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Zhang, Xiaohan

Study of Epoxy
Toughening in Bulk
and Disbond
Initiation at
Epoxy/Aluminum
Interfaces

January 2007

**Study of Epoxy Toughening in Bulk and
Disbond Initiation at Epoxy/Aluminum
Interfaces**

by

Xiaohan Zhang

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Presented to the Graduate Committee of Lehigh University

in

Polymer Science and Engineering

2006

Thesis Signature Sheet

This thesis is accepted and approved in partial fulfillment of the requirements for the Master of Science.

Date 12/6/2006

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Abstract

This research project includes two parts. Part one is to study the fracture toughness of DGEBA epoxy. And part two is the study of the disbond initiation at epoxy/aluminum interfaces.

Epoxies are widely used as electronic packaging materials. It has excellent bonding properties. However, neat epoxy is very brittle, which limits its applications. Epoxy toughening has drawn a great interest in both industry and academic research for years. Part I of this work is to investigate the effect of various toughening material on a DGEBA epoxy system.

Part II of this work is to study the disbond initiation at epoxy/aluminum interfaces by using a stress singularity approach. This work focused on disbond initiation under shear load. The moisture uptake of various epoxy systems was measured. The effect of moisture on yield strength was also investigated.

Chapter 1 Overview of the project

1.1 Overview

Epoxies are excellent structure adhesives. They can bond to various substrates and have excellent properties [1-4]. Epoxies are widely used for electronic packaging industry. However, neat epoxy systems are normally very brittle. This nature limits the application of epoxies because the bonded joints will be more susceptible to shock or vibration [5]. The neat epoxy systems can be toughened by many kinds of fillers, including glass spheres, rubber particles or nano-particles, to increase their toughness and bond strength.

On the other hand, when epoxy systems are used as adhesives to bond adherends together, the crack initiation plays an important role in joint failure. As soon as the crack initiates, it can propagate very fast.

This project contains two parts. The epoxy toughening in bulk; and the crack initiation at epoxy/aluminum interfaces will be studied respectively.

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Part I

Chapter 2 Study of Fracture Toughness of DGEBA Epoxy Composites

2.1 Introduction

Poor epoxy toughness can be improved by adding toughening agents, such as rubbery phase [1,2] or inorganic fillers [3,4]. These are very effective methods to reinforce the material. Several factors can influence the toughening effect, such as, the volume fraction of the modifier, particle size, or particle distribution [5]. Adding rubbery phase can improve the toughness by cavitation and shear banding [6,7]. The addition of the rubber modifier can toughen the epoxy by promoting process zone mechanisms such as cavitation/shear banding and plastic void growth [1,7]. Carboxyl terminated butadiene acrylonitrile copolymers (CTBN) rubber particles are commonly used to toughen epoxy polymers. Inorganic fillers can cause microshearing banding, microcracking and crack pinning [8]; adding fibers can toughen the material by cracking pinning, fiber breakage, fiber bridging, debonding and pull-out [9,10]. Pinning the crack tip causes the crack front to bow between the rigid impenetrable particles, thereby absorbing more energy due to line tension.

Rigid particles can also cause microcracking. The type of toughening mechanism appears to be related to particle size and matrix particle adhesion.

If the rubbery particles and rigid inorganic fillers are both added into epoxy, a positive interaction between the two toughening mechanism may happen [11,12]. However, too many rigid particles suppress may matrix shear banding. The use of nanometer size particles may work better since shear band may simple deflect around such small particles.

2.2 Objectives

To use various fillers to study the toughening effects on a DGEBA epoxy system.

2.3 Experimental Approach:

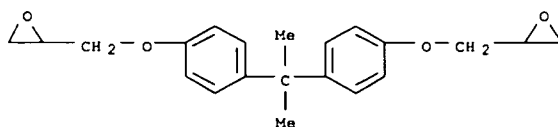
2.3.1 Materials

DER 331 resin, a diglycidyl ether of bisphenol A epoxy resin (DGEBA) with a equivalent weight of 187 g/mol, was supplied by Dow Chemical Company.

The curing agent used is aminoethyl piperazine (AEP) from Air Products and Chemicals, Inc. and piperidine (PIP). Carbonxyl-terminated butadiene-acrylonitrile (Hycar® CTBN 1300X8) was supplied by B. F.

Goodrich; Solid glass spheres (SGS 6000), mean diameter 7 μm , was supplied by Potters; solid glass spheres SGS 3000, mean diameter 15 μm .

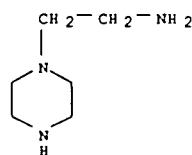
Table 2.1 Chemical Structures of Resin and Curing Agents



diglycidyl ether of bisphenol A epoxy resin



Piperidine



aminoethyl

piperazine

Nano-particles were treated silica suspended in DER^h 332 concentrate (40 wt% nano-particles in DER^h 332) from 3M^h. particle size is 40 nm.

Table 2.2 Descriptions of the toughening agents

Modifier	Description of modifier
CTBN	Carboxyl terminated liquid copolymer of butadiene and acrylonitrile from B.F Goodrich [Hycar CTBN 1300X8]
SGS (3000)	Solid glass spheres, mean diameter is 15 μm from Potters
SGS (6000)	Solid glass spheres, mean diameter is 7 μm from Potters
Nano-material	Treated silica (particle size is 40 nm) suspended in DER ^k 332 concentrate (40 wt % nano-particles in DER ^k 332) from 3M

2.3.2 Sample Preparation

The neat DGEBA/AEP material was made through the following steps. The stoichiometric ratio of DGEBA epoxy resin and curing agent AEP were mixed together at room temperature then degassed and stirred under vacuum for 5 minutes. The solution was cast into a 6mm thick aluminum mould. The mould was put under room temperature for 1 hour to gel and then put in oven

at 125°C for 2 hours. The same curing schedule was also applied on all toughened epoxies.

The neat DGEBA/PIP material was made at a ratio of DGEBA/PIP is 100:5 by weight. Epoxy resin and curing agent PIP was mixed together at room temperature then degassed and stirred under vacuum for 5 minutes. The solution was cast into a 6mm thick aluminum mould. The mould was put into oven at 160°C for 16 hours. The same curing schedule was also applied on all toughened epoxies.

CTBN was added to the epoxy resin and agitate to disperse at 80°C for 24 hours, then cool down to room temperature; nano-material was added and mixed for 24 hours at room temperature, then curing agents were added.

2.2.3 Three-point-bending test

Plain strain fracture toughness, K_{IC} , values were obtained by three point bending (3PB) test. The ASTM D5045 guideline [13] was followed to measure K_{IC} in the test. Six-millimeter-thick samples were used for the 3PB geometry. Pre-cracks were made by hammering a razor blade that was chilled in liquid nitrogen. A screw-driven Instron testing frame at a crosshead speed of 1mm/min was used for these tests.

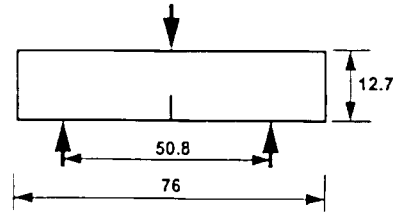


Fig. 2.1 Three point bending test for K_{IC}

The following equation is used for the calculation:

$$K_{IC} = \frac{10^{3/2} \times P \times S}{B \times W^{3/2}} \cdot \frac{3x^{1/2} [1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)]}{2(1+2x)(1-x)^{3/2}}$$

(Equation 2.1)

Where

P = load, S = span, B = thickness, W = sample width, $x = a/w$

2.4 Results and Discussion

2.4.1 Fracture toughness of neat epoxies:

The fracture toughness of neat epoxy varied with different type of curing agent. The K_{IC} value for AEP cured neat epoxies is $1.45 \text{ MPam}^{0.5}$, which is much higher than that of neat epoxies cured by PIP. Neat epoxies cured by

different curing agent may have different cross-linking density, which has significant effect on the fracture toughness. That could be the reason of the toughness difference between the neat epoxies cured by different curing agents. Pearson and Yee studied the effect of crosslink density on fracture toughness [14]. Kinloch and Levita also looked at cross-link density [15,16].

Table 2.3 Fracture toughness values of the neat epoxy system studied

Designation	DGEBA (g)	Curing agent (g)	K_{IC} (MPa m ^{0.5})
AEP cured epoxy	374	86	1.45
PIP cured epoxy	100	5	0.85

2.4.2. Fracture toughness of SGS toughened DGEBA/AEP epoxy system

For the SGS 6000 toughened epoxy, there is no significant change on fracture toughness when the volume fractions of SGS varied from 5% to 10%. For the SGS 3000 toughened epoxy, the fracture toughness is almost constant at volume fraction of SGS is 2.5% or 5%. Further increase of SGS volume fraction, 7.5% and 10%, fracture toughness increases too. Since the larger particles are more effective due to micro cracking. However, this phenomenon is different with what Lee and Yee published [17,18]. DGEBA-PIP system was used and conclusion was that no significant influence on glass bead size on

fracture toughness. The reason for the disagreement could be the different curing agent used (AEP instead of PIP).

Table 2.4 Formulations of SGS modified epoxy (AEP cured) studied.

Modifier	DGEBA (g)	AEP (g)	Modifier (vol %)	K_{IC} (MPa m ^{0.5})
Neat	187	46	0	1.45
SGS 6000	187	46	5.0	1.41
SGS 6000	187	46	10.0	1.46
SGS 3000	187	46	2.5	1.19
SGS 3000	187	46 <td>5.0</td> <td>1.19</td>	5.0	1.19
SGS 3000	187	46	7.5	2.05
SGS 3000	187	46	10.0	2.33

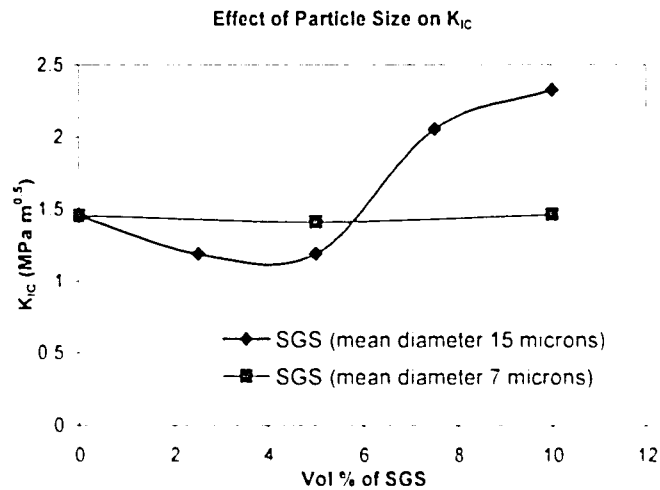


Fig. 2.2 Effect of particle size of SGS on the fracture toughness of the DGEBA/AEP system

2.4.3 Nano-material and hybrid toughened epoxy:

Table 2.5 shows the results of adding CTBN and nano-material into the DER331/AEP system.

The fracture toughness is increased by adding nano-particles and CTBN. The toughness of epoxy is improved further by the modification of adding nanoparticles. In the hybrid system, the composite with nanoparticles has higher fracture toughness than the ones that only have CTBN. It shows that CTBN and the nanoparticle have a synergistic effect on the epoxy toughening. Compared with Azimi's work [19] in which CTBN and glass spheres were added to increase fracture toughness, the synergistic effect can be got at a lower modifier content in our system.

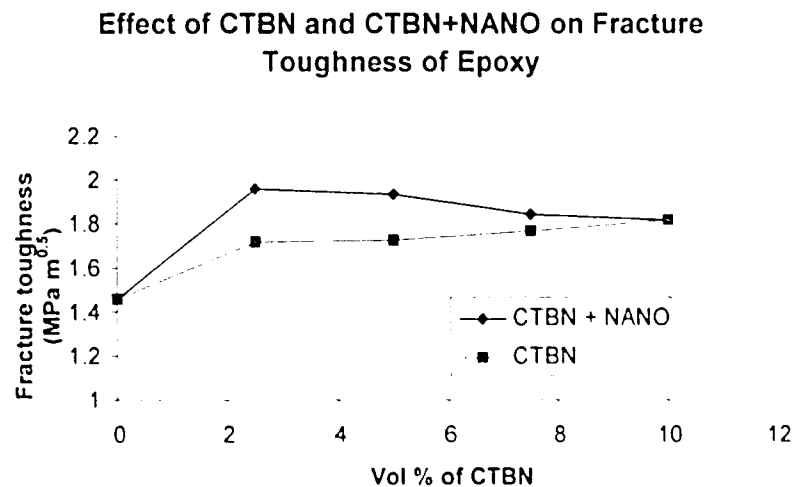


Fig 2.3 Effect of hybrid modifier (CTBN and NANO) on the fracture toughness

**Table 2.5 Fracture toughness values of CTBN and Nano-particles
modified epoxy (DER331/AEP)**

Modifier (vol %)	Fracture toughness (MPa m ^{0.5})
Neat epoxy	1.45
CTBN (2.5)	1.72
CTBN (5.0)	1.72
CTBN (7.5)	1.76
CTBN (10)	1.82
Nano-material (0.25)	1.57
Nano-material (0.5)	1.61
Nano-material (2.5)	1.80
Nano-material (5.0)	1.40
CTBN (2.5) + Nano (2.5)	1.96
CTBN (5.0) + Nano (2.5)	1.93
CTBN (7.5) + Nano (1.1)	1.84

2. 5 Summary and Conclusions

Several modifiers have been used to toughen DGEBA epoxy. Neat epoxy

cured with different curing agent may result very different fracture toughness. SGS, CTBN and Nano-material can increase the toughness of epoxy. SGS with mean diameter 15 microns is more effective than the smaller glass spheres. Hybrid epoxy composites with CTBN and nano-material were studied. There is a synergistic effect in CTBN and nano-material toughened epoxy. Compared with CTBN/glass sphere hybrid composites, the synergistic effect can be got at lower modifier content.

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Part II

Chapter 3 Study of Disbond Initiation by using Stress Singularity Approach

3.1. Introduction

Polymeric encapsulants are widely used in the automotive, aerospace, and electronic devices. The encapsulants can keep a component in space and protect it from damage. Nowadays product trends such as chip scale packaging and high density electronic interconnect are driving technologies towards increased complexity and reduced feature size, while the likelihood of smaller disbond causing failure increases. Electronic failure due to delamination in electronic component draws much attention from both academic and industry. For example, in a typical flip-chip assembly, delamination can pass three rows of solder joints after 200 thermal cycles (Figure 3.1). However, there is no apparent delamination generated until 100 thermal cycles. In fact many electronic failures happen soon after the initiation starts. It would be very helpful if we can have a standard method to study the initiation phenomena.

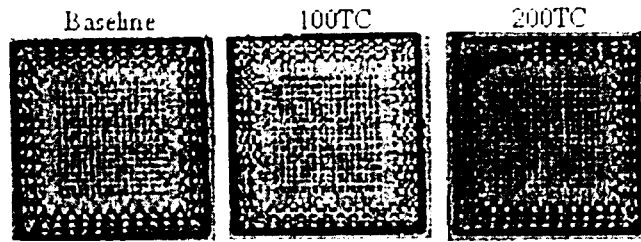


Figure 3.1 Illustration of flip-clip delamination by thermal cycles (Courtesy of David Peterson, Sandia National Laboratories)

Currently standard test methods such as double cantilever beam and four-point bending tests are widely accepted to quantify adhesion in microelectronic packaging. Both tests are based on a fracture mechanics approach. In this study, we try to use singularity approach to study disbond initiation.

There are several methods to predict the strength of bonded joints: using shear-lab-based, elastic-plastic stress analyses for bond stress and strain [1]; detailed finite element analysis of a joint and linear-elastic fracture mechanics approach using elastic-plastic adhesive material model [2]; linear elastic fracture mechanics concepts are also applied for predicting strength of bonded joints [3,4]. Stress singularity approach can also be used to predict the strength of adhesive joints [5].

3.1.1 STRESS SINGULARITY

3.1.1.1 Critical Stress Singularity Factor

Singularity is a point at which the derivative of a given function of a complex variable does not exist, but the derivative of every neighborhood exists.

The interface of adhesively bonded materials has a stress system in the vicinity of the free surface under loading. In such regions, high interface stresses can be produced. Stress singularity that exists at the interface corners or edges between the adherend and the adhesive layer might lead to adherend-adhesive debonding. A stress singularity of type Kr^δ ($\delta < 0$) exists at the interface corner between bonded elastic quarter planes [6,7,8]. The intensity of this stress singularity, referred to here as the free-edge stress intensity factor K_f , characterizes the magnitude of the stress state in the region of the interface corner. Several experimental studies have investigated the use of a free-edge stress intensity factor as a failure criterion for bonded materials, and these studies lend some support to its use [9,10,11]. K_{FC} refers the critical free edge stress intensity factor.

$$K_{FC} = \sigma^* h^{1-\lambda} A_s(\nu)$$

where σ^* is the characteristic stress, h is layer half thickness, $1-\lambda$ is the

inverse order of the stress singularity, and $A_s(v)$ is a function of shear loading [10].

3.1.1.2 Shear Butt Joint Test

Some work has been done for tensile butt joint test. [10-15] However, there are limited [7,9] references for shear test by using stress singularity approach. In one flip chip package, shear force is the primary reason to cause delamination due to thermal cycles and CTE mismatch. Therefore it is important to investigate disbond initiation behavior under shear load.

Extreme conditions such as high temperature or high humidity environment will weaken adhesion at the interfaces [15]. In many cases, the electronic component has to work under severe conditions. So investigation on moisture effects becomes very important. The yield strength and cohesive strength can be affected due to poor adhesion after moisture exposure. Cohesive strength and yield strength are related to energy release rate through a decohesive process occurring at the crack tip.

Many references illustrated the effect of water on bonded joints where the interfacial regions suffered attack and resulted a significant weakening of the

joint. [15-20] Unstressed joints can have almost 100% of adhesion drop under hot/wet condition for a long time (several years). The weakening effect in stressed situation is even worse [18]. Thus, moisture effect is an important factor when considering a joint's lifetime.

3.2. OBJECTIVES

To quantitatively evaluate the stress intensity under shear load and determine the onset of disbond initiation under shear loading conditions. The objectives also include to predict bond failure for certain geometries by using critical stress intensity factor obtained from the methodology established. Also investigate the effect of moisture on yield strength and cohesive strength of underfill epoxies.

3.3. EXPERIMENTAL APPROACH

3.3.1 MATERIALS

Most of the commercial underfill systems are complex blends of epoxy resins, curing agents and additives. To simplify the systems of the study, model epoxy systems were used to eliminate the factors from the additives. Besides the simplified model systems, several commercial underfill materials

will also be used this study to examine the established methodology and also try to solve the real world problems.

Model system I is cycloaliphatic resin with anhydride curing agent. The curing condition is at 125°C for 3 hours and then post cure at 200°C for 1 hours. Besides the neat resin, this model system will also be filled with 40% fillers (2 μ glass spheres) resembles commercial underfill resin Dexter FP4531. Model system II is based on a diglycidyl ether of Bisphenol F epoxy (Bisphenol F) with 2, 4 EMI curing agent. The curing condition is at 60°C for 4 hours then post cure at 150°C for 2 hour. This model system filled with 40% fillers (2 μ glass spheres) resembles the commercial under fill resin Zymet X6-82-5LV. The Structures of the chemicals used in the model systems are listed in Table 3.1.

The model systems and Zymet X6-82-5LV are provided by Zymet. The resins and curing agents were premixed and then stored in freezer at -40°C. Underfill resin Dexter FP4531 and Loctite 3563 are provided by Loctite. The curing conditions are: Dexter FP 4531 is at 165°C for 7 minutes, Loctite 3563 is at 150°C for 5 minutes, and Zymet X6-82-5LV is at 150°C for 15 minutes.

The disposal aluminum plates for shear butt joint tests are aluminum 6061. The dimensions of the plates are shown in the following figure (Figure

3.2).

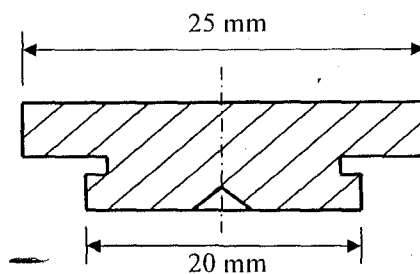


Figure 3.2 Scheme of the Disposal Plate

Table 3.1 The structures of the chemicals used in the model systems.

Chemical Name	Chemical Structure
Cycloaliphatic Resin	
Anhydride	
Diglycidyl ether of Bisphenol F epoxy	
2-Ethyl-4-methyl-imidazol	

3.3.2 EXPERIMENTAL APPROACH

3.3.2.1 Shear Butt Joint Test with epoxy/aluminum and epoxy/stainless steel interfaces

The substrate material for the two cylindrical adherends is 6061-T6 aluminum and stainless steel 446. The two plates are identically machined to a 25.0 mm diameter and a 1.5 mm height with a 2.5 mm ridge machined on the backside for gripping the adherends with the shear testing fixtures. The front side is polished down to 1 μ m, with careful attention so that the edge remains sharp and is not broken.

A clamped collar fixture, as shown in Figure 3.3, aligns and locks three samples in place with a gap of 250 μ m using shim stock placed in four machined holes along the circumference of each sample, with each set of adherends placed with the coated surfaces facing in. The shims are removed after the collar is locked and underfill is flowed in one of the four shim holes, with the others serving for air evacuation. The sample is placed in a circulating air oven at 80°C for 1 hour to equilibrate thermally. The underfill resin is flowed with the fixture lying vertically and the holes are sealed. The sample is then cured with the fixture mounted vertically in the circulating air oven. The cured sample then is tested under shear load by using a screw-driven Instron.

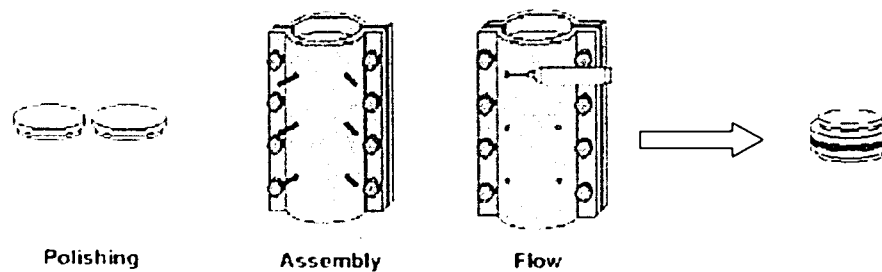


Figure 3.3 Shear butt joint sample preparation.

The following are some preliminary results from shear butt joint tests of various underfill. Table 3.2 listed the value of critical free edge stress intensity factor, K_{FC} , from the shear butt joint test. The K_{FC} of the filled system of Bisphenol F/2,4-EMI is slightly higher than the unfilled system; however, the K_{FC} of the filled system of Cycloaliphatic/Anhydride is much lower than the unfilled system. These trends for both materials are consistent with the butt tensile test and DCB test results. The reason for the decrease of K_{FC} due to the filler was not known yet.

Among the three commercial underfill materials, K_{FC} of Loctite 3563 is the highest, while Dexter FP4531 is much lower than the other materials.

Compare the K_{FC} of shear and tensile test results, only the values of Dexter FP4531 are close for both tests. All the other materials have a much higher value for shear butt joints than the tensile butt joints. The reason could be that the yielding zone for shear test is much larger than the tensile test.

Table 3.2 K_{FC} results of shear butt joint tests.

Underfill	$K_{FC}(\text{MPa}\cdot\text{m}^{0.268})$
Bisphenol F/2,4-EMI (Unfilled)	36.7
Bisphenol F/2,4-EMI (Filled)	40.4
Cycloaliphatic/ Anhydride (Unfilled)	36.0
Cycloaliphatic/ Anhydride (Filled)	11.0
Loctite 3563	41.19
Dexter FP 4531	6.26
Zymet X6-82-5LV	28.51

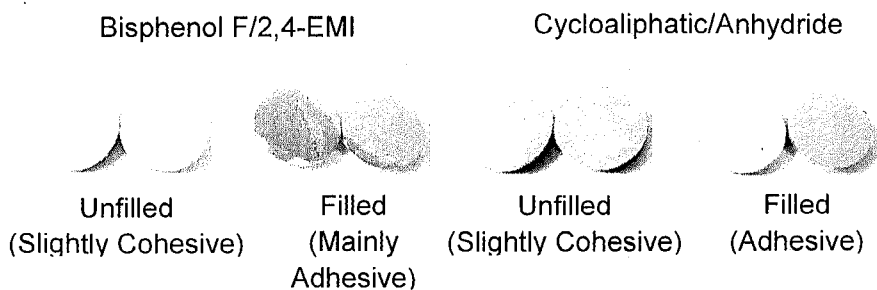


Figure 3.4 Fracture surfaces of butt joint samples

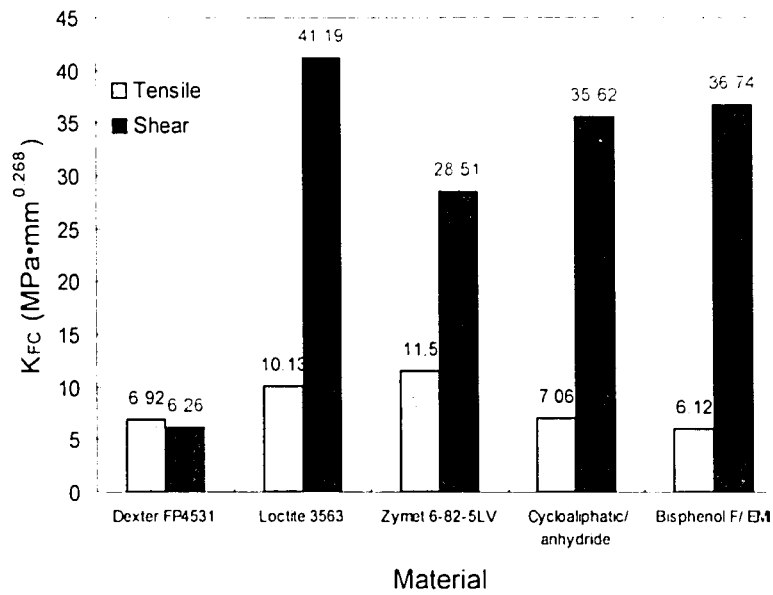


Figure 3.5 Comparison of K_{FC} under tensile load and shear load.

Table 3.3 K_{FC} results of tensile butt joint test of underfill resins [21]

Underfill	K_{FC} (MPa·m ^{0.268})
Bisphenol F/2,4-EMI (Unfilled)	6.12
Cycloaliphatic/ Anhydride (Unfilled)	7.06
Loctite 3563	10.30
Dexter FP 4531	6.92
Zymet X6-82-5LV	11.50

K_{FC} is associated with the interface corner. It should not be affected by the characteristic length scale or cross section area. Reedy and Guess have shown that the observed deduction in the tensile strength of adhesively bonded cylindrical butt joints with increasing bond thickness is predicted by a K_{fc} criterion (37 - 26). So if the characteristic length scale h is changed, K_{FC} should still be the same value. Table 3.4 shows the K_{FC} values of Zymet X6-82-5LV/Aluminum interfaces obtained from the shear butt joint test with different characteristic length scale. It can be seen that the K_{FC} values are consistent at various h .

Table 3.4 . Result of K_{FC} values of Zymet X6-82-5LV/Aluminum interfaces with various characteristic length scale.

h (in)	$K_{FC}(\text{MPa}\cdot\text{m}^{0.268})$
0.0060	35.8
0.0120	34.9
0.0125	36.0
0.0140	29.4
0.0210	31.4

3.3.2.2 Effect of Moisture on Cohesive Strength and Yield Strength

Test specimens for weight gain in moisture uptake and yield strength test

are prepared as following procedure. The epoxies will be cured at the corresponding conditions and then machined to dimensions of 6.0 mm × 6.0 mm × 12.7 mm. Test specimens were kept in a humidity chamber set at 85°C and 85% relative humidity for more than 2000 hours in order to ensure equilibrium moisture uptake. Specimens were weighed periodically for the purpose of monitoring moisture uptake. The weight gain $M(t)$ is calculated by using the following equation:

$$M(t) = \frac{W - W_0}{W_0}$$

where W_0 and W are the initial weight and the weight at time t , respectively.

The following are some preliminary results of Task III. Weight gain and yield strength results are shown in Figure 3. and Figure 3.. Figure 3. shows the curves of the yield strength before and after moisture uptake. Experiment results of weight gain show that the model systems pick up moisture faster than the commercial systems. That is because the commercial systems have very high filler contents, which are around 60% to 80%. So the moisture uptake of those commercial systems is less than the model systems. For the same reason, lower diffusion coefficient values are expected for the commercial systems than the model systems. Loctite 3563 has higher moisture absorption than the other systems. This could be a result of a weaker interface adhesion between its matrix and filler. After moisture exposure, the

matrix and filler disbond. So there are more voids in the system to absorb additional water.

At early stage of the weight gain experiments, the absorption curves in Figure 3. are linear with the square root of time. It is well known that the linear region can be explained using Ficks second law of diffusion (27), and the diffusion coefficient D can be calculated from the diffusion curve by using the equation (28):

$$D = \pi \left(\frac{sb}{4M_s} \right)^2$$

where s is the slope of the diffusion curve in the early stage linear region:
 b is the thickness of the specimen. M_s is the moisture content at saturation.

3.4 Results and discussions

In Figure 3.7 and Figure 3.8 the moisture uptake doesn't have much effect on the stress-strain curves or the yield stress of the two model systems. For commercial systems Loctite 3563 and Dexter FP 4531, the moisture uptake causes a small percentage loss of yield strength. Zymet X6-82-5LV has the biggest drop on yield strength among all five materials. Also, the three commercial underfill materials all have a noticeable loss of ultimate strength after moisture uptake.

Figure 3.9 contains the apparent diffusion coefficients. The diffusion

coefficients are lower for the filled systems, as expected.

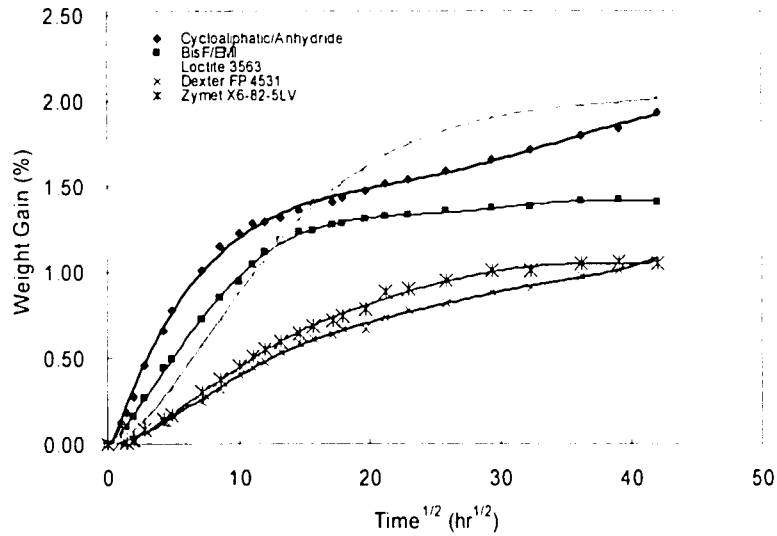


Figure 3.6. Weight gain as a function of time of underfill materials under the exposure of 85°C /85% RH

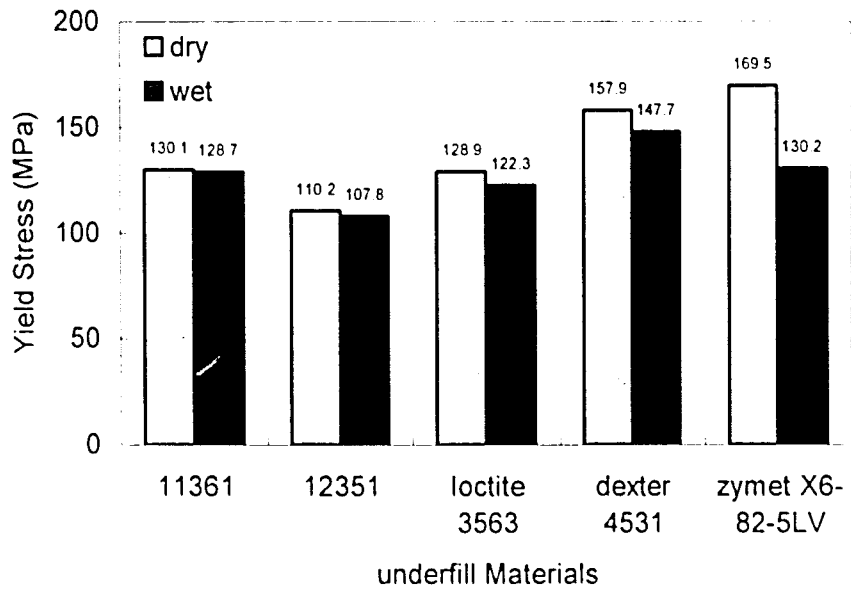
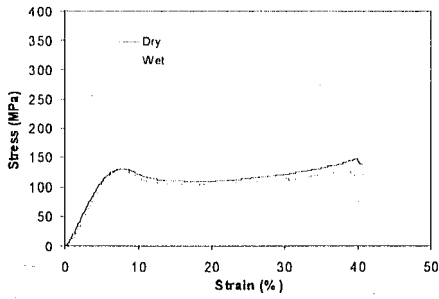
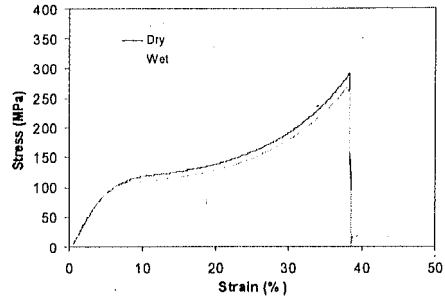


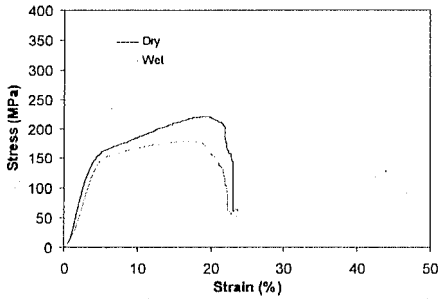
Figure 3.7 The yield strength of various epoxy system before and after moisture pickup.



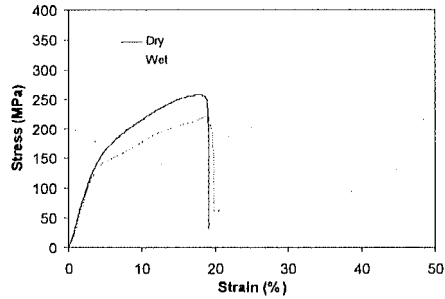
Cycloaliphatic/anhydride



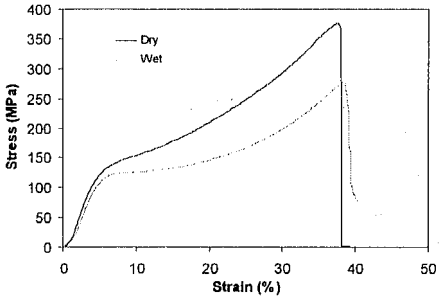
Bisphenol F/2,4-EMI



Loctite 3563



Dexter FP 4531



Zymet X6-82-5LV

Figure 3.8 Stress-strain curve of underfill materials before and after

85°C/85%RH moisture exposure.

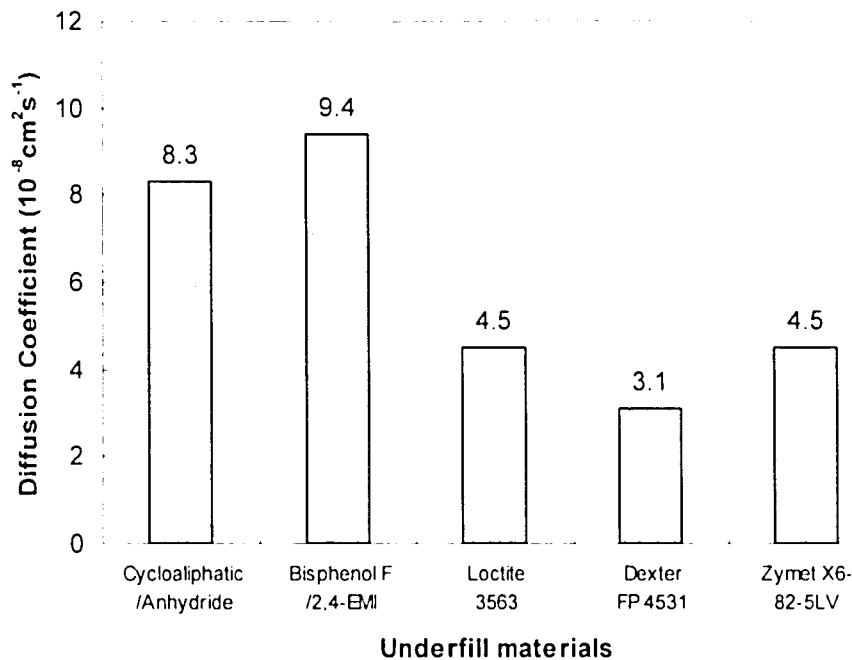


Figure 3.9 Results at 85°C/85% RH of apparent diffusion coefficient of underfill systems

Yield Strength of the underfill materials are obtained by using a screw driven Instron following ASDM-695. The crosshead speed is 1mm/min. Figure 3.10 compares the yield strength of model systems and the commercial underfill materials. In Figure 3.10, the model system I cycloaliphatic/Anhydride shows a higher yield strength than the model system II Bisphenol F/2,4 EMI. Among the 3 commercial systems, the yield strengths of Dexter FP 4531 and Zymet X6-82-5LV are quite close. Loctite 3563 has the lowest yield strength in the 3 commercial underfill materials. Possible reason

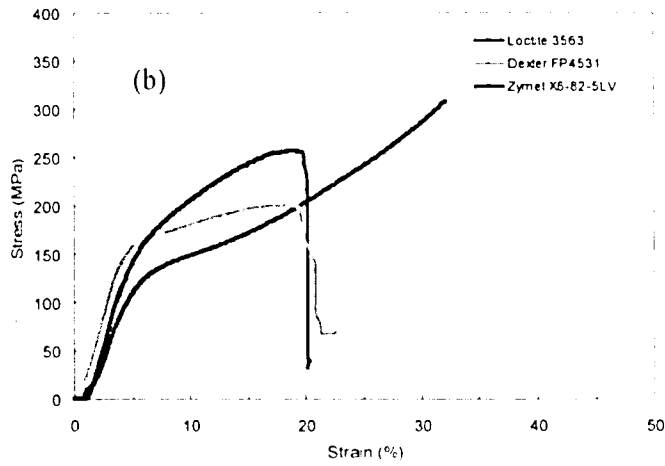
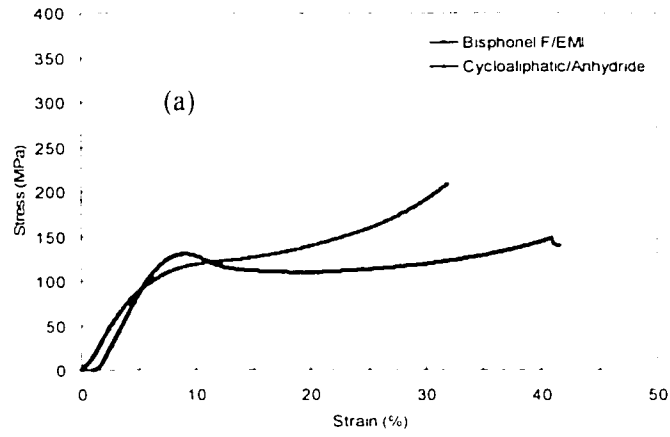


Figure 3.10 Stress-strain curve of (a) model systems (b) commercial systems.

is the adhesion of the fillers and epoxy is weaker in Loctite 3563. That's also the reason for its highest percentage of moisture uptake. The color of the Loctite 3563 specimens become lighter after the moisture uptake, while all the other specimens do not have obvious change in color.

Comparing Figure 3.10(a) and (b), the model systems have lower yield strength than the commercial systems. This result is expected, as the commercial systems have very high filler contents while the two model systems don't have any filler.

3.5 Summary and Conclusions

Free-edge stress intensity factor (K_{FC}) of five different materials (including two model systems and three commercial materials) has been quantitatively evaluated by shear testing.

Differences in critical stress intensity was found for some materials when comparing the values obtained from shear or tensile tests. The reason could be that the yielding zone is large compared to the singularity controlled area in shear tests (to be confirmed).

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Vita

Xiaohan Zhang was born in February, 1975 in Tianjin, China, to Mr. Guoning Zhang and Ms. Xiuzhi Dong. In 1993, she went to Chemical Engineering Department in Tsinghua University, Beijing. In 1998, she received Bachelor Degree in Chemical Engineering. She was admitted by the Polymer Science and Engineering Program of Lehigh University under the supervision of Prof. Raymond A. Pearson in 2000. In Jan. 2007, she got a M.S. degree on the study of crack initiation and propagation of epoxy bulk and epoxy/aluminum interfaces. She completed her research project on the study of adhesion loss at epoxy/glass interfaces under hygrothermal conditions and received her Ph. D. degree in Jan. 2007. She received Graduate Student Merit Award from Lehigh University in 2004.

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