

## Chapter 1

### INTRODUCTION

With the increasing use of structural adhesives in civil construction, micro-electronic packaging, and the aerospace and automotive industries, the need for understanding the failure behavior of adhesively bonded joints is greater than ever. Since cracks and flaws are inevitable in materials and their propagation will result in unexpected failures, predictions of the locus of failure and crack propagation behavior in adhesive bonds are essential to understand and improve the durability of the bonded joints.

As shown in Fig. 1, four types of failure have been observed in mode I tests of double cantilever beam (DCB) specimens using aluminum adherends and an epoxy adhesive (details of material specifications and specimen fabrication are given in the next section). These modes are: a) cohesive failure, where the crack propagates along the middle of adhesive layer; b) interfacial failure (visually), where the failure occurs at the interface between adhesive and adherends; c) oscillatory failure, where the trajectory of the crack oscillates about the midplane of the bond but remains within the adhesive layer; and d) alternating failure, where the crack alternates between the two adhesive/adherend interfaces. Similar phenomena had also been observed by Wang and Suo [1], Cao and Evans [2], and Chai [3-5] in different systems such as 420 stainless steel/epoxy, plexiglass/epoxy and fiber-reinforced epoxy laminates.

Fig. 1 shows different failure locations, either within the adhesive layer (a and c) or at/near the interface (b and d), as well as different crack trajectories, either directionally stable (a and b) or directionally unstable (b and d). This information leads to two important issues closely related to the fundamental mechanism of crack path selection in adhesive bonds: locus of failure and directional stability of cracks.

To predict the locus of failure, conventional wisdom suggests that materials always fail at the weakest location. However, Dillard *et al.* [6] indicated that the locus of failure, while closely related to material properties such as tensile strength, quality of adhesion at the interface, and fracture toughness of the bonds, depends also on the stress state at the crack tip. Consequently, the final locus of failure is the result of the competition between the material properties and the stress state within the system. Supportive information can also be found in Cao and Evans [2], and Akisanya and Fleck [7] where both the locus of failure and the crack propagation behavior were shown to be dependent on the mode mixity of external loads.

Mechanically, the locus of failure is closely related to the direction of crack propagation, which has been demonstrated to be dependent on the stress and energy state at the crack-tip [8-11]. According to the theory of fracture mechanics, the stress state in the vicinity of a crack in a homogeneous medium is characterized by the William's asymptotic stress expansion [12]

$$\begin{aligned}
 \begin{bmatrix} \sigma_{xx} & \sigma_{xy} \\ \sigma_{xy} & \sigma_{yy} \end{bmatrix} &= \frac{K_I}{\sqrt{2\pi r}} \cos\left(\frac{\theta}{2}\right) \begin{bmatrix} 1 - \sin\left(\frac{\theta}{2}\right) \sin\left(\frac{3\theta}{2}\right) & \sin\left(\frac{\theta}{2}\right) \sin\left(\frac{3\theta}{2}\right) \\ \sin\left(\frac{\theta}{2}\right) \sin\left(\frac{3\theta}{2}\right) & 1 + \sin\left(\frac{\theta}{2}\right) \sin\left(\frac{3\theta}{2}\right) \end{bmatrix} \\
 + \frac{K_{II}}{\sqrt{2\pi r}} &\begin{bmatrix} -\sin\left(\frac{\theta}{2}\right) \left[ 2 + \cos\left(\frac{\theta}{2}\right) \cos\left(\frac{3\theta}{2}\right) \right] & \cos\left(\frac{\theta}{2}\right) \left[ 1 - \sin\left(\frac{\theta}{2}\right) \sin\left(\frac{3\theta}{2}\right) \right] \\ \cos\left(\frac{\theta}{2}\right) \left[ 1 - \sin\left(\frac{\theta}{2}\right) \sin\left(\frac{3\theta}{2}\right) \right] & \sin\left(\frac{\theta}{2}\right) \cos\left(\frac{\theta}{2}\right) \cos\left(\frac{3\theta}{2}\right) \end{bmatrix} \\
 + \begin{bmatrix} T & 0 \\ 0 & 0 \end{bmatrix} &+ O(\sqrt{r})
 \end{aligned} \tag{Equation 1}$$

where the coordinate is set at the crack tip with x-axis pointing along the crack plane, and  $r$  and  $\theta$  are the polar coordinates. In Equation 1,  $K_I$  and  $K_{II}$  are the mode I and mode II stress intensity factors, respectively, and the terms with  $K_I$  and  $K_{II}$  are singular stresses. The term with  $T$  in Equation 1 is non-singular and only the component along the crack plane is not zero. By convention, this non-zero stress component is referred as the T-stress. In the vicinity of the crack-tip, the stress state is dominated by the singular terms and consequently, the onset of fracture is characterized by the magnitude of the stress intensity factors.

To determine the direction of crack propagation, over the years, several criteria have been developed to determine the direction of crack propagation for cracks in brittle homogeneous isotropic solids. Among these criteria, three primary ones have been widely discussed in the literature; namely,

1. **Maximum opening stress criterion** (by Ergodan and Sih, (1963) [8]). This criterion dictates that the direction of cracking is perpendicular to the direction of maximum opening stress at the crack tip. Shown in Fig. 2 is a crack under a pure shear loading. Using this criterion, the direction of crack propagation is determined as schematically shown in the figure.
2. **Maximum energy release rate criterion** (by Palaniswamy and Knauss, (1978) [9]). By applying this criterion, the direction of crack propagation can be obtained by maximizing the energy release rate as a function of the angle of crack kinking as schematically shown in Fig. 3.
3. **Mode I fracture criterion** (by Goldstein and Salganik (1974) [10] and Cotterell and Rice (1980) [11]). According to this criterion, a crack will propagate along a path such

that pure mode I fracture is maintained at the crack tip, i.e.  $K_{II} = 0$  at the growing crack tip as shown in Fig. 4.

Although the three criteria specify different aspects, they all yield similar results and no experimentally distinguishable differences have been observed [7, 13, 14].

These criteria, although developed primarily for cracks in homogeneous materials, can be readily extended into bi-materials systems such as adhesively bonded joints. However, care should be used when applying these criteria to determine the direction of cracking for cracks located at a bi-material interface due to differences in fracture toughness in the vicinity of an interface. For adhesively bonded joints, Chen and Dillard [15] provided an example showing how to use these criteria to determine the direction of cracking when the crack is located at the interface.

According to these criteria, a crack in an adhesive bond can be steered to different locations if the local stress state at the crack tip is in mixed mode. Consequently, various failure locations can result and failure does not necessarily occur at the weakest site within the material, which conflicts with conventional wisdom. In this dissertation, various aspects that influence the locus of failure in adhesive bonds are investigated extensively. Using post-failure analysis methods such as x-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM), the locus of failure is characterized accurately, and the effects of fracture mode mixity and rate of crack propagation on the locus of failure in adhesive bonds are investigated. Through preparing the specimens with various surface preparation techniques, the effect of interface properties on the locus of failure is also studied, and the results demonstrated that the locus of failure is closely related to the external loading conditions as well as the materials properties of the system.

To predict the directional stability of crack propagation, Cotterell and Rice [11] showed further information other than the stress intensity factors is necessary. In investigating slightly curved or kinked cracks in homogeneous materials, Cotterell and Rice [11] showed that the T-stress plays an important role in the directional stability of crack propagation. When the loading is predominately mode I, curved or kinked cracks will converge back to the original path resulting in a directionally stable crack trajectory if the T-stress is negative (compressive). On the other hand, the crack will deviate further away from the original path and the resultant crack path is directionally unstable if the T-stress is positive (tensile). Through considering higher order terms in the William's asymptotic stress expansion for a crack in homogeneous materials [12], Chao *et al.* [16, 17] further investigated the effect of the T-stress on the crack propagation manner. The results indicated that the direction of the maximum opening stress at a crack tip varies with the T-stress level and consequently, so does the direction of crack propagation according criterion 1. The study of Chao *et al.* [16, 17] provided important insights into understanding the directional stability of cracks in homogeneous materials and the fundamental physics of the T-stress effect.

The discussion of directional stability of cracks in adhesively bonded joints was first emphasized by Chai [3-5], who described a unique crack trajectory in the mode I delamination failure of graphite reinforced epoxy composite laminates and aluminum/epoxy bonds. Similar to the crack trajectory in specimen d) of Fig. 1, overall, the crack periodically alternated between the two interfaces with a characteristic length of 3-4 times the thickness of the adhesive layer. More specifically, as the crack advanced, the crack propagated along one interface and then gradually deviated away with an increasing slope until the other interface was approached. An abrupt kink then occurred when the crack approached the opposite interface and the crack stayed at the interface for a distance about 2-3 times the thickness of the adhesive layer before deviating from the interface again. This trajectory obviously reflects very directionally unstable crack propagation. Daghyani, Ye, and Mai [18] later showed that the directional stability of cracks is closely related to the residual stress state in adhesive bonds. Fleck, Hutchinson, and Suo [19] and Akisanya and Fleck [7, 20] investigated this directional stability issue analytically and indicated that as with homogeneous materials, the directional stability of cracks in adhesively bonded joints also depends on the T-stress level. Cracks in adhesive bonds tend to be directionally stable if the T-stress is negative and tend to be directionally unstable if the T-stress is positive.

Chen and Dillard [21] experimentally verified the prediction and demonstrated the T-stress dependence of the directional stability of cracks in adhesive bonds through mechanically altering the T-stress levels in DCB specimens. Details of the T-stress calculation and alteration procedure for DCB specimens can be found in reference 21. Fleck, Hutchinson, and Suo [19] and Chen and Dillard [21] also showed that the T-stress is closely related to the specimen geometry i.e. the thickness of the adhesive layer and the thickness of adherends for DCB specimens. The T-stress increases with the thickness of the adhesive but decreases with the thickness of the adherend for this bonding geometry. This specimen geometry dependence of the T-stress level suggested a variation of the directional stability of cracks as the specimen geometry changes and the experimental results in reference 21 on DCB specimens with different adherend thicknesses verified the prediction.

This dissertation is to investigate the different aspects of the directional stability of cracks in adhesively bonded joints, and each aspect will be discussed in detail in the following four chapters. Each of the four main chapters forms an independent study and is written in the form required for formal publication in various journals. Consequently, some information may be repeated in each chapter for the purpose of independent publication. The material systems used in this research are Dow Chemical epoxy resin DER 331<sup>®</sup> with various levels of rubber concentration as adhesive, and aluminum 6061-T6 alloy with various thicknesses as adherends.

Chapter 2 discusses the effect of the T-stress on the directional stability of cracks in adhesively bonded joints. Using the finite element analysis (FEA) method, the T-stress for DCB specimens is

calculated and comparison is made with the analytical solution obtained by Fleck, Hutchinson, and Suo [1] for the bi-materials sandwich geometry with semi-infinite adherends. Various T-stress levels are achieved in double cantilever beam (DCB) specimens through mechanically stretching the specimens uniaxially until the adherends are plastically deformed. Mode I test results demonstrate that cracks tend to be directionally stable when the T-stress is negative (compressive) and directionally unstable when the T-stress is positive (tensile). The FEA results also show that the T-stress increases as the thickness of the adherends decreases, indicating a mild effect of adherend bending on the directional stability of cracks. This prediction is verified in this paper using DCB specimens with different thicknesses adherends. The coauthor of this chapter is David A. Dillard and the paper formed by this chapter will be submitted for the publication in the *International Journal of Adhesion and Adhesives*.

In Chapter 3, based on the energy balance concept, an analytical model is proposed analyzing energy flows in adhesively bonded DCB specimens to predict the directional stability of cracks. The results are consistent with the analytical predictions made by Fleck, Hutchinson, and Suo [1] using a stress analysis approach, and also are consistent with the experimental observations in references 5, 6, and 21. Both interface mechanics and the finite element method are then employed to analyze the stress state at the crack tip and to predict crack trajectories. Through extending the criteria for direction of crack propagation to bi-material systems, the trajectory for directionally unstable cracks is simulated. The simulation results accurately reflect the alternating nature of directionally unstable cracks in adhesive bonds such as the characteristic length and the overall shape of the crack. The coauthor of this chapter is David A. Dillard and the paper formed by this chapter will be submitted for the publication in the *International Journal of Solids and Structures*.

A paper prepared for submission to the *International Journal of Fracture* forms Chapter 4, and the coauthors are David A. Dillard, John G. Dillard, and Richard L. Clark, Jr. This study investigates the roles of external loads and specimen geometry in crack path selection in adhesively bonded joints. Through testing DCB specimens (both symmetric and asymmetric) under mixed-mode fracture loading and conducting post-failure analysis on the failure surfaces, the fracture mode dependence of the locus of failure and directional stability of cracks is demonstrated. Post-failure analyses on the failure surfaces suggest that the failure tends to be more interfacial as the mode II fracture component in the loading increases, and the direction of debond is mostly stabilized when more than 3% of mode II fracture component is present. Using a high-speed camera to monitor the fracture sequence in both quasi-static and low-speed impact tests, the effect of debond rate on the locus of failure and on directional stability of cracks is also investigated. Post-failure analyses including XPS, Auger electron spectroscopic depth profile, and scanning electron microscopy indicate that as the debond rate increases, the failure tends to be more cohesive and the cracks tend to be more directionally unstable. Last, as indicated by the finite

element analyses results, the T-stresses, and therefore the directional stability of cracks in adhesive bonds are closely related to the thickness of adhesive layer and also to the thickness of the adherend. This specimen geometry dependence of crack path selection is studied analytically and is verified experimentally.

As a continued study of Chapter 4, Chapter 5, which is prepared along with coauthors John G. Dillard, David A. Dillard, and Richard L. Clark, Jr. for submission to the *Journal of Adhesion*, discusses the effects of material properties on crack path selection in adhesive bonds. First, the material system dependence of directionally unstable cracks is investigated through a parametric study and alternating crack trajectories are simulated for adhesive bonds with different material systems. Second, DCB specimens with substrates treated using various surface preparation methods are tested under mixed mode fracture loading, and the effect of interface properties on crack path selections is investigated. Also in this study, through varying the rubber content in the adhesive, DCB specimens with various fracture toughnesses are prepared and the effect of adhesive toughness on the directional stability of cracks is investigated. The results are consistent with the predictions made using the energy balance model discussed in reference 15. The future work of this study is discussed in Chapter 6.

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