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Combinatorial library designs for quantifying thin film adhesion via the edge delamination test

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Abstract
We have demonstrated a combinatorial thin film library design for investigating the adhesion between film and substrate as measured by the edge delamination test. This library design allows rapid screening of critical parameters that control thin film reliability and bond strength in films and coatings. Specifically, our library design was aimed at quantifying the effect of film thickness and composition on the interfacial integrity between the film and underlying substrate by applying thermal stress. To create the combinatorial library, a single specimen was fabricated having a thickness gradient of an epoxy film on glass or silicon substrates. After sectioning the film into individual squares, a temperature gradient was applied orthogonal to the thickness gradient to induce debonding events where the adhesion is below a critical value. In addition, another combinatorial library was carried out using the epoxy films with concentration gradient and constant thickness, and applying a constant temperature. The combined results clearly demonstrate that our combinatorial library design and approach provide a large parameter space for accurately and reproducibly mapping the interfacial integrity and bond strength of film/substrate systems.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

One area of concern for both manufacturers and end users in the use of thin polymer films is the reliability of the bond strength between film and substrate. For example, next generation electronic components will require novel thin films to be integrated into their architecture and construction, and these materials will need to withstand demanding conditions and demonstrate exceptional durability. In developing these new materials, it is important to assess the reliability of the bond strength in a fast, practical and reproducible fashion. Accordingly, the application of combinatorial approaches to adhesion tests has been demonstrated [1–4]. Among them, the framework for a combinatorial approach to the edge delamination test has been conceptually proposed to map the failure of film/substrate bond as a function of both temperature and film thickness using a single specimen in one step [1]. This framework includes the theory, experimental design, stress analysis and simulation of the approach. Also, the concept of the combinatorial approach has been adapted to include various adhesion-controlling parameters such as substrate surface energy [5]. In this study, in order to complete our early success in the development of the combinatorial approach that were achieved from quantifying the relatively weak bonding system

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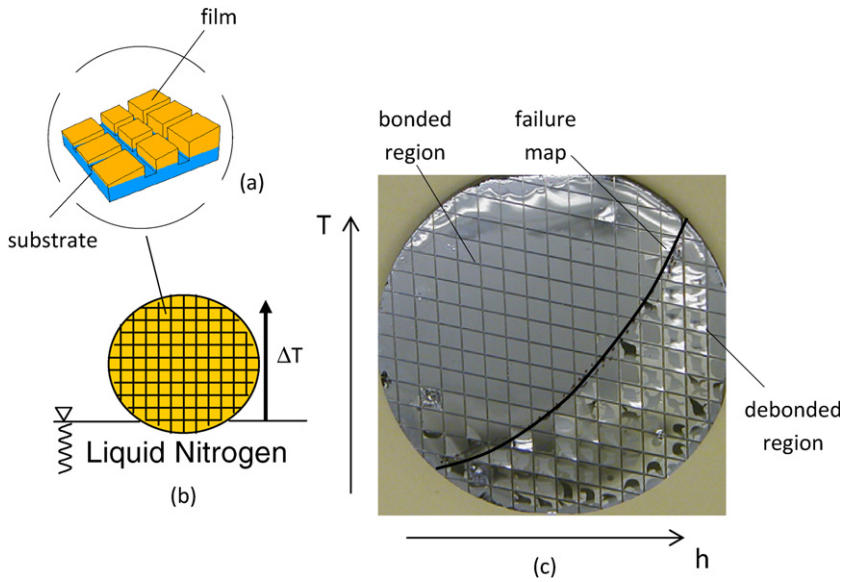

Figure 1. A schematic representation of the combinatorial approach to the edge delamination test: a square pattern array of individual edge delamination samples is fabricated on the substrate to form a specimen with film thickness gradient (*a*), one side of the specimen is dipped into liquid nitrogen to form a temperature gradient, *T*, orthogonal to the film thickness gradient (*b*), debonding for those edge delamination samples having critical stresses can be observed by the un-aided eye (*c*).

(PMMA/silicon system), we have extended the approach to investigate the interface of epoxy/silicon or glass systems.

Generally, the edge delamination test quantifies a bond strength of a film/substrate system by monitoring debonding events that are driven by the thermal stress due to a mismatch of the coefficients of thermal expansion at the critical temperature. Based on this self-debonding concept and practical applications of polymer coatings [6, 7], a film is coated onto a relatively rigid substrate in such a way that the film has a controlled thickness gradient in one direction, which is then subdivided into an array of individual edge delamination samples, as depicted in figure 1(*a*). The specimen is then cooled with a temperature gradient orthogonal to the thickness gradient, as shown in figure 1(*b*). Instantaneously, debonding events will be observed for those samples having critical stresses greater than a critical value. These stresses depend on the combination of local temperature and film thickness. As a result, a failure map (distribution of failure) as a function of film thickness and temperature can be constructed (see figure 1(*c*)). Subsequently, the bond strength between the film coating and the substrate can be deduced from the failure map if the stress–temperature relationship between the film and the substrate is given. This relationship can be obtained from a separate experiment, such as *in situ* curvature measurements of a bi-material circular plate (comprised of a film and substrate) due to a change in temperature [5], or it can be established using mechanical and physical properties of the film and substrate.

In this study, we focus on epoxy/silicon and glass systems, since these systems are widely used in the electronics industry. This work also experimentally demonstrates the reproducibility of the combinatorial approach in mapping the interface integrity and the accuracy in quantifying the bond strength deduced from the combinatorial approach. The bond strength is defined here as the debonding energy or the fracture toughness. In addition, a similar approach is applied to quantify the interface integrity of the epoxy films on substrates as a function of epoxy composition for adapting to the high throughput test which is considered to be very useful in the industry.

2. Experiment[6]

2.1. Combinatorial edge delamination test with thickness and temperature gradients

The epoxy resin was 3,4-epoxycyclohexyl methyl-3,4-epoxy cyclohexyl carboxylate (ERL-4221, SPI-Chem, West Chester, PA, USA). Hexahydro-4-methylphthalic anhydride and cobalt (III) acetylacetonate (both from Aldrich) were used as a hardener and a catalyst, respectively. All these chemicals were used as received. In the preparation of thin film materials, the epoxy resin and hardener were mixed together with a ratio of 1 : 1 mol fraction and stirred for 2 h at 70 °C. Then, 1 mol% of catalyst was added to the mixture and dispersed by agitating the mixture for an additional 2 h at 70 °C. This uncured resin/hardener/catalyst mixture was deposited on a substrate. In this study, borosilicate glass (75 mm × 75 mm × 3 mm) and silicon wafers (75 mm × 75 mm × 5 mm) were used for substrates. Prior to the deposition, the surface of the substrate was thoroughly cleaned using toluene, ethanol and acetone in order to remove organic contaminants. After this cleaning process, the substrate was placed into a UV/ozone cleaner (Model 342, Jelight Company, Inc, Irvine, CA, USA) for 20 min to render the surfaces extremely hydrophilic.

A poly(dimethylsiloxane) (PDMS) gasket was placed onto the edge of the cleaned substrate to form a corral for confining

[6] Certain commercial materials and equipment are identified in this paper in order to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology nor does it imply that they are necessarily the best available for the purpose.

the epoxy mixture inside a prescribed area. The thickness of the gasket was 5 mm and the inside area of the gasket was about 60 mm × 60 mm, in which the uncured epoxy mixture was spread. Then, the specimen (film/substrate system) was placed onto a tilt stage (TGN-120, Newport Corporation, Irvine CA, USA) to produce a thickness gradient. The slope of the thickness gradient can be tuned by adjusting the tilt angle of the stage, while the magnitude of the film thickness can be controlled by the total volume of epoxy dispensed. The tilt stage assembly was fixed inside a curing oven so that the film can be cured while remaining in the tilted position. The result is an epoxy film with a gradient in thickness along one axis of the film. For the epoxy/glass specimen, about 1.0 mL of the uncured epoxy mixture was used. For the epoxy/silicon system, the volume was much less than 1.0 mL. The specimen (film/substrate system) was pre-cured at 70 °C for 1 h and subsequently cured at 170 °C for 2 h. The pre-cure procedure was adopted to maintain an adequate viscosity for the flow and levelling of the mixture along the thickness gradient. After curing, the PDMS gasket was removed and the thickness gradient was examined using a Dektak 8 stylus profiler (Veeco Instruments, Woodbury, NY).

The cured specimen was diced using a precision diamond saw into a square grid pattern (~5 mm × 5 mm) to form an array of about 11 × 11 (total of 121) individual edge delamination samples (figure 1). The cut penetrated some distance (~200 μm) into the substrate and formed a 90° edge at the film/substrate interface. The cutting depth, a design parameter for the combinatorial specimen, was chosen based on the findings from a previous study [1].

A gradient in temperature was produced using an aluminium plate, (102 × 62 × 4) mm^3 with a heat source on one end and a heat sink on the opposite end, as shown in figure 2(*a*). The heat source was a cartridge heater powered by a temperature controller (CN77000, Omega Engineering Inc., Stamford, CT, USA). The heat sink was a refrigerated circulating bath (RTE-220, Neslab, Newington, NH, USA) thermostated to a constant temperature. To perform the combinatorial edge delamination test, the specimen with the thickness gradient is placed on the temperature stage orthogonal to the temperature gradient to induce debonding. The debonding events were observed with the un-aided eye for those samples having the critical stress that is a function of local temperature and film thickness. Consequently, a failure map as a function of thickness and temperature can be constructed within a single specimen and in one step. It is worthwhile to note that the thickness and temperature gradient in each individual sample are negligible.

The temperature at a given position, x, on the stage (figure 2(*a*)) can be estimated from the following equation [12]:

$$T(x) - T_a = \frac{(T_1 - T_a)\mathrm{Sinh}(\alpha(l-x)) + (T_2 - T_a)\mathrm{Sinh}(\alpha x)}{\mathrm{Sinh}(\alpha l)} \quad (1)$$

with α being the smallest positive solution of

$$\alpha \tan\left(\frac{\alpha h}{2}\right) = \frac{C}{K} \quad (2)$$

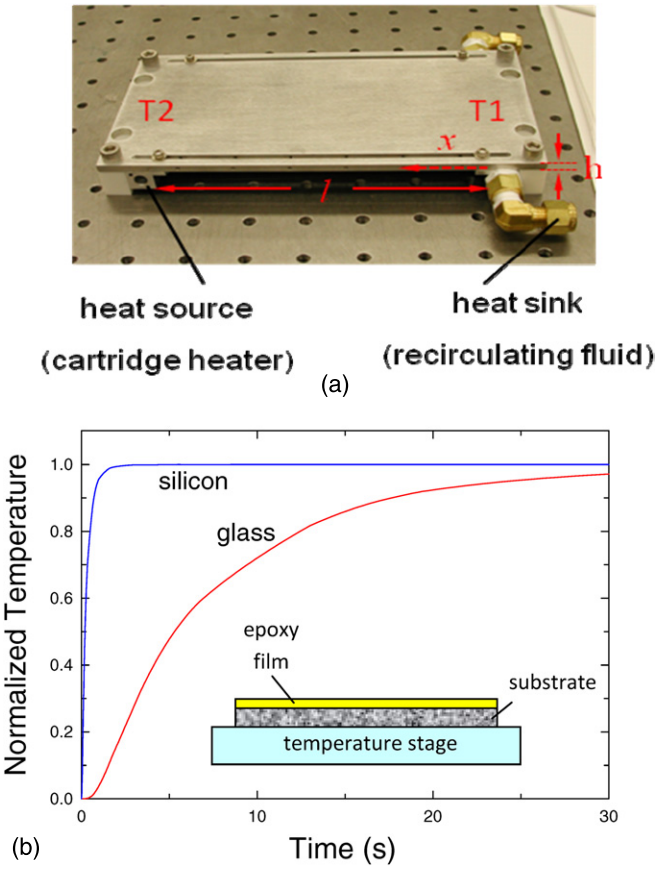

Figure 2. Photograph of the temperature stage with heat source and sink (*a*), and transient state of temperature at the interface between an epoxy film and glass and silicon substrates (*b*).

where C is the coefficient of natural (free) convection and K is the thermal conductivity of the stage. T_a is the ambient temperature. T_1 and T_2 are the temperatures of the refrigerating bath and the heating bar, respectively. l and h are the length and thickness of the stage, respectively. The ratio of l to h should be greater than 10 in order to have a uniform temperature gradient. In this study, the temperature gradient typically ranged from −60 °C to −105 °C and from −120 °C to −150 °C over 60 mm for the epoxy/glass and the epoxy/silicon systems, respectively. We assume that the temperature of the sample is the same as the temperature of the stage since the thickness of the sample is relatively small. Our finite element analysis indicates that for a substrate with either a relatively low thermal conductivity such as glass ($k \approx 1.4\,\mathrm{W\,m^{-1}\,K^{-1}}$) or a high thermal conductivity such as silicon ($k \approx 148\,\mathrm{W\,m^{-1}\,K^{-1}}$), the temperature of specimen equilibrates to the corresponding stage temperature within a few seconds (1 s for silicon and 30 s for glass). This is shown graphically in figure 2(*b*).

2.2. *Combinatorial edge delamination test with composition gradients at constant thickness and temperatures*

Figure 3 shows a schematic depicting our approach for preparing combinatorial specimens possessing discrete gradients in epoxy composition. The deposition system consisted of meter/mix/dispense to generate the composition

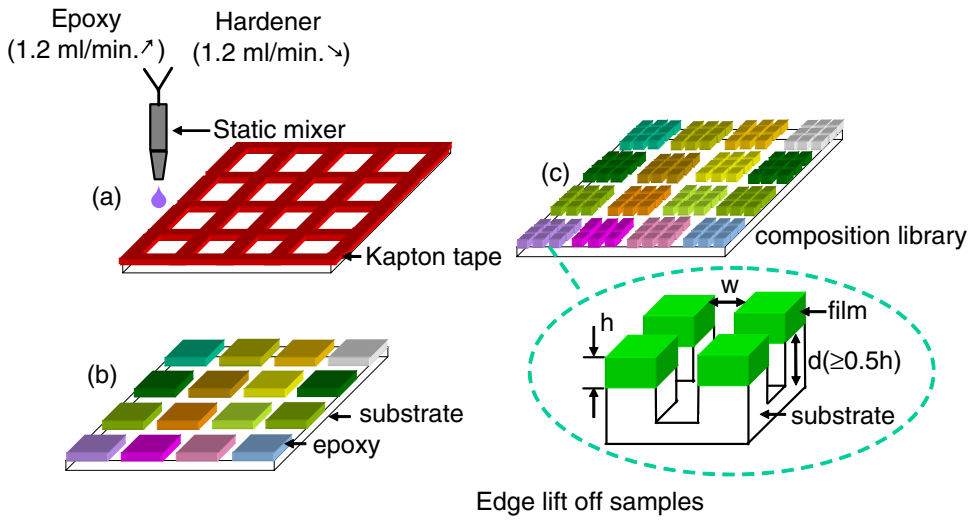

Figure 3. A schematic of the fabrication process for the composition library. After the deposition of the epoxy mixture on the substrate (*a*), the composition library was cured and Kapton film spacers were removed (*b*) and diced to generate the geometry of the sub divided sample (*c*).

gradient on the substrate. To control the epoxy film thickness and generate a discrete composition gradient, Kapton tape with approximately 130 μm thickness was used as a spacer on the substrate (see figure 3(*a*)). For metering the epoxy resin and hardener, syringe pumps were used to inject the epoxy resin and curing agent through polyethylene (PE) tubing, and these two components (i.e. epoxy resin and hardener) were mixed together using an in-line static mixer, and the mixture was dispensed into each cavity surrounded with Kapton tapes. The pumping speed of each syringe pump was started at 1.2 mL min^{-1} for the first cavity, with a 0.05 mL min^{-1} increment for the epoxy resin and the same speed decrement for the curing agent in each subsequent cavity. This allows us to create a composition gradient (total 16 compositions) with constant volume of mixture on a substrate.

After depositions, the epoxy/substrate system (specimen) was degassed at room temperature for 40 min. Then, glass beams coated with the polydimethylsiloxane (PDMS) sheets were used to press and spread the droplets of mixture in the cavity, such that a uniform film thickness can be obtained. After the curing process at 170 °C for 2 h, the Kapton film spacers and glass covers were removed from the composition library (see figure 3(*b*)), and the specimen was diced into individual edge delamination samples (5 × 5 mm^2), as shown in figure 3(*c*).

The compositional library for the edge delamination test was placed on a cryogenic stage with a constant temperature to induce a thermal stress. The temperature was lowered by controlling the flow amount of liquid nitrogen to a critical value, such that failure events of the epoxy/substrate were recorded using a digital camera. In order to verify the composition of the cured epoxy film in the library, FTIR microspectroscopy in reflectance mode was applied to each of the films containing various ratios of epoxy/hardener. The epoxy/substrate systems were deposited on copper plates with a mirror-like surface, using the Nic-Plan IR microscope (Thermo Nicolet Inc. Madison, WI, USA) interfaced with

Table 1. Thermal and mechanical properties of the epoxy films with various epoxy compositions.

Epoxy concentration (Mass %)	Modulus (GPa)	Glass transition temperature (°C)
51	2.8 ± 0.21	140 ± 4.2
56	2.9 ± 0.13	162 ± 5.5
60	3.0 ± 0.15	155 ± 2.1
66	2.9 ± 0.20	122 ± 7.7
73	2.4 ± 0.11	80 ± 2.6

Note: Unless otherwise indicated the ± refers to the standard deviation and is taken as an estimate of the standard uncertainty.

Nicolet Magna-IR 550 FTIR spectrophotometer. Three to five spectra were collected from each discrete ratio of epoxy/curing agent film in the 650 to 4000 cm^{-1} region with 8 cm^{-1} resolution, 256 scans and a beam spot size of 120 μm × 120 μm. The ratios between the integrated areas under the 1860 cm^{-1} anhydride peak of the hardener (1880 cm^{-1}–1830 cm^{-1} spectral region) and the 1750 cm^{-1} constant CO peak (1775–1722 cm^{-1} spectral region) used as an internal control for thickness differences were obtained using the ISys software package (Spectral Dimensions Inc., Olney, MD). Averages and standard deviations of these peak ratios were calculated for each composition ($n = 3$ to 5).

Glass transition temperatures (T_g) of the epoxy films were measured by TA Instrument Q-1000 differential scanning calorimeter as a function of epoxy composition. Approximately 5 mg of the cured films collected from each batch of the epoxy mixtures were encapsulated in aluminium DSC pan. The temperature for scanning was elevated to 250 °C by 5°C min^{-1} under N_2 atmosphere. The measured T_g value for various compositions is listed in table 1.

In order to quantify the stress–temperature relationship of epoxy coated on a relatively rigid substrate (shown later in equation (5)), the modulus of epoxy as a function of composition was determined using the flexural test (ASTM D790). Flexural specimens with 2 mm square of the cross

section and a span-to-thickness (l/h) ratio of 10 : 1 were deflected in 3 point bending mode with 0.5 mm min^{-1} speed. The flexural modulus, E, was calculated using the following equation:

$$E = \frac{l^3 s}{4wh^3}, \tag{3}$$

where w represents the specimen width. s is the initial slope of the load–deflection curve. The measured flexural properties are listed in table 1. The l/h ratio used in the study is shorter than 16 : 1 of ASTM D790, some contribution of a shear to flexural deformation can be expected for the flexural test. The finite element analysis indicates this contribution is less than 5%, thus the influence of the shear on the flexural test is negligible.

3. Results and discussion

3.1. Combinatorial library with thickness and temperature gradients

In the combinatorial edge delamination test, precise control of the gradients (film thickness and temperature) is vital and ultimately reflects the accuracy of the method in quantifying bond strength. Figure 4(a) shows a measured surface temperature gradient that is nearly linear in a typical combinatorial edge delamination specimen (epoxy/glass). In addition, the result shown in figure 4(a) indicates good agreement between the measured and predicted temperatures that were obtained from equation (1). Figure 4(b) shows a typical thickness variation in the direction orthogonal to the temperature gradient of the edge delamination specimen. The film thickness varies linearly from 30 to 300 μm over 60 mm distance.

The micrograph of the pre-cracks at the film/substrate interface due to dicing is shown in figure 5. The length of pre-cracks obtained from the micrograph is much longer than 4% of the film thickness; this means that the stress states in the film/substrate system would be independent of the pre-crack length [1]. Figure 6(a) shows the photographs of fracture events of epoxy/glass system obtained from combinatorial edge delamination test. The fracture was observed for those samples having the critical stresses that depend on the combination of local thickness and temperature in the specimen. By tracing the line of the transition between bonded and debonded regions (locus of the fracture), one can obtain a failure map shown in figure 6(b) as a function of thickness and temperature. Our analysis on the fractured surfaces suggests that failures took place in the glass substrates very near the interface (cohesive failure) and were dominated by an opening mode (mode I failure). Accordingly, the stress intensity factor in an opening mode, K_I, can be expressed as [8]

$$K_I = \sigma_o \sqrt{\frac{h_f}{2}}, \tag{4}$$

where σ_o is the internal biaxial stress of the epoxy film that depends on the mismatch of the coefficients of thermal expansion and the temperature at failure. The fracture toughness, K_{IC}, can be quantified by fitting equation (4) to

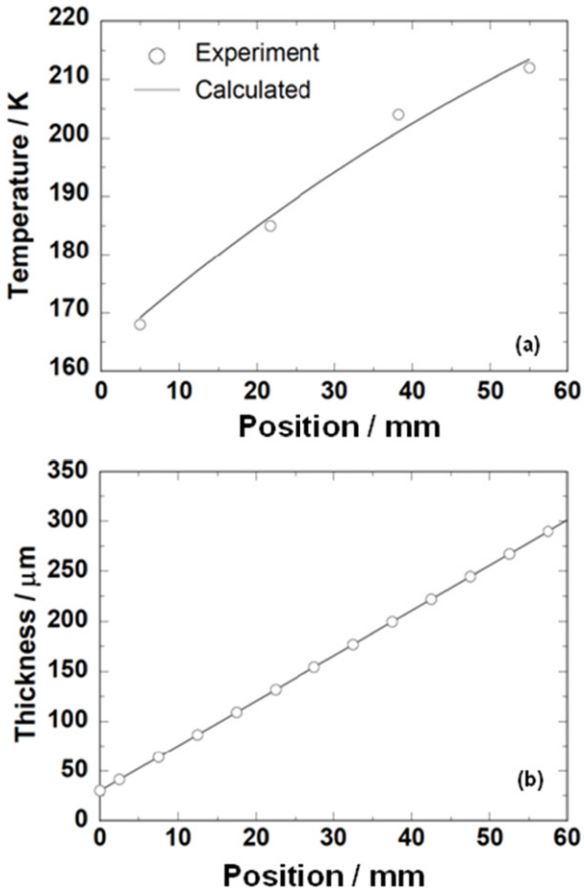

Figure 4. Temperature (a) and epoxy film thickness (b) as a function of position in a typical combinatorial edge delamination specimen of epoxy/glass system.

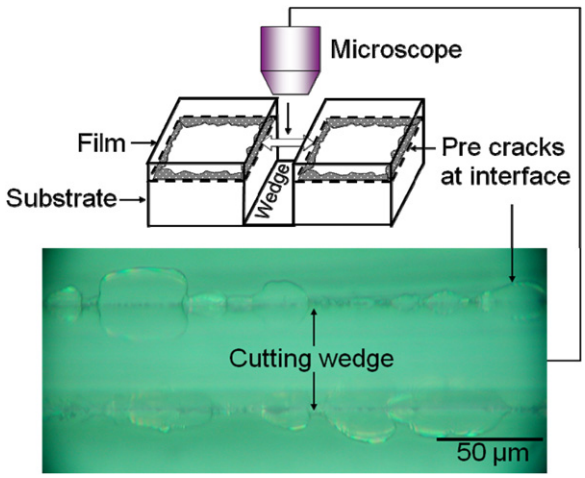

Figure 5. Micrograph of the pre-cracks at the epoxy/silicon interface after dicing.

the experimentally constructed failure map once the stress–temperature relationship (i.e. σ_o) of the epoxy/glass system is determined. The σ_o of a film on a substrate can be readily obtained in a separate experiment through Stoney's equation based on an *in situ* curvature measurement as a function of temperature for a coated bi-material circular plate (such as a film/substrate system) [9, 10]. However, if the mechanical and physical properties of film and substrate are

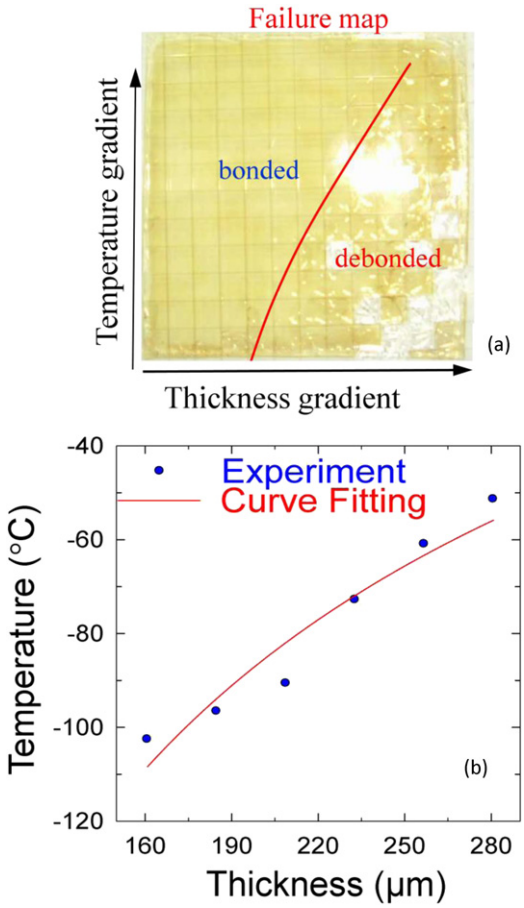

Figure 6. Experimental observation of the combinatorial edge delamination test obtained for epoxy/glass (*a*) and the failure map extracted from the experiment as a function of temperature and epoxy film thickness (*b*).

given, the stress–temperature relationship of a thin film coated on a relatively rigid substrate can be established through the following equation:

$$\sigma_o = \overline{E}_f(\alpha_s - \alpha_f)(T - T_{ref}), \tag{5}$$

where $\overline{E}_f = E_f/(1 - \nu_f)$. The subscripts f and s represent the film and substrate, respectively. E and α are the elastic modulus and coefficients of thermal expansion, respectively. T_{ref} is the reference temperature at which the film/substrate system is in a stress-free state, and it can be reasonably chosen as the T_g of the film.

In this study, we chose to use equation (5) to estimate σ_o of the epoxy/glass system. For the epoxy film, $E_f = 3.68$ GPa, $\nu_f = 0.33$, $\alpha_f = 80 \times 10^{-6}\,^{\circ}\mathrm{C}^{-1}$; for the glass substrate, $\alpha_s = 3.95 \times 10^{-6}\,^{\circ}\mathrm{C}^{-1}$ [11]. By setting T_{ref} equal to 150 °C (T_g reported in the literature for the epoxy used in this study), the calculated σ_o of the epoxy/glass system is rearranged in the following form:

$$\sigma_o = C_1 + C_2 T, \tag{6}$$

where $C_1 = 70.89$ MPa and $C_2 = -0.47$ MPa °C^{-1}. This estimate of σ_o obtained through equation (5) can be higher than the actual measured value simply because of the assumption on T_{ref} and independence of E_f and α_f in equation (5) on the thermal history. While this equation is not complete, it does provide a quick and reasonable estimation of the stress. By combining equations (4) and (6), one can obtain

$$T = \frac{1}{c_2}\left(K_{IC}\sqrt{\frac{2}{h_f}} - c_1\right). \tag{7}$$

This equation is used to fit the experimental failure map as a function of thickness and temperature, and using K_{IC} as a fitting parameter. The values for K_{IC} deduced from the fitting data are 0.92, 1.09 and 1.12 MPa m$^{1/2}$ as shown in figure 6(*b*) for the representative failure maps. The data indicate that K_{IC} for this material system is reproducible and has an average value of 1.04 ± 0.11 MPa m$^{1/2}$. This value is close to the fracture toughness of borosilicate glass reported in the literature (0.8 MPa m$^{1/2}$, [11]), since the actual fracture occurred in the glass substrate, not at the interface.

The similar test using a silicon substrate with the film thickness gradient from 40 to 173 μm was carried out, and failure was cohesive in the silicon phase near the interface of the epoxy/silicon. The K_{IC} of silicon deduced from the combinatorial specimen is 0.88 ± 0.19 MPa m$^{1/2}$, which is consistent with the reported value (0.73–0.83 MPa m$^{1/2}$) in the literature [13]. The applied temperature range in the epoxy/silicon system was quite lower than that in the epoxy/glass system, although the fracture toughness of glass and silicon is very similar. This is due to the film thickness on the epoxy/silicon system being smaller than that in the epoxy/glass system, and the energy for failure (either cohesive or interfacial) depends not only on the local temperature but also on the local thickness. For the epoxy/glass specimen, failures occurred within a few seconds after placing the specimens on the desired temperature gradient. For the epoxy/silicon specimen, the failure occurred instantaneously after the placement. These observations on immediate debonding are attributed to the very short transient state of temperature in the specimens and are in line with the expectation shown in figure 2(*b*). In this study, the CTE of silicon substrate (α_s) was assumed to be $2.5 \times 10^{-6}\,^{\circ}\mathrm{C}^{-1}$.

3.2. Compositional library using the epoxy films with the various epoxy concentrations

The epoxy and hardener mass in each deposition can be estimated by multiplying pump speeds, duration time and their densities. The estimated mass of mixture after deposition is compared with the measured mass for validating the accuracy of the deposition system in figure 7(*a*). The discrepancy between the estimated and measured masses is less than 4% throughout the deposition. Based on the relationship shown in figure 7(*a*), the desired epoxy concentration in each deposition can be obtained by controlling pump speed, as shown in figure 7(*b*).

After completing the deposition and curing process for the library, FTIR-RTM measurements were performed to verify the epoxy composition gradient. The FTIR peak ratios of epoxy and anhydride obtained from the cured epoxy spectra are presented in figure 8 as a function of the epoxy

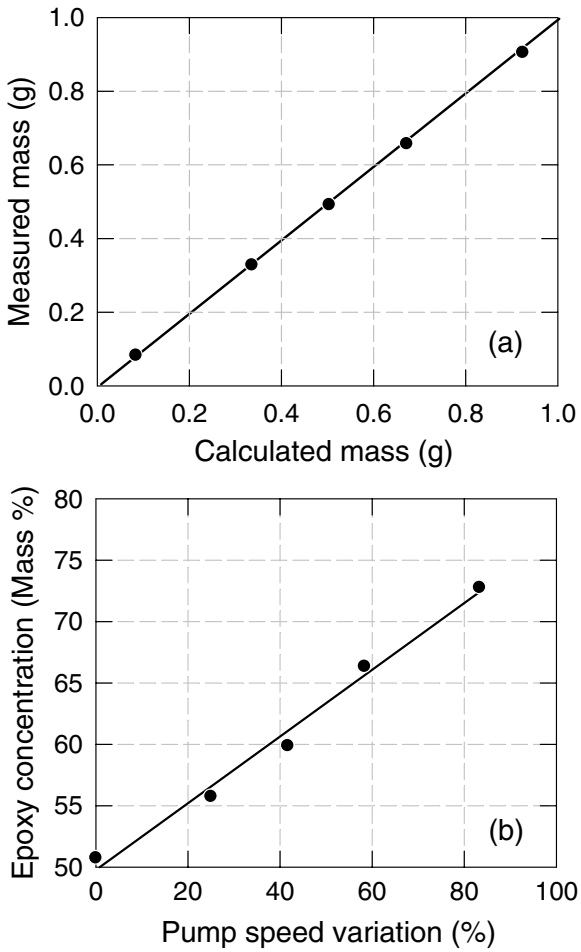

Figure 7. The measured versus calculated masses of epoxy mixture (*a*), the relationship between the epoxy concentration in each deposition and the pump speed (*b*).

concentration. The gradual decrease in this ratio reflects the degree of reaction of the anhydride hardener with the epoxide groups. The amount of the unreacted anhydride decreased monotonically in the epoxy film up to the 60% region and is fully consumed by the reaction with the epoxy groups beyond this region. Therefore, anhydrides of the hardener over 60% of the epoxy concentration region are not detected by FTIR-RTM in this study. Although FTIR-RTM does not sufficiently capture concentration having greater than 60% epoxy, figure 8 qualitatively demonstrates that a compositional gradient was established.

Using this library, the fracture events of the samples during the test were recorded while applying the temperature down to −180 °C, as shown in figure 9. In general, samples with lower epoxy concentrations failed at lower temperatures, and their failure occurred at different phases of the sample. For example, interfacial failure occurs at −100 °C when the epoxy concentration of samples is approximately 73%. At −187 °C, the samples with 51% of the epoxy concentration (the lowest concentration studied) exhibit substrate failure. For films with epoxy concentration from 66% to 73%, the failure appears to be at a mixture of interface and substrate (cohesive). These failure patterns suggest that the actual interfacial toughness mentioned in figure 6 could have been obtained if the epoxy concentration

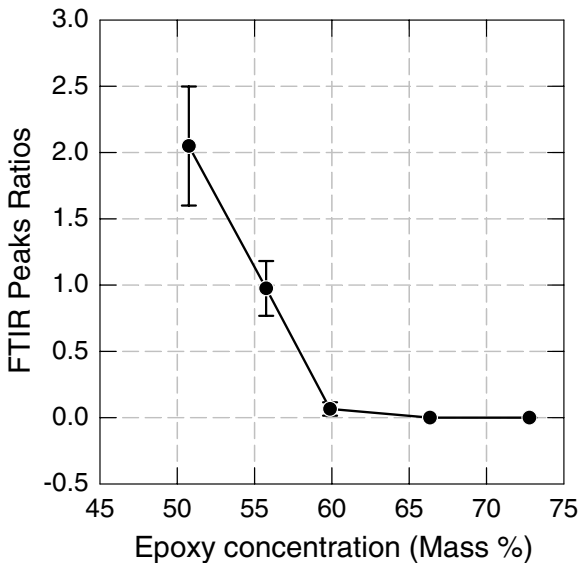

Figure 8. FTIR peak ratios versus the epoxy concentration.

was properly controlled. Figure 10 presents quantitative failure of the samples as a function of temperature based on the image analysis of figure 9. The result in figure 10 clearly shows the reliability of the film/substrate system at certain film thickness. This suggests that the combinatorial methodology of the edge delamination test in this study qualitatively enables a rapid estimation of the interfacial integrity of the samples.

In the case of the composition gradient, the method (failure map) for obtaining the fracture toughness of the combinatorial specimen with thickness and temperature gradients cannot be used here. This is due to different sample material properties in the composition gradient library. Therefore, for quantifying the interfacial integrity in this case, we used equations (4) and (5) to determine the mode I fracture toughness of film/substrate as a function of concentration. The modulus and T_g used for calculating the stress in films is shown in table 1. The K_{IC} shown in figure 11 has a nearly constant value for the epoxy concentrations from 51% to 66%. Also, the failures are cohesive in the substrate for the concentration range. In addition, the K_{IC} values obtained from the test agree with the value reported in the literature for the glass (0.80 MPa $m^{1/2}$, [11]) and silicon (0.73 MPa $m^{1/2}$ to 0.83 MPa $m^{1/2}$, [13]). For the concentration beyond 66% the K_{IC} then decreases with further increase in the concentration, and the failures become interfacial. In this study, Poisson's ratio of epoxy film was assumed to be independent of epoxy concentration.

4. Conclusions

We have demonstrated the applicability of a combinatorial approach to the edge delamination test by probing the interface integrity of an epoxy film with two different substrates, glass and silicon. By combining two practical parameters (temperature/film thickness or temperature/composition) that are important and readily controllable, the effect of these parameters on the bond failure can be mapped in one

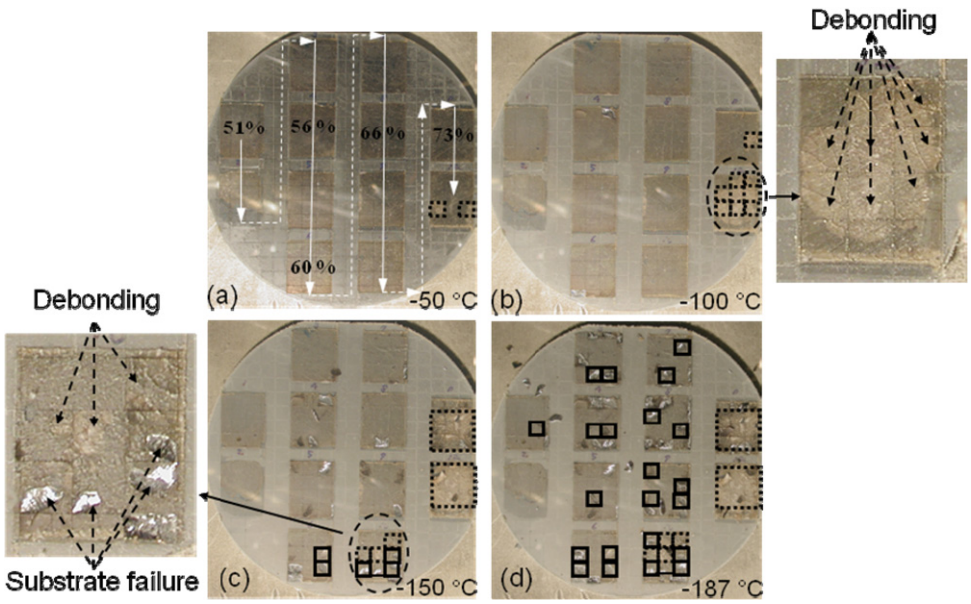

Figure 9. Photo images of the fracture behaviour of the epoxy/silicon specimens in the composition library during the edge delamination test. Squares with solid and dotted lines in the image indicate the cohesive failure and interfacial debonding, respectively. The composition gradient is indicated by white arrows.

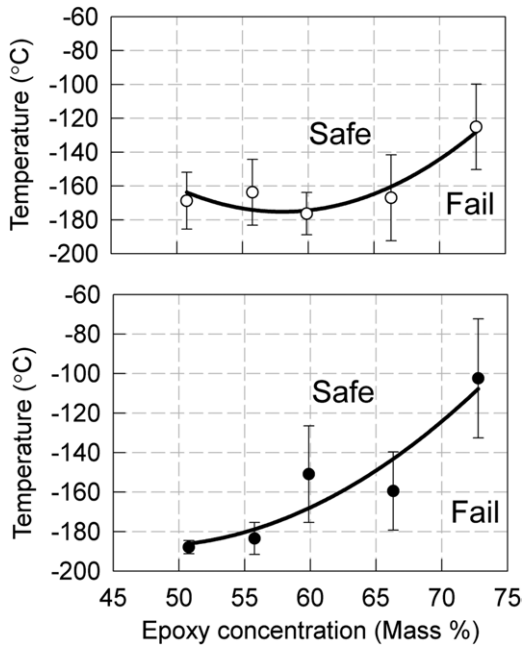

Figure 10. Failure maps of the epoxy/glass (open symbols) and epoxy/silicon (filled symbols) after the edge delamination test as a function of failure temperatures.

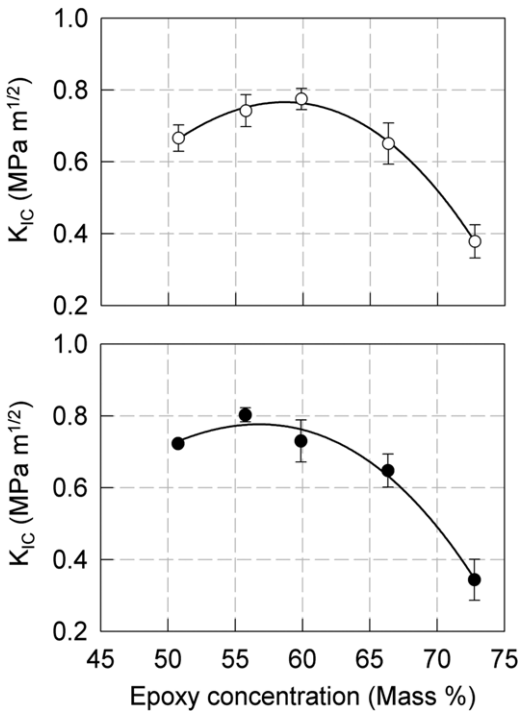

Figure 11. Mode I fracture toughness of the epoxy/glass (open symbols) and epoxy/silicon (filled symbols) as a function of epoxy concentration.

experiment with a single specimen. Also, once the stress–temperature relationship of a film bonded to a substrate is known, the failure map can be used to quantify the fracture toughness of the film/substrate system. This study demonstrates the accuracy and reproducibility of the combinatorial approach for evaluating the interface integrity of film to substrate. In conjunction with our early success for the conceptual development of the combinatorial approach, this study seems to give a completeness of the approach for evaluating the interface integrity.

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