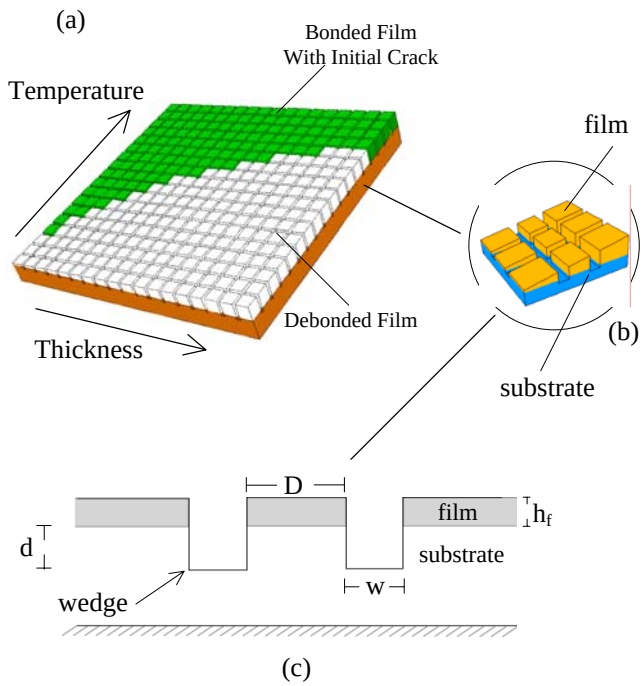

Figure 1. A schematic of the combinatorial approach to the edge delamination test: the multivariant specimen with film thickness and temperature gradients, and final failure map (a); a square pattern array of individual edge delamination samples on the substrate (b); the cutting depth, d, and width, w (c).

Sometimes, especially for large film thickness, the residual stresses (or internal energy) resulting from the solidification of the test film on a substrate (the film preparation step) could be large enough to cause premature failure of the film or interface before further cooling in the edge delamination test [5]. Conversely, it could be the case that a film has such a strong bond with the substrate that the stress concentration generated during the cooling process

is insufficient to induce debonding. In either case, by adjusting the film thickness and the upper and lower limits of temperature, one can ensure that the debonding condition will be met.

To measure the adhesion for films with thickness in sub-micron region, instead of coating a test film with a thickness gradient (Fig. 1a), a test film with a very small constant thickness is coated onto the substrate. Then, an overcoating layer (stress-generating layer) with a thickness gradient is deposited on the top of the thin test film (Fig. 2). The rest of the experimental procedures are identical with the original one. The thickness of the overcoating layer needs to be much larger than that of the test film such that the debonding energy contributed from the test film during the cooling can be neglected, and only the overcoating layer serves as the stress-generating layer. One assumption in this modified approach is that the bonding strength between the test thin film and the overcoating layer is much higher than the bond between the test film and substrate. In this case, the stress-temperature relation of overcoating is only needed to calculate the adhesion. Consequently, one may use this modified combinatorial approach to obtain the critical bond energy for the thin film in the sub-micron range. It is worthwhile to note that once a well-characterized overcoating layer has been chosen, it can be used as a standard overcoating layer for different test films as long as a good adhesion exists between the overcoating and the test film.

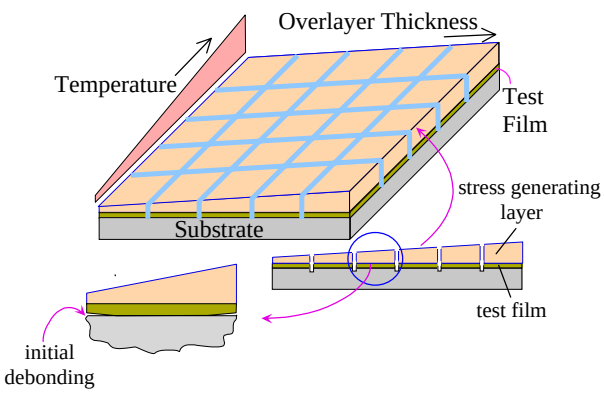

Figure 2. A schematic of the combinatorial approach to the modified edge delamination test: the multivariant specimen with constant film thickness, overcoating layer thickness and temperature gradients.

Results and Discussion

Based on suggestions on geometric requirements for the proposed approach [1], we prepared a combinatorial specimen using silicon as the substrate and a commercial epoxy as the test film. To introduce the loading, one side of the specimen was dipped into the liquid nitrogen for 15 min to form a temperature gradient from -180 ^{o}C to -120 ^{o}C (the standard uncertainty is 2 ^{o}C). Afterwards, interfacial debonding events were visible with the un-aided eye for those samples having the critical relationship of stress and temperature (Fig. 3). This preliminary test result demonstrates feasibility for the proposed combinatorial edge delamination test for thin film adhesion measurement. Also, test result from using PMMA as the test film and the commercial epoxy as the overcoating further demonstrates that this approach is very promising (Fig. 4). The test film thickness was 10 nm. The overcoating layer thickness varied from 40 μm to 220 μm (the standard uncertainty is 5 μm). The contrast in the photograph of the figure (Fig. 4a) is due to the reflected light. Next, again one side of the specimen was dipped into the liquid nitrogen (Fig. 4b) for 15 min to form a temperature gradient from -180 ^{o}C to -120 ^{o}C (the standard uncertainty is 2 ^{o}C). Finally, interfacial debonding for those edge delamination samples having critical stresses can be observed by eye (Fig. 4c).

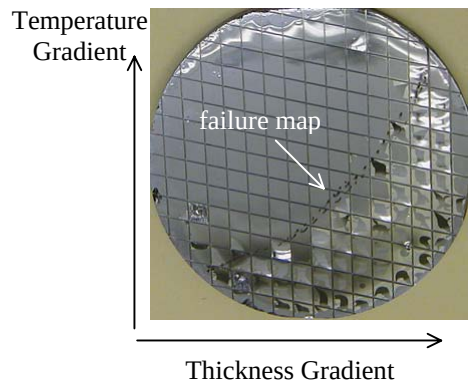

Figure 3. Test result from a combinatorial specimen using silicon as the substrate and a commercial epoxy as the test film.

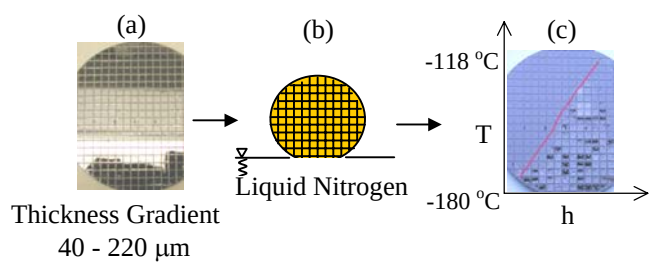

Figure 4. The experimental result of the combinatorial edge delamination test for PMMA adhesion to silicon substrate.

In order to demonstrate the variability of the proposed combinatorial methodology, a single specimen of a thin PMMA film bonded to a substrate with orthogonal film

thickness and contact angle gradients was subdivided into separate samples. The contact angle gradient on the substrate was introduced by modifying the contact angle of a self-assembled monolayer (SAM) generated on the substrate. Fig. 5a shows the variation of the film thickness (h_f) along the X-axis of the specimen for the combinatorial edge delamination test, while Fig. 5b displays the variation of the contact angle (θ_c) along the Y-axis of the specimen. The solid lines are the curve-fittings to the experimental measurement (symbols). The film thickness ranges from 3.66 μm to 9.11 μm over a 30 mm distance. θ_c ranges from 23.1° and 68.3° over a 50 mm distance. Within experimental uncertainties, the results in the figure indicate that both the orthogonal thickness and contact angle gradients have a linear variation within the specimen. After cooling the specimen having 104 individual edge delamination samples, debonding events were observed for those samples having the critical relationship of the stress and adhesion as shown in Fig. 6a. By tracing the locus of far-field debonding, a failure map as a function of film thickness and contact angle was constructed. Fig. 6b gives the quantitative information of the critical relationship between the h_f and θ_c.

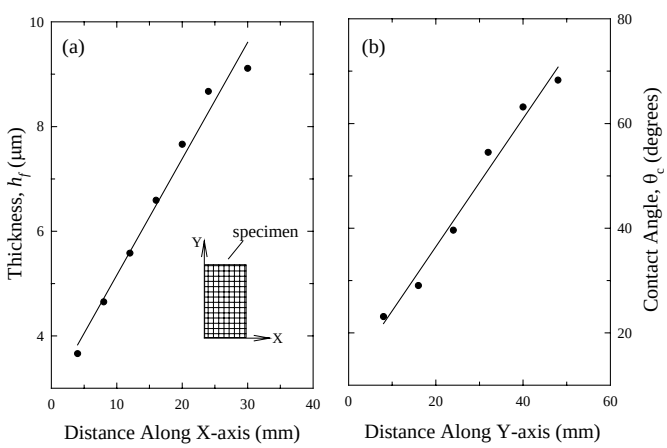

Figure 5. Variation of the film thickness along the X-axis of the combinatorial edge delamination specimen (a), variation of the contact angle along the Y-axis (b).

Conclusions

By combining variables that are important and readily controllable in practice (temperature and film thickness), the effect of stress concentration on the debonding of the film from the substrate is spatially varied in one experiment. Consequently, the failure map of the adhesion as a function of both film thickness and temperature can be constructed in a single step. Also, by applying a constant temperature field to a combinatorial specimen with orthogonal film thickness and contact angle gradients, we demonstrate the variability of the approach for evaluating the adhesion as a function of contact angle in a single step. These results can be used to predict the reliability of adhesion as a function of film thickness and temperature for each specific contact angle. The detailed analysis will be reported in our future publications.

References

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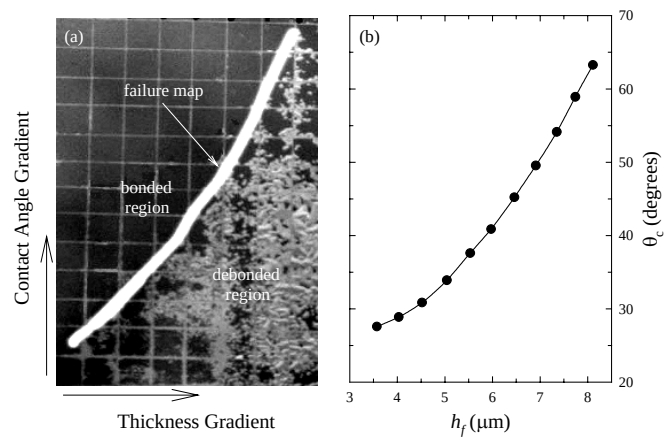

Figure 6. Debonding events in a typical combinatorial edge delamination specimen having PMMA film bonded to the silicon substrate (a); the variation of contact angle with the film thickness along the failure map (b).