Relating Polymer Matrix Composite Delamination Behavior to Constituent Properties

by

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Submitted to the Department of Mechanical Engineering in partial fulfillment of the requirements for the degree of

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Abstract

Developing predictive capabilities of composite material behavior from constituent properties is an important component of accelerating materials insertion. Many models exist that accomplish this objective for a range of material properties, but no such method is available for delamination properties. This thesis explores the issues associated with predicting polymer matrix composite Mode I delamination behavior from constituent properties by examining the topic from a variety of perspectives. Nanoindentation tests of matrix materials in composites and their associated neat polymers analyze the assumption that neat matrix properties are the same as unconstrained in situ properties and therefore may be used in models predicting composite behavior from constituent properties. Quasi-static and fatigue fracture experiments using a graphite/epoxy composite and its neat resin at a variety of temperatures and loading rates allow for an examination of the dominant mechanisms involved in the fracture process, an analysis of the shifts in quasi-static behavior with temperature and potential implications for fatigue predictions, and measurement of values that will act as inputs and verification of a delamination initiation model. A global-local finite element model of a double cantilever beam specimen is used to study the prediction of delamination initiation by examining inelastic matrix deformation at the crack tip. In addition, a fiber bridging fatigue model is created to analyze crack propagation data that effectively separates the bridging and resin crack tip contributions. The final component of the thesis is to tie the various experimental and analytical studies together to create methodologies that may be used in a design or research environment to accelerate materials insertion. An important conclusion from the thesis is that quantitative predictions of composite fracture behavior using unmodified neat matrix properties is not feasible, but the constrained matrix properties may be used to predict composite delamination behavior. It is also shown that following a process of implementing mechanism-based models in conjunction with experimental observations is essential when implementing models that bridge lengthscales.
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Although there is only one author on the title page, this thesis clearly would not have been completed without the efforts and support of a significant number of people. I must begin by thanking my advisor, Mark Spearing for his guidance, patience, and good humor throughout this process. My temperament has apparently caused him to flee the country, but it was fun while it lasted. I also would like to express my gratitude to the chair of my thesis committee, Professor Argon. I am forever in his debt for taking a chance on bringing a kid from the cow country of Montana to MIT. My other two committee members, Simona Socrate and Paul Lagace, were continuously supportive of my efforts throughout the research and I am extremely grateful for their assistance. My only regret is that I was not able to find a way for my research to bring the World Series Title to the Red Sox as a way of getting Paul to allow me to graduate early. Better luck next time.

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Foreword

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Chapter 1

Introduction

Insertion of materials into aerospace programs requires an enormous expenditure of time and resources. Aerospace companies typically spend dozens of millions of dollars and anywhere between five and fifteen years in order to properly characterize a material for design usage and the subsequent insertion into the desired product [e.g. 1]. This characterization involves the generation of large design-allowable databases from coupon, sub-element, element, and component testing in a sequential and inflexible process that is often referred to as the Building Block Approach (BBA) [2]. The inflexibility of the process lies in the large cost associated with modifications that must be made when unexpected material behavior occurs at a higher lengthscale, such as the element or component level.

The advent of advanced composite materials and the development of improved fiber and matrix combinations has made the insertion process more critical than ever before. There is a need for an improved method of evaluating and characterizing potential composite materials that does not rely on the BBA. Members of the aerospace industry and composites community have recognized this need and organized efforts to tackle the problem. One such effort is named Accelerated Insertion of Materials-Composites (AIM-C) [1] and involves the use of analytical predictions of composite behavior to reduce the number of tests required to characterize a material’s properties. AIM-C does not propose to eliminate testing from material characterization, rather, it strives to determine which tests are essential to describing material behavior.
and which tests are less important because results can be confidently predicted or do not have a statistically significant influence on other outcomes.

The ultimate goal of AIM-C is to develop a methodology to accelerate material insertion into aerospace products. This methodology would be applicable to new materials that are under consideration and for existing materials that are being used in new designs. This process is important because it would allow designers to evaluate the potential capabilities and design applicability of the candidate material and its constituents in parallel with the associated experiments, as opposed to the sequential process of the BBA.

Analytical tools are the primary mechanism used by AIM-C to predict composite behavior. In particular, the effort takes advantage of the natural lengthscales embodied in composite materials ranging from the constituent level to the ply, laminate and structural levels. An advantage of such a framework is that one may examine the effects that constituent properties have on behavior at higher lengthscales. Models that predict composite behavior from constituent properties are particularly powerful in analyses that examine the statistical significance of a selection of fiber and resin properties on composite properties. Such studies are an important tool in determining the essential experiments that need to be conducted to predict material behavior accurately.

Numerous models are available to predict composite behavior from constituent properties. Models calculating elastic and hygrothermal properties have been available for decades [3, 4]. In addition, strength [5], durability [6], and processing models [7] are increasingly relying on the individual properties of the fibers and the matrix to predict composite behavior. An important area that is lacking predictive capabilities is in the determination of composite delamination behavior from constituent properties. In addition, delamination fatigue behavior has not received much attention at the constituent level. Indeed, delamination is one of the most common defects found in manufactured composite aerospace components and damage often manifests itself in the form of matrix cracking and/or delamination. There is an increasing emphasis
on examining the effects of these defects and fracture mechanics\(^1\) is an appropriate framework to study these problems.

In spite of the importance of delamination properties, there are no methods available to predict them from resin and fiber properties. This thesis seeks to address this gap by exploring the relationship between constituent and composite quasi-static and fatigue fracture behavior and determining the feasibility of making qualitative and quantitative predictions of composite delamination properties from constituent properties. The focus of the research is limited to polymer matrix composite (PMC) Mode I delamination behavior in part because of the aforementioned prevalence of delaminations in manufactured aerospace composite components (usually made from PMCs) and also because of an effort to limit the scope of the study to allow a thorough examination of the issues surrounding Mode I delamination from a variety of perspectives. Structural delaminations generally involve a mixture of Mode I and Mode II loading. Thus, measurement of delamination properties involves experiments performed using Mode I, Mode II, and mixed-mode levels. This thesis examines Mode I loading as the first step in understanding the broader issues associated with constituent and composite mixed-mode fracture properties.

Although fracture mechanics is a logical approach to use when studying delaminations in composite structures, it is not commonly used in industry as a design tool. This is due to two main factors: a reliance on the assumption that there are no defects in the manufactured structure and a lack of understanding related to techniques implementing fracture mechanics in design. However, both of these attitudes are changing and methodologies are being developed to use fracture mechanics to study the effects of delaminations in composite structures. This thesis is an important component of a larger overall effort that must be undertaken to examine the issues related to the measurement of delamination properties and their implementation in a design

---

\(^1\)A distinction must be made between the usage of the term “fracture” in the composites community to denote ultimate strength or failure (i.e. separation) and the term “fracture mechanics”, which is the classical area of study. The former typically uses measures of stress whereas the latter uses stress intensity factors and strain energy release rates. Studies of delamination often involve the measures used in classical fracture mechanics and hence, the usage of the terms fracture and fracture mechanics in this thesis refer to the more classical definitions and do not refer to ultimate strength.
environment.

The ultimate objective of the present work is to apply the AIM-C methodology of linking properties and mechanisms over a range of lengthscales to relate the fracture behavior of the individual constituents with that of the composite. Although quantitative predictions may not be possible, the important contribution of the work will be an assessment of the capabilities of being able to use fracture measurements and mechanisms in the constituents to make predictions of composite quasi-static and fatigue Mode I delamination behavior. The increasing use of fracture mechanics in composite structural design makes these links key components of accelerating materials insertion.

This thesis is organized into chapters that examine various issues associated with linking constituent and composite fracture properties through experimental and analytical means. The approach is described in Chapter 2, which details the steps taken in the research to accomplish the overall objective. Chapter 2 also includes a review of the literature related to models that predict composite behavior from constituent properties. Chapter 3 describes nanoindentation experiments performed on neat polymers and the same polymers in composites that are used to question a basic assumption in most micromechanical models that the neat and unconstrained in situ matrix properties are the same. Quasi-static and fatigue fracture experiments using neat resin and composite materials are detailed in Chapter 4 and act as an examination of the mechanisms involved in the two materials. Models for delamination initiation and fatigue fiber bridging are presented in Chapters 5 and 6, respectively, using the experimental work as a foundation. The discussion in Chapter 7 is included to tie the work presented in the previous chapters together. Finally, Chapter 8 lists conclusions, contributions, and recommendations for future work. Appendices are included that detail some of the codes used in the modeling portions of the work. Due to the varied nature of the topics, the background and literature review for each area are reviewed at the beginning of each chapter.
Chapter 2

Background and Approach

Before describing the approach used to explore the objective of the thesis, it is useful to examine previous work that has been accomplished related to models that link lengthscales to define composite behavior. The clear distinction between the constituents within composite materials, the fibers and the matrix, has made models that predict composite behavior using constituent properties prevalent. In addition, there are several natural lengthscales present in the design of aerospace structures that contain composite materials, as shown in Figure 2-1. There are a variety of models available to connect behavior between two adjacent lengthscales (e.g. classical laminated plate theory links ply properties to laminate behavior), but there has been little consideration of the effect of these connections across the spectrum of lengthscales. This chapter first reviews the literature for models that make predictions of composite behavior from constituent properties and then reviews frameworks that have been discussed to consider issues associated with making predictions across a range of lengthscales. The literature review is followed by a description of the approach used in this thesis.

2.1 Background

The initial experimental evaluations of advanced composite materials were accompanied by models that used fiber and matrix properties to predict elastic composite
properties [8]. Countless models were created in the following years, but arguably the most comprehensive sets of models that predict composite hygrothermal and mechanical properties from constituent properties have been developed by Chamis [3] and Hashin [4]. Both sets of models were created using different theoretical approaches, but they essentially have the same inputs and outputs.

The most interesting issue surrounding the models is that there is virtually no data available validating the models' predictions for composites that contain anisotropic fibers, such as carbon fibers. While some experimental data is available for composites that use glass fibers, which are isotropic [8], the same cannot be said of carbon fibers because methods are not available to measure all the properties of anisotropic fibers. Thus, the most common practice in industry is to back out properties that cannot be measured, such as transverse and shear moduli, from composite experimental data. This obviously indicates that there is less value in using the models as predictive tools, but there is still utility in using the models to study the statistical significance of input parameters or the effects of variations in constituent properties on composite properties.

Processing models developed by Poursartip et al. have shown success at predicting processing-induced deformations in composite structures [7, 9]. They are inherently based on constituent properties and particularly the time-temperature dependence (viscoelasticity) of the resin.
Not surprisingly, many models predicting unidirectional composite strength also use the properties of the constituents. The majority of these models are similar to those created by Curtin [10-12], which predict the longitudinal strength of a unidirectional composite. They primarily use the statistical distribution of the longitudinal strength of the fibers to determine composite strength. Aerospace structures that contain composite materials nearly always use angle ply layups and thus, composite longitudinal strength is merely one consideration of many that must be used in examining structural strength.

A more recent model developed by Gosse named the Strain Invariant Failure Theory (SIFT) predicts damage in composite structures using the properties of the constituents [5]. The important inputs are the strength properties of the fibers and the constrained in situ strength properties of the matrix. Damage is determined by examining the stresses in a micromechanical finite element model of the fibers and matrix that result from globally applied loads to the modelled structure. The type of loading will determine whether damage occurs in the matrix or the fibers and whether the damage is initial, propagation, or critical. While the fiber properties can be determined from neat fiber experiments, the resin strength properties are backed out of a specific set of composite experiments. Tsai and Kuraishi have extended the method and applied SIFT to durability analyses by using the viscoelastic properties of the resin and time-temperature superposition [6]. These models are notable for their attempt to predict composite behavior using a range of lengthscales from constituent to structural levels.

Chamis and colleagues have spent a significant amount of time developing a comprehensive framework from which one may predict composite damage behavior. They have packaged their methods into a software package called COnposite Durability STructural ANalysis (CODSTRAN) [13]. The analysis framework spans several lengthscales and includes calculations from the constituent to the structural level. Numerous studies have been performed using CODSTRAN [e.g. 14] that predict damage progression from initiation to failure. The studies often focus on composite shells as a structural member [15], but there is virtually no comparison with experimental data.
One comparison with coupon-level experimental data as part of the World Wide Failure Exercise showed that the method predicted fiber-dominated fractures well and performed poorly at predicting matrix-dominated failures [16]. The authors of COD-STRAN state that the framework predicts fracture behavior, but this is in accordance with the aforementioned definition commonly used in the composites community of fracture as ultimate strength. The failure criteria within the model are stress-based and phenomenological, similar to many other failure criteria [17].

Reifsnider and colleagues have also spent a significant amount of time developing a framework for durability strength predictions of composite materials [e.g. 18], which has culminated in the creation of a code named MRLife. The method specifically relies on the “critical element” concept: determining the local ply that causes global failure and then examining how the local failure function changes with loading history. The method is somewhat similar to Chamis’ in that it spans many lengthscales and makes phenomenological strength predictions, but it differs in the way it determines the location of failure and the modifications it incorporates for durability.

A specific example of the application of fracture mechanics to structural predictions is the work of Krueger et al. [19]. They have developed a methodology to predict the fatigue life of bonded composite skin/stringer configurations using fracture mechanics and fatigue properties. Material property inputs such as critical strain energy release rates and crack propagation rates are used in conjunction with a finite element model of the structural configuration. Comparisons with experimental data indicate that the methodology makes reasonable predictions. Although the model does not begin at the constituent level, the properties involved span several lengthscales from the lamina to the structural level.

It is clear that there are many models that predict composite behavior from constituent properties and also many methodologies that link various models to create a framework for analyses from constituent to structural levels. Indeed, AIM-C is another methodology that hopes to accomplish this same objective. However, there is less discussion in the literature of the considerations associated with making predictions that span several lengthscales. Lagace, Spearing, and McManus have proposed
a design methodology that is an alternative to the building block approach and specifically examines the role of lengthscale in design of composite structures [20, 21]. The BBA begins at the lamina, or coupon, level and generates design allowables that are used in models to predict behavior at virtually all higher length scales up to the structural level, with empirical modifications made at each step to account for the increasing complexity. A problem with this approach is that it does not incorporate an understanding of the physical mechanisms involved and therefore leaves little room to improve the overall methodology.

Lagace et al. have proposed a methodology that begins at the constituent level with the choice of fiber, matrix, architecture, and processing and then progresses from one length scale to the next using “mechanism-based” models in addition to experimental results. They acknowledge that mechanism-based models are based, at some level, on experimental observations and experience. However, the mechanistic models have the advantage over phenomenological models in that they incorporate the underlying mechanisms controlling the failure process. In composite materials, these damage mechanisms can be quite complex, typically involving several key length scales, and often interacting with one another. It is a challenge to incorporate these components into a single framework, but in the end, predictions of structural behavior will be much stronger with an understanding of the mechanisms involved, as opposed to purely empirical modifications.

A key component of Lagace and coworkers’ methodology is that a step between lengthscales should occur with a combination of mechanism-based modeling and experimental observation. This evaluation is often lacking in many modeling frameworks, as it is often simply easier to make predictions without verifying all the assumptions. It is the intention of this thesis to examine one of these steps between lengthscales in an area that has not been previously examined and to assess the feasibility of its implementation into a methodology that incorporates mechanistic modeling.
2.2 Approach

The need for fracture mechanics models that predict composite behavior from constituent properties was articulated in Chapter 1 and the objective was stated as filling this need and examining the issues associated with this link between length-scales. While it is beyond the scope of this work to study the application of fracture mechanics models to multiple length-scales, the thesis results will lead to a better understanding of considerations surrounding this issue.

The approach used in this thesis was to examine the objective from a variety of perspectives and analyze the fundamental assumptions and mechanisms that should act as the foundation for a model linking constituent and composite fracture properties. Figure 2-2 shows the five areas studied in this thesis and the method used to study those topics. The numbers indicate the order in which the topics are presented in the thesis. Together, they are contributions that act as tools for design and materials engineers to evaluate and implement delamination properties of composite materials and thereby accelerate materials insertion. The order of the chapters in the thesis is meant to provide an assessment of the decisions that are part of connecting the two lengthscales from the assumptions made in the process to the experimentally observed mechanisms to the models. The specific contributions of each area as they apply to the overall approach are as follows.

First, nanoindentation is performed on polymers within composites in addition to the associated unreinforced neat polymers as a means of comparing the hardness and modulus properties of the two materials. The micromechanical models that make predictions of composite behavior typically use neat polymer properties that have been measured from specimens that were not necessarily manufactured in the same environment as the polymer within a composite. These differences may lead to different properties. The nanoindentations will probe the inherent assumption of the micromechanical models that the neat and unconstrained in situ polymer properties are the same. The experiments will explore the limitations of this assumption by comparing mechanical properties measured in the bulk polymer and the polymer in
the composite in between fibers. Differences between the two responses may have an effect on predictions of composite delamination behavior from constituent properties.

Second, quasi-static and fatigue fracture experiments are performed on neat resin and composite specimens at a variety of temperatures. There are several objectives of these tests. It is essential to have an understanding of the mechanisms involved in the fracture of the neat resin and the composite in order to make predictions of fracture or fatigue properties. In addition, shifts in quasi-static properties as a function of temperature or applied loading rate may allow for predictions to be made of fatigue behavior. Finally, quantitative measurements are needed as a basis for input and validation of predictive models.

Third, a model is created to predict composite delamination initiation behavior from constituent properties. This is an important component of determining whether these predictions will fit within the AIM-C analysis framework and are capable of estimating the effect of constituent properties on behavior that occurs at higher lengthscales.

Finally, a model is created that separates the effects of resin crack tip propagation and fiber bridging in fatigue delamination propagation. Fiber bridging is commonly encountered in Mode I quasi-static and fatigue delamination experiments and the tests performed for this research were no exception. However, the phenomenon is
observed to a far less extent in delaminating structures. Thus, the model calculates the strain energy release rate in the constrained resin that should be representative of fatigue propagation rates in an actual structure. The fatigue propagation data would be meaningless without the bridging model and hence, the model is an important data interpretation tool. In addition, it is a further example of the need to observe constituent behavior within the composite in order to make predictions of composite behavior.

The commonality amongst these areas is that they examine the issues associated with predicting composite delamination behavior using constituent properties. Although the various areas combine important contributions, an equally important component of the thesis will be an assessment of the use of constituent to composite delamination models. The final step of the approach is to tie the experimental and analytical work together to make such an assessment and in particular recommend methodologies that may be used in an academic or industrial setting to evaluate delamination behavior in composites. These issues are addressed in the Chapter 7.
Chapter 3

Nanoindentation of neat and \textit{in situ} polymers

Chapter 2 listed some of the numerous micromechanical models that use discrete fiber and matrix properties to predict composite behavior. Material property inputs for these models typically come from tests performed on the neat polymer (i.e. unreinforced cured resin or thermoplastic) or individual fibers. Implicit in the use of these measured properties is the assumption that the materials behave the same in the composite as they do individually. While it is unlikely that fiber properties would change, there is a distinct possibility that polymer properties could chemically change as a result of the consolidation and/or cure processes. For example, differences in curing procedures between the neat polymer and composite or infiltration of the sizing on the fibers into the polymer during composite processing could alter the unconstrained \textit{in situ} cured polymer behavior.

This chapter describes an effort to determine experimentally, through the use of nanoindentation, if there is a difference between neat and unconstrained \textit{in situ} polymer hardness and modulus properties for three polymers used in polymer matrix composites. While it is not clear that the indentation measurements accurately reflect bulk properties, comparisons using identical experimental techniques can be quite instructive.

The approach used in the study includes indentation of the matrix (and an ad-
hesive) in the composite and indentation of the same matrix (or adhesive) material in bulk form. The objective is to measure the unconstrained *in situ* load-penetration depth response of the matrix and compare it with identical measurements performed on the bulk matrix to determine whether the matrix material properties have changed as a result of the curing process. In this case, *in situ* refers to the *cured* matrix deformation in the composite. However, the goal is to find the *in situ* polymer modulus or hardness that is an *unconstrained* material property (not affected by the constraint of the fibers) of the *cured* resin. If the fiber constraint affects the matrix deformation response, then the comparison between bulk matrix properties and *in situ* properties is no longer “acceptable” because the measured behavior will be affected by the material properties and the mechanical constraint. Thus, numerical parameters are used to evaluate the shape of the loading portion of the load-penetration depth curves in the neat matrix material and the *in situ* matrix material. *In situ* tests that contain loading curves that deviate from the neat material behavior are considered to be affected by the fiber constraint and therefore unacceptable (modulus is measured during unloading). The quantitative comparisons of the neat and *in situ* curves determine whether any differences between the two responses are truly due to changes in material behavior. A finite element model is also created to determine the amount of *in situ* matrix area needed to perform a test that is unaffected by the constraint of the fibers.

### 3.1 Background

Indentation techniques have been used for decades to measure hardness properties of materials. More recently, nanoindentation has been used to measure properties in materials at smaller indentation depths. This is particularly useful for material systems where the penetration depth is important, such as thin films, or the indentation area is limited, such as composite materials.

Indentation testing was first applied to composite materials as a means to measure fiber-matrix interfacial properties [22–25]. Tests were performed using polymer,
ceramic, and metal matrix composites and properties were usually calculated using shear-lag and finite element models. Matrix properties have also been measured in ceramic [26] and metal [27] matrix composites. The elastic moduli and hardness of the matrices in the ceramic matrix composite tests were significantly less than the same properties in bulk ceramics of the same composition [26].

The majority of indentation testing has been performed on metallic and ceramic materials, but there has been an increasing amount of indentation experiments conducted using polymers [e.g. 28]. A thorough review of nanoindentation applied to polymers can be found in [29]. Elastic modulus and hardness properties have been obtained without much difficulty, but there have been problems in measurements using some polymers with a dependence of properties on depth, which may be due to surface properties. In addition, there have been few studies examining polymer viscoelastic response to a dynamically applied indentation load.

It is clear from the literature that nanoindentation techniques are well suited to composites and to polymers. One study in a ceramic matrix composite even indicated that the \textit{in situ} matrix properties are different than bulk values [26]. There have been no direct comparisons, however, of indentation on neat and \textit{in situ} polymer matrices in composites.

\section{3.2 Methods}

Nanoindentation tests involve an indenter (often diamond-tipped Berkovich, or pyramid, shaped) contacting a material surface and penetrating to a specified load or depth and then unloading. Load is measured as a function of penetration depth. Figure 3-1 shows a typical load-penetration depth curve. In this case, penetration depth is the displacement into the surface of the specimen. Calculation methods to determine hardness and modulus are typically based on the methods of Oliver and Pharr [30]. Hardness, $H$, is calculated using the load, $P$, and the projected contact area, $A$, at that load:

\begin{equation}
H = \frac{P}{A}.
\end{equation}
The projected contact area is dependent on the geometry of the indenter and is calculated from the depth of the indent, $h$. The slope of the initial portion of the unloading curve, $S = \frac{dP}{dh}$ (indicated in Figure 3-1), and the projected contact area are used to calculate the reduced modulus:

$$E_r = \frac{\sqrt{\pi S}}{2\beta \sqrt{A}}.$$  \hspace{1cm} (3.2)

$\beta$ is a constant that depends on the geometry of the indenter ($\beta=1.034$ for a Berkovich indenter [31]). The reduced modulus accounts for the fact that the measured displacement includes contributions from both the specimen and the indenter. The elastic modulus for the test material, $E$, is then calculated using the Poisson’s ratio of the test material, $\nu$, the modulus of the indenter, $E_i$, the Poisson’s ratio of the indenter, $\nu_i$, and the reduced modulus:

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}.$$ \hspace{1cm} (3.3)

The resulting equation for modulus is:

$$E = (1 - \nu^2) \left[ \frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i} \right]^{-1}.$$ \hspace{1cm} (3.4)
where the Poisson’s ratio of the test material must come from tests on the bulk material or an estimation. For a diamond-tipped indenter, $E_i=1140$ GPa and $\nu_i=0.07$.

The aforementioned procedure measures hardness and modulus at the maximum penetration of a single load-unload indent cycle. However, it is also possible to measure these quantities continuously during loading by superimposing a small oscillation on the loading signal and analyzing the system response. The result is that one obtains hardness and modulus as a function of penetration depth. This is known as continuous stiffness monitoring (CSM) and the method used to determine modulus by this method is given by the manufacturer of the nanoindenter [31]. Calculations are made using the standard analysis of a simple harmonic oscillator subject to a forced oscillation. The ordinary differential equation (ODE) resulting from the force summation on a mass is:

$$m\ddot{z} + D\dot{z} + Kz = F(t), \quad (3.5)$$

where $z$ is the vertical displacement of the indenter and $m$ is the mass of the system (including the indenter and the indenter column). $K$ is an equivalent stiffness that includes the contact stiffness, $S$, load-frame stiffness, $K_f$, and stiffness of the support springs for the indenter column, $K_s$:

$$K = (S^{-1} + K_f^{-1})^{-1} + K_s. \quad (3.6)$$

$D$ is damping that occurs in the indentation head, $D_i$, and the sample, $D_s$:

$$D = D_i + D_s. \quad (3.7)$$

$F(t)$ is a forcing function that has the form:

$$F(t) = F_0e^{i\omega t}, \quad (3.8)$$

where $t$ is time, $F_0$ is the magnitude of the function, and $\omega$ is the frequency. The resulting displacement is assumed to have the same form, but with a lag, characterized
by the phase angle, $\phi$:

$$z(t) = z_0 e^{i(\omega t - \phi)}.$$  \hspace{1cm} (3.9)

The displacement solution is substituted into the ODE, Equation 3.5, and when the magnitudes are equated the results are:

$$\left| \frac{F_0}{z_0} \right| = \sqrt{(K - m\omega^2)^2 + (\omega D)^2},$$  \hspace{1cm} (3.10)

$$\tan \phi = \frac{\omega D}{K - m\omega^2}.$$  \hspace{1cm} (3.11)

These equations are solved simultaneously for $K$ and $D$ and then the contact and dampening stiffness may be determined from Equations 3.6 and 3.7:

$$S = \left[ \frac{1}{\frac{K}{z_0} \cos \phi - (K_s - m\omega^2)} - \frac{1}{K_f} \right]^{-1},$$  \hspace{1cm} (3.12)

$$D_s \omega = \frac{F_0}{z_0} \sin \phi - D_i \omega.$$  \hspace{1cm} (3.13)

The parameters $K_f$, $m$, $K_s$, and $D_i$ are measured by the manufacturer and do not change. During an experiment, the excitation frequency, $\omega$ is set and the displacement amplitude, $z_0$, phase angle, $\phi$, and excitation amplitude, $F_0$ are all measured. Thus, $S$ and $D_s$ can easily be calculated using Equations 3.12 and 3.13. $S$ is then used to calculate modulus in the same equations used in the basic load-unload method, Equations 3.2 and 3.3.

Two nanoindenters were used in this study because of their different capabilities. The first, an MTS Systems Nano Indenter® XP, has a maximum load capability of 500 mN and includes a continuous stiffness monitoring system. The second, a Hysitron Triboindenter®, has a maximum load capability of 12 mN, but does not include a continuous stiffness monitoring system. The MTS system allowed for larger penetration depths and continuous stiffness measurements while the Hysitron system gave high resolution at smaller penetration depths. Load and displacement resolutions for the MTS system are 50 nN and 0.01 nm, respectively, whereas the load and displacement...
resolutions for the Hysitron machine are 1 nN and 0.0002 nm, respectively.

Three material systems were analyzed as part of this experimental study. Both the neat polymers and the composites (or constrained adhesive) were tested. The materials were selected to represent a range of polymers used in composite materials. The composite systems and constrained adhesive tested were IM7/977-3, FM 300 adhesive joining IM7/977-3 adherends, and AS4/APC-2. 977-3 is an epoxy resin, FM 300 is an epoxy film adhesive, and APC-2 is a thermoplastic (PEEK). Neat polymer plaques approximately 3 mm thick of 977-3, FM 300, and APC-2 were provided by the manufacturer, Cytec Engineered Materials. Specimens measuring approximately 20 mm long by 10 mm wide were cut for testing using a diamond-bladed saw. The IM7/977-3 and AS4/APC-2 composite specimens were cut from 28-ply unidirectional double cantilever beam (DCB) specimens manufactured for a separate set of fracture tests described in Chapter 4. The adhesive joint specimen contained two layers of 250 μm thick FM 300 film adhesive between 14-ply IM7/977-3 composite adherends. All material systems were manufactured in accordance with manufacturer recommended guidelines. The IM7/977-3 specimens (including the FM 300 specimen as a co-cure) were fabricated using an autoclave cure at 0.59 MPa pressure and 179 °C. The AS4/APC-2 specimens were fabricated in a hot press under 1.72 MPa pressure at 391 °C in atmospheric conditions.

The DCB specimens were 25 mm wide and 150 mm long and contained 63 mm long and 13 μm thick Teflon® (in the IM7/977-3 specimens, including the adhesive joint specimen) or Kapton® (in the AS4/APC-2 specimens) inserts. IM7/977-3 specimens were approximately 7 mm thick while AS4/APC-2 specimens were approximately 3 mm thick. The large difference in thickness is due to the difference in fiber areal weight for the two materials: 290 g/m² for IM7/977-3 and 144 g/m² for AS4/APC-2. Specimens measuring 25 mm long by 10 mm deep were cut using a diamond-bladed saw from the DCB specimens for indentation testing.

Figure 3-2 is a schematic of the specimens created from each of the materials. The specimen containing FM 300 was cut so that the indentation surface was perpendicular to the fiber direction. The adhesive layer in this specimen was in a region
Two types of IM7/977-3 and AS4/APC-2 specimens were created. The first had the indentation direction parallel to the fiber direction and the second had the indentation direction perpendicular to the fiber direction and in the lamina plane. The latter specimens were also cut so that the region ahead of the inserts was included. These areas often contain polymer rich regions that are ideal for indentation testing and are of interest to the delamination model presented in Chapter 5. All the composite specimens were molded inside resin cylinders measuring approximately 30 mm in diameter and 15 mm tall with the indentation surface in the same plane as the top flat surface of the resin cylinder. Neat polymer specimens were mounted on aluminum cylinders of the same dimensions as the resin cylinders. These blocks facilitated polishing of the indentation surfaces, which was performed up to a 0.5 μm particle size polishing solution finish.

Indentation tests conducted on the neat polymers were straightforward in that the indentations could be performed anywhere on the surface away from a free edge (approximately ten times the maximum penetration depth). This allowed for a series of tests at different locations and using different loading conditions to be automatically entered into the software controlling the nanoindenters, which performs all the tests automatically. Neat polymer tests were conducted in two different manners on the MTS and Hysitron machines. Seven “basic”, or monotonic, tests were conducted on
each polymer using the MTS machine; five indents were conducted per test. Each test consisted of five load-unload cycles in the same location in which the maximum load in the first test was 31.25 mN and was doubled until the maximum load of 500 mN was reached at the fifth test. The time to load for each test was fifteen seconds and the maximum load was held for two seconds. The resulting data includes modulus and hardness as a function of penetration depth at five different depths where each data point is an average of seven measurements.

Seven continuous stiffness monitoring (CSM) tests were also performed in seven separate locations on each polymer. The inputs for these tests included a maximum penetration depth, set to correspond to the penetration depth of a 500 mN load (8-12 \( \mu \)m depending on the material), a frequency target of 45 Hz, a harmonic displacement target of 2 nm, and a strain rate target of 0.050 s\(^{-1}\). The CSM tests produce seven distinct plots of hardness and modulus as a continuous function of penetration depth.

Tests using the Hysitron indenter were performed in a somewhat different fashion. Six separate indents at increasing maximum loads were conducted five times for a total of thirty tests. The first indent reached a maximum load of 2 mN and in each indent thereafter the maximum load increased by 2 mN up to a final maximum load of 12 mN. The loading and unloading rate was 1.2 mN/s and the maximum load was held for five seconds. The data from these tests include modulus and hardness as a function of penetration depth at six different depths where each data point is an average of five measurements.

Testing in the composite specimens was more complicated than in the neat polymers because the exact location for the test must be chosen. A pocket of the polymer must be chosen for a test such that the surrounding fibers do not influence the deformation behavior of the polymer. However, the same loading rates, CSM inputs, and load and depth ranges were used in the composite tests, but it was not possible to perform the same number of tests because the available testing area was much more limited. One CSM test and five basic tests (one at each load level) were performed on each polymer in each composite using the MTS testing machine. The CSM and basic tests were performed in different polymer pockets. Anywhere from four to eight basic
tests were conducted at three loads (2, 7, 12 mN) on each polymer using the Hysitron machine. Each test was performed in a different polymer pocket. The objective was to obtain four test results that were not clearly affected by the fiber constraint (indicated by modulus results under 10 GPa). In some cases, this was not possible (see discussion in the following section).

3.3 Results

Figure 3-3 shows results from tests performed on neat and in situ 977-3 using the MTS nanoindenter. A Poisson’s ratio of 0.37 was used in all modulus calculations, which was obtained from an AIM-C database that included properties from the manufacturer. Results in the figure for the neat resin “basic” tests (i.e. monotonic or single load-unload) are the average of seven measurements. Error bars are plotted for the neat data indicating standard deviation, but the error is on the order of the size of the marker in all cases and thus the error bars cannot be seen in the plot. Seven CSM tests were also conducted on the neat resin, but results from only one of the tests are shown in the plot for clarity; all of the CSM results were virtually indistinguishable from one another (the “noise” from each test created variations of approximately ±5% and the variation amongst all tests was within this range). The in situ resin tests were conducted on the specimen with fibers running in the same direction as the indentation test. The basic and CSM results are from individual tests. As mentioned previously, these tests were conducted on pockets of resin where it was believed that the mechanical constraint of the surrounding fibers would not affect the test results (this issue is explored more in the next section). This is desirable since the objective is to compare the deformation of the resin itself, which can only be compared in the unconstrained state. Typical indentations from the MTS indenter and the associated resin pockets are shown in Figure 3-4.

The CSM results show a steep increase in modulus and hardness at low penetration depths until a plateau is reached. The in situ plots, particularly modulus, continue to increase at higher depths, indicating that the presence of the fibers is having a strong
Figure 3-3: Neat and *in situ* 977-3 modulus and hardness measured using the MTS nanoindenter. *In situ* measurements are parallel to the fiber direction. Error bars are plotted for the neat data indicating standard deviation, but the error is on the order of the size of the marker in all cases and thus the error bars cannot be seen in the plot.
Figure 3-4: Indentations parallel to the fiber direction in two separate ((a) and (b)) IM7/977-3 resin pockets.
influence on resin deformation. This is also seen in the basic \textit{in situ} data, which is fairly constant at depths below 5 \(\mu\text{m}\), but sharply increases thereafter. Basic and CSM neat resin test data are virtually identical for modulus and fairly independent of penetration depth (above 2 \(\mu\text{m}\)) for both modulus and hardness. There is a slight offset between the basic and CSM hardness data. Differences between neat and \textit{in situ} data are on the order of 10-20\% for maximum indentation depths below 5 \(\mu\text{m}\).

Tests performed perpendicular to the fiber direction using the MTS nanoindenter on resin pockets in IM7/977-3 were significantly affected by fibers beneath the surface of the resin pockets. Figure 3-5 shows results from a CSM test conducted in a resin pocket directly ahead of the Teflon\textsuperscript{®} insert. The effects of the fibers on resin deformation are quite clear. Other tests conducted in resin pockets away from the insert produced similar results.

All \textit{in situ} FM 300 tests were performed perpendicular to the fiber direction. Tests were not performed in the orthogonal direction because the isotropic nature of the adhesive material was not expected to produce different results. Figure 3-6 shows typical indents made using the MTS indenter and results from the MTS tests are shown in Figure 3-7. No Poisson’s ratio value could be found in the literature, so an estimate of 0.3 was used in all modulus calculations. Once again, error bars are plotted for the neat data indicating standard deviation, but the error is on the order of the size of the marker in many cases and thus the error bars cannot be seen in the plot for some data points. Although there is some variation in the results with penetration depth, it is not a strong effect. This has particular significance for the \textit{in situ} results because it appears to indicate that the constraining layers most likely do not have a large effect on the resin deformation. The modulus and hardness results from the basic tests are nearly the same for the neat and \textit{in situ} resins, and the basic test data is quite close to the neat CSM data. There is an offset of approximately 10-20\% between the neat and \textit{in situ} CSM data.

Meaningful \textit{in situ} data was virtually impossible to obtain in the AS4/APC-2 specimens due to a lack of polymer pockets of adequate size. Data measured using the MTS nanoindenter with \textit{in situ} tests performed directly ahead of the Kapton\textsuperscript{®} insert
Figure 3-5: Neat and in situ 977-3 modulus and hardness measured using the MTS nanoindenter. In situ measurements are perpendicular to the fiber direction immediately ahead of the Teflon® insert.
(perpendicular to the fiber direction) are plotted in Figure 3-8. This indent is shown in Figure 3-9a (the large indent). Neat basic and CSM results are fairly consistent and independent of penetration depth, but in situ data, particularly modulus, increases sharply as the depth increases. No indents were performed on the specimen with fibers in the same direction as the indentation because there were no polymer pockets large enough to accommodate the size of an indent. One of the larger (and rare) polymer pockets is shown in Figure 3-9b. As with FM 300, an estimate of 0.3 was used for Poisson’s ratio in all modulus calculations.

The experiments performed on the MTS indenter illuminated the need for testing at smaller penetration depths. These were deemed necessary for several reasons. First, all CSM tests exhibited a sharp increase in hardness and modulus at depths under 2 μm. It was not clear whether this behavior was the result of a thin surface layer with different characteristics than the bulk material (perhaps created during polishing) or if it was an anomaly of the CSM test. Second, the in situ test results generally exhibited increases in modulus and hardness as penetration depths increased. This was almost certainly a result of resin deformation being affected by the fibers at higher depths. The Hysitron nanoindenter was used to explore these

Figure 3-6: Indentations perpendicular to the fiber direction in a constrained FM 300 layer.
Figure 3-7: Neat and in situ FM 300 modulus and hardness measured using the MTS nanoindenter. In situ measurements are perpendicular to the fiber direction in the constrained layer. Error bars are plotted for the neat data indicating standard deviation, but the error is on the order of the size of the marker in many cases and thus the error bars cannot be seen in the plot for some data points.
Figure 3-8: Neat and in situ APC-2 modulus and hardness measured using the MTS nanoindenter. In situ measurements are perpendicular to the fiber direction immediately ahead of the Kapton® insert. Error bars are plotted for the neat data indicating standard deviation, but the error is on the order of the size of the marker in all cases and thus the error bars cannot be seen in the plot.
Figure 3-9: (a) Indentations perpendicular to the fiber direction and (b) one of the larger polymer pockets for indentations parallel to the fiber direction in AS4/APC-2.
issues by making indents with depths of less than 2 μm.

Figure 3-10 shows neat and in situ results for 977-3 obtained using the Hysitron indenter, where the in situ data is from measurements parallel to the fiber direction. The neat resin data points are an average of five tests while the in situ data points are an average of four tests. Each of the in situ tests was performed in a different resin pocket. All results show little variation with penetration depth, but there is more statistical variation in the in situ data. Neat and in situ hardness data are virtually identical, but the in situ modulus is 20-30% higher than the neat modulus.

Results from similar tests on FM 300 are shown in Figure 3-11. Once again, the data are fairly independent of penetration depth and there is more variation in the in situ results. The in situ values are 20% higher than neat values for hardness and
Figure 3-11: Neat and *in situ* FM 300 modulus and hardness measured using the Hysitron nanoindenter. *In situ* measurements are perpendicular to the fiber direction in the constrained layer. Error bars indicate standard deviation.

30-40% higher for modulus.

Indentations were performed on neat and *in situ* APC-2 using the Hysitron indenter as well. The smaller indent size allowed for several tests to be performed on polymer pockets in the AS4/APC-2 specimen with fibers aligned in the indentation direction. However, modulus values in the tests performed at the smallest load (2 mN) were an average of 38.5 GPa with a standard deviation of 10 GPa, clearly indicating that the fiber constraint was affecting polymer deformation in all cases. Thus, only neat polymer test data were considered acceptable, which are shown in Figure 3-12.

Data from tests performed using both nanoindenters is plotted in Figures 3-13, 3-14, and 3-15 for 977-3, FM 300, and APC-2, respectively. Several conclusions may
Figure 3-12: Neat APC-2 modulus and hardness measured using the Hysitron nanoindenteter. Error bars indicate standard deviation.
be drawn from the data. First, the unconstrained polymer modulus and hardness are independent of penetration depth for both the basic method at all depths tested and the CSM method at depths above 2 μm. Second, the sharp increase in modulus and hardness values in the initial portion of the CSM plots appears to be an artifact of the test method, potentially caused by tip blunting effects and decreased signal-to-noise ratios at low load and displacement levels [29]. This conclusion is supported by the observation that monotonic indentation tests performed over the same depth ranges produced results similar to those produced by CSM at higher depth ranges, in which the CSM data were constant. Third, the neat polymer modulus is the same for monotonic indentation and CSM tests performed using both indenters. Fourth, hardness values measured using the Hysitron indenter are higher than those measured using the MTS indenter. Fifth, the in situ hardness appears to be less affected by fiber constraints than the modulus. Sixth, the statistical variation of the in situ test data is higher than that of the neat polymer test data. Seventh, the modulus of 977-3 is 10-30% higher in the in situ tests, whereas the hardness is 0-20% higher in the in situ tests. Eighth, the neat and in situ modulus and hardness measurements of FM 300 are nearly the same in the MTS data whereas the in situ data is 20-40% higher for Hysitron measurements. Ninth, the lack of large polymer pockets in AS4/APC-2 makes meaningful in situ testing virtually impossible. And finally, moduli measured using nanoindentation for the neat polymers were similar to the bulk properties, with the exception of the neat thermoplastic specimen, which showed higher values than typically reported. Measured values from the Hysitron experiments were 4.0 GPa for 977-3, 3.0 GPa for FM 300, and 5.6 for APC-2. Reported tensile moduli for the materials are 3.8 GPa for 977-3 [32], 3.0 GPa for FM 300 [33], and 3.6-4.1 GPa for APC-2 [32, 34].

In general, the in situ properties are higher than neat polymer properties. The fundamental question is whether this increase is due to a change in material properties from the curing process or whether the fibers play a mechanical role in modifying resin deformation. The load-penetration depth plots of each test are the best source of information as to whether the deformation is affected by a constraint. Unconstrained
Figure 3-13: Neat and in situ 977-3 modulus and hardness measured using both nanoindenters. In situ measurements are parallel to the fiber direction. Error bars indicate standard deviation.
Figure 3-14: Neat and *in situ* FM 300 modulus and hardness measured using both nanoindenters. *In situ* measurements are perpendicular to the fiber direction in the constrained layer. Error bars indicate standard deviation.
Figure 3-15: Neat and \textit{in situ} APC-2 modulus and hardness measured using both nanoindenters. \textit{In situ} measurements are perpendicular to the fiber direction. Error bars indicate standard deviation.
deformation should theoretically follow Kick’s Law [35],

\[ P = C h^2 \]  \hspace{1cm} (3.14)

where \( C \) is the loading curvature. However, experimentally the exponent in this equation deviates slightly from a value of two due to effects such as blunting of the indentation tip. A more general expression of this relationship is:

\[ P = C h^m. \]  \hspace{1cm} (3.15)

When the load-penetration depth data are plotted using log scales, the data are linear and the slope of the line is \( m \) and the \( y \)-intercept is \( C \). All 977-3 and FM 300 experimental data were analyzed in this fashion and values for \( m \) and \( C \) were determined for each test using a least squares fit of the logarithmic data. \( (\text{In situ} \) APC-2 data sets were not analyzed because the modulus values made it clear that these tests were affected by the fiber constraint.) Since the neat polymer tests are unconstrained, deviations in the \( \text{in situ} \) fit parameters from the neat polymer values would indicate that the \( \text{in situ} \) tests were mechanically affected by the fiber constraint.

An example of the fitting process is shown in Figure 3-16, which depicts a neat resin 977-3 load-penetration depth plot and two \( \text{in situ} \) 977-3 plots. Figure 3-16a clearly shows that \( \text{in situ} \) test \#2 was affected by the constraint of the fibers because the deformation curve is quite different than the neat resin curve; the high modulus from this test reflects the fiber constraint. It is not clear, however, merely from the deformation curve whether \( \text{in situ} \) test \#1 was affected by the fiber constraint. The logarithmic plots in Figure 3-16b and the associated fit parameters prove that the \( \text{in situ} \) test \#2 was affected by the fiber constraint because its fit parameters are quite different than the neat resin parameters. The fit parameters from \( \text{in situ} \) test \#1 are nearly identical to the neat resin parameters, indicating that this test was not affected by the fiber constraint. It is important to note that the \( m \) and \( C \) parameters are determined from the loading portion of the curve, whereas the modulus is determined from the unloading curve. Thus, it is possible for an \( \text{in situ} \)
test to have similar loading but different unloading when compared to a neat resin test.

The fit values for the neat and *in situ* 977-3 MTS and Hysitron tests are shown in Figure 3-17 and Figure 3-18 and values for the neat and *in situ* FM 300 MTS and Hysitron tests are shown in Figure 3-19 and 3-20. The values plotted are the average of all tests at that load and resulting penetration depth. The initial portion of the loading curve is not linear on a logarithmic plot, so the \( m \) and \( C \) parameters were determined from a least squares fit of the highest 60% of the loading curve where the behavior was linear in every test. The goodness of fit \( (R^2) \) in all cases was above 0.998 (1 being perfect).

The values for \( m \) and \( C \) are nearly identical for the first three penetration depth levels in Figure 3-17 for the 977-3 MTS tests, indicating that the *in situ* and neat comparison is acceptable. This is not the case for the highest two penetration depth levels where the fiber constraint most likely affected the resin deformation response. The data for the 977-3 Hysitron tests in Figure 3-18 indicates that it is acceptable to compare all measurements because the curvature and exponent values are virtually the same. It should be noted that higher scatter in the *in situ* \( C \) values is expected because this parameter is extremely sensitive to slight variations in the fitted data and *in situ* data had more scatter for all measured values. However, all data were fit over the same load range and thus, the comparison between the neat and *in situ* data is reasonable.

Figure 3-19 depicting the \( m \) and \( C \) values from the FM 300 MTS tests shows a consistent offset between the neat and *in situ* tests, demonstrating that a comparison between the two sets of tests is unacceptable. Conversely, the similarity of the parameters in Figure 3-20 from the FM 300 Hysitron tests demonstrates that a comparison is acceptable.

The ratios of the *in situ* and neat \( m, C, E, \) and \( H \) values are listed in Table 3.1 for 977-3 and Table 3.2 for FM 300. All ratios were calculated using average values. Loads ranging from 2-12 mN were performed using the Hysitron machine and loads ranging from 31-500 mN were performed using the MTS machine. A ratio for \( m \)
Figure 3-16: (a) Load-penetration depth plots for three 977-3 tests performed using the Hysitron nanoindenter and (b) the same plots on logarithmic scales.
Figure 3-17: Neat and *in situ* 977-3 load-penetration depth fit parameters from MTS tests.
Figure 3-18: Neat and in situ 977-3 load-penetration depth fit parameters from Hysitron tests. Error bars indicate standard deviation.
Figure 3-19: Neat and in situ FM 300 load-penetration depth fit parameters from MTS tests.
Figure 3-20: Neat and in situ FM 300 load-penetration depth fit parameters from Hysitron tests. Error bars indicate standard deviation.
Table 3.1: Ratio of 977-3 in situ to neat properties. Subscripts: $is=in situ$ and $n=neat$.

<table>
<thead>
<tr>
<th>Load (mN)</th>
<th>$m_{is}/m_n$</th>
<th>$C_{is}/C_n$</th>
<th>$E_{is}/E_n$</th>
<th>$H_{is}/H_n$</th>
<th>Acceptable Test?</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>1.02</td>
<td>1.01</td>
<td>1.21</td>
<td>0.93</td>
<td>Y</td>
</tr>
<tr>
<td>7</td>
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<td>0.74</td>
<td>1.23</td>
<td>0.96</td>
<td>Y</td>
</tr>
<tr>
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<td>1.03</td>
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<td>0.97</td>
<td>Y</td>
</tr>
<tr>
<td>31</td>
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<td>0.89</td>
<td>1.10</td>
<td>1.16</td>
<td>Y</td>
</tr>
<tr>
<td>62</td>
<td>1.01</td>
<td>0.94</td>
<td>1.16</td>
<td>1.19</td>
<td>Y</td>
</tr>
<tr>
<td>125</td>
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<td>0.82</td>
<td>1.22</td>
<td>1.18</td>
<td>Y</td>
</tr>
<tr>
<td>250</td>
<td>1.12</td>
<td>0.13</td>
<td>1.55</td>
<td>1.23</td>
<td>N</td>
</tr>
<tr>
<td>500</td>
<td>1.15</td>
<td>0.08</td>
<td>1.72</td>
<td>1.29</td>
<td>N</td>
</tr>
</tbody>
</table>

and $C$ that are near one indicates that a comparison between the in situ and neat tests is “acceptable” and that the measured in situ modulus and hardness should be considered unaffected by the fiber constraint. A ratio that deviates from one indicates that the in situ test was affected by the fiber constraint and is not relevant to this study. Thus, an “acceptable” test is an unconstrained measurement. In general, in situ $m$ values that were no more than 10% different than the neat values (within the experimental statistical deviation of the neat polymer experiments) were considered acceptable. The aforementioned scatter in the in situ $C$ values made comparisons of these values more difficult. However, the same 10% rule was generally applied to these values as well. The values of both $m$ and $C$ ratios were used to determine whether the in situ tests were acceptable and the final column in the tables lists the verdict for each test. All tests performed using the Hysitron were acceptable tests whereas only some MTS tests were acceptable for 977-3 and none were acceptable for FM 300.

Once the acceptability of each data series is determined, one may make definitive conclusions regarding the changes of the 977-3 and FM 300 modulus and hardness properties within the composite using the acceptable data. The unconstrained in situ 977-3 modulus is 10-30% higher than the neat modulus and the hardness ranges from 7% less to 20% more than the neat hardness. Furthermore, the unconstrained in situ FM 300 modulus is 30-40% higher than the neat modulus and the hardness is 20-26% higher than the neat hardness. It is clear that unconstrained in situ cured properties
Table 3.2: Ratio of FM 300 in situ to neat properties. Subscripts: is=in situ and n=neat.

<table>
<thead>
<tr>
<th>Load (mN)</th>
<th>m_{is}/m_n</th>
<th>C_{is}/C_n</th>
<th>E_{is}/E_n</th>
<th>H_{is}/H_n</th>
<th>Acceptable Test?</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
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<td>3.32</td>
<td>1.41</td>
<td>1.26</td>
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</tr>
<tr>
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<td>1.28</td>
<td>1.19</td>
<td>Y</td>
</tr>
<tr>
<td>12</td>
<td>1.02</td>
<td>1.01</td>
<td>1.31</td>
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</tr>
<tr>
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<td>0.08</td>
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</tr>
<tr>
<td>62</td>
<td>1.13</td>
<td>0.09</td>
<td>0.99</td>
<td>0.93</td>
<td>N</td>
</tr>
<tr>
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<td>0.11</td>
<td>1.06</td>
<td>1.01</td>
<td>N</td>
</tr>
<tr>
<td>250</td>
<td>1.09</td>
<td>0.18</td>
<td>1.11</td>
<td>1.08</td>
<td>N</td>
</tr>
<tr>
<td>500</td>
<td>1.06</td>
<td>0.29</td>
<td>1.15</td>
<td>1.12</td>
<td>N</td>
</tr>
</tbody>
</table>

are not the same as the neat properties.

### 3.4 Finite Element Simulations

A remaining question for any in situ tests is the size of the polymer pocket that is required in order to measure polymer deformation that is unaffected by the fiber constraints. A characteristic dimension of a polymer pocket is the distance across the pocket. Since the pockets are rarely circular, this dimension is open to interpretation, but the dimensions that characterize these pockets are within a reasonable range. Many of the large resin pockets tested in the IM7/977-3 specimen, such as those in Figure 3-4, had regions without fibers that ranged from 70 to over 200 μm across, whereas a rare large polymer pocket in the AS4/APC-2 specimen, such as the one in Figure 3-9b, was 35 μm across. The FM 300 layer was 120 μm thick.

A finite element model (FEM) was created to determine the smallest pocket size that could be tested without the constraint of the fibers affecting polymer deformation. The FEM was based on a model used by Dao et al. [35]. A schematic of the model is shown in Figure 3-21a and the indented mesh at maximum displacement is shown in Figure 3-21b. The mesh in Figure 3-21a is coarser than the mesh used in these studies in order to depict clearly the density in different regions. The model was created and run using the ABAQUS® finite element package (a sample input file is included in Appendix A.1) and used four-noded, bilinear axisymmetric quadrilat-
eral elements that incorporated large deformation theory. A rigid surface was used to simulate the indenter. In the case of this axisymmetric model, the rigid surface is conical, which is not the same as the Berkovich indenter used in the experiments. However, previous work has shown that a conical half apex angle of 70.3° (angle \( \theta \) in Figure 3-21a) produces the same contact depth and area as a Berkovich indenter [35]. A fine and uniform mesh is used in the contact region (50 elements in distances \( x_1 \) and \( y_1 \)) whereas a gradually coarser mesh is used in areas further from the contact region (25 elements in distances \( x_2 - x_1 \) and \( y_2 - y_1 \)). The mesh density in the contact region determines the resolution of the load-displacement curve whereas the density outside this region does not have a significant effect on the model’s results. At maximum displacement, the mesh in the contact region contained at least ten elements in contact with the rigid surface.

The material properties used in this study corresponded to those of 977-3. The polymer was modeled as elastic-plastic using properties that were based on compression tests described in Section 5.2. In all simulations, the bottom of the model was constrained in the \( y \)-direction, whereas the axis of symmetry (the left side of the model) was constrained in the \( x \)-direction, but was unconstrained in the \( y \)-direction. The boundary condition on the outer edge of the model (the right side) was assigned one of two definitions depending on whether the model was simulating an unconstrained or constrained boundary. An unconstrained boundary involved no constraints in either direction and simulated a neat resin test whereas a constrained boundary prevented movement in the \( x \) and \( y \) directions and simulated an \textit{in situ} test where the fibers would inhibit displacement at the boundary. This assumes the fibers do not deform, which is based on the longitudinal fiber modulus being three orders of magnitude higher than that of the resin. Load-penetration depth data was obtained from each simulation and curve parameters \( m \) and \( C \), in addition to modulus, were calculated using this data and the same methods implemented in the experiments. Each simulation was performed in load-control up to a maximum specified load, analogous to the method used in the experiments. Simulations performed in displacement control gave identical load-penetration depth curves. Modulus val-
Figure 3-21: (a) Finite element model schematic and (b) deformed mesh at maximum penetration in contact region.
ues and load-penetration depth curves were reasonably similar to experimental data, indicating that the material definition was adequate for this study.

A simulation was conducted using an unconstrained model and then constrained simulations were conducted in which the dimension $x_2$ (as depicted in Figure 3-21a) was decreased. In all simulations the dimensions $y_1$, ten times the maximum displacement, and $y_2$, three hundred times the maximum displacement, did not change. (The general term "maximum displacement" refers to the maximum displacement for a given load in an unconstrained simulation. Since the same load is applied in all simulations, the maximum displacement in the constrained simulations will actually decrease depending on the level of the constraint.) The dimension $x_2$ began as 300 times the maximum displacement in the unconstrained simulation, whereas the constrained model was simulated at values of 300 down to 2.5 times the maximum displacement. The dimension $x_1$ remained ten times the maximum displacement for all simulations in which $x_2$ was greater than ten times the maximum displacement. Figure 3-22 shows a load-penetration depth plot from an unconstrained simulation and a plot from the most constrained simulation.

The results of the simulations are shown in Figure 3-23, which plots $E$, $m$, and $C$ values from the constrained analyses normalized by the unconstrained values as a function of the model size, $x_2$, divided by the maximum displacement. Simulations performed at different maximum load levels produced identical curves. As $x_2$ decreases, the constrained modulus remains within 10% of the unconstrained modulus for $x_2$ values twenty-five times the maximum displacement and above. This is clearly seen in the steep increase of the constrained $C$ values below this model size. Thus, the important conclusion of this analysis is that the size of the polymer pocket must be at least fifty times the maximum displacement ($x_2$ in the model is only half the size of the polymer pocket) in order to have modulus measurements that are within 10% of unconstrained values and therefore relatively unaffected by the fibers. These specific results are applicable only to 977-3 and they are actually conservative given that the constraint overestimates the effects of the fibers (i.e. no polymer pocket is completely surrounded by a rigid wall of fibers), but the study certainly can give
guidance to tests on other polymer matrix composite systems. The controlling factor that will differentiate material systems will be the inelastic material response.

In light of the simulation results, the experimental results may be reexamined. For example, the smallest penetration depth in the in situ APC-2 tests was 0.5 μm. Fifty times this value is 25 μm, which is larger than almost all of the polymer pockets found in the AS4/APC-2 specimen and explains why no acceptable results could be obtained with this specimen. The smallest penetration depth in the in situ FM 300 MTS tests was 3 μm and the largest penetration depth in the Hysitron tests was 1.8 μm. Given that the FM 300 layer was 120 μm thick, it is now clear why MTS tests were unacceptable and the Hysitron tests were acceptable. The large pockets in the IM7/977-3 specimen were clearly essential to obtaining acceptable results for the material. Tests on other materials may need to rely on artificially created polymer pockets of adequate size in the composite in order to obtain acceptable experimental results.
Figure 3-23: Constrained $E$, $m$, and $C$ values from the finite element simulations normalized by unconstrained values as a function of model size normalized by maximum penetration depth.
3.5 Conclusions

Nanoindentation experiments were conducted on three different polymer matrix composite systems to determine whether polymer properties change as a result of the consolidation and processing procedure. Both neat and in situ tests showed that unconstrained polymer deformation does not vary significantly with penetration depth. However, the majority of the in situ experiments produced results that were measurably different than the neat polymer data. Quantitative measures derived from the load-displacement data were used to determine which tests had been influenced by the constraint of the fibers and were therefore unacceptable. The acceptable tests indicated that the unconstrained in situ modulus and hardness values were clearly different than the neat polymer values and differed by as much as 30%. Finite element simulations of the experiments showed that a polymer pocket should be at least fifty times larger than the maximum displacement in order to obtain acceptable unconstrained in situ measurements.

A certain amount of error in polymer deformation behavior may be acceptable in predictions of certain composite properties that are not sensitive to these inputs, such as longitudinal modulus. However, micromechanical models are increasingly being used to predict composite strength and these models may require higher fidelity of material properties. A 30% error in polymer modulus for these models may have significant consequences on strength predictions.
Chapter 4

Constituent and Composite
Quasi-static and Fatigue Fracture Experiments

Any models that predict composite quasi-static and/or fatigue behavior must be based on the appropriate damage mechanisms. The purpose of the work in this chapter is to examine the quasi-static and fatigue fracture mechanisms in a polymer matrix composite and its neat resin. The experimental results will serve three main purposes: the mechanisms will act as the foundation for models describing delamination behavior, the measurements will act as inputs and validation for models, and the shifts in neat resin and composite quasi-static fracture behavior as a function of loading rate and temperature may be useful in describing composite fatigue delamination behavior.

The focus for the composite experiments is on delamination, which primarily involves resin fracture between plies, and thus, constituent experiments only involve the neat resin. Some specific objectives for the tests include: measuring neat resin and composite Mode I quasi-static fracture properties as a function of temperature and loading rate, comparing neat resin and composite quasi-static fracture mechanisms, comparing shifts in quasi-static toughness with temperature and rate, determining the effect of temperature on resin Mode I fatigue crack propagation and composite Mode I fatigue delamination onset and propagation, and analyzing shifts in composite fatigue
behavior with temperature and comparing them with static or constituent behavior. The neat resin quasi-static fracture mechanisms and loading rate and temperature shifts can act as the foundation for explaining and potentially predicting composite quasi-static and fatigue behavior. These types of techniques are already used to predict composite elastic and strength fatigue behavior [36], but have not yet been applied to delamination properties.

4.1 Background

A significant amount of effort has been directed toward developing the current standard test method measuring Mode I interlaminar fracture toughness, which uses a unidirectional double cantilever beam (DCB) specimen. There are many references available in the literature that summarize the experiments that led to the key decisions made for the development of the standard [e.g. 37, 38], but some of the important results are presented here.

Layup is an important consideration in test specimen design and is relevant in delamination studies because delaminations often form due to interlaminar shear stresses between plies of different orientations. Early test configurations using angle ply laminates bordering the midplane experienced problems with crack branching (the crack departing from the original plane) [37]. Later angle ply configurations that avoided crack branching showed that the initiation toughness (i.e. initial propagation from an existing crack) is independent of ply orientation [39, 40]. Other studies showed that the only measured property that was a true material property, that is, independent of test specimen configuration, was the initiation toughness; crack propagation toughness was highly dependent on specimen geometry [41, 42] and even volume fraction [43]. In addition, fiber bridging was typically observed during crack propagation, but this was shown to be an artifact of the test method [37, 38, 44].

Another consideration in delamination experiments is the thickness of the insert used to create the artificial delamination. Thicker inserts can create large resin pockets ahead of the crack tip, which may alter initiation measurements. Expere-
iments using inserts of various thickness showed that results were independent of insert thickness for inserts 13 μm thick or less [37]. The most appropriate method for precracking from the insert received extensive consideration and there still is no clear consensus. Proposed methods for precracking have included fatigue loading, static Mode I loading, wedge insertion, and static Mode II loading. However, the current ASTM standard [45] recommends no precracking at all because precracking typically leads to fiber bridging, which skews the test results. Furthermore, the small size of the insert is believed to create a negligible resin pocket ahead of the insert.

There is also little consensus regarding an appropriate initiation criterion and strain energy release rate (SERR) calculation method. The ASTM standard lists three criteria for initiation (deviation from linearity, visual observation, and 5% offset/maximum load) and three calculation methods for SERR (modified beam theory, compliance calibration, and modified compliance calibration) [45]. Thus, the standard provides nine possible ways to acquire values of \( G_{IC} \), the critical Mode I interlaminar SERR. To add to the confusion, studies have shown that fracture propagation occurs in the interior of the DCB specimen prior to visual observation on the side of the specimen [46].

Fatigue delamination in PMCs has been studied extensively [47–50]. Nearly all studies cite difficulties associated with fiber bridging during the crack propagation phase of the experiments. The major problem with the bridging is that the rate of change of the fatigue crack propagation rate in a specimen with fiber bridging as a function of the maximum SERR is very high compared to unbridged systems. Thus, any uncertainty in the SERR will lead to large uncertainties in the crack propagation rates. Two methods have been suggested to deal with the uncertainties related to the fiber bridging in crack propagation and both methods essentially ignore the crack propagation rates. The first method recommends that delamination onset be used as the method for determining the end of fatigue life [51], where delamination onset is defined as the number of cycles to crack propagation from an existing delamination. The second method suggests using delamination threshold data, which is the applied SERR at which crack propagation does not occur [47]. Both methods
are recommended as conservative design practices. ASTM only has a standard for delamination onset, partially due to the uncertainties surrounding the fiber bridging [52].

Although there has been extensive research on delamination, there have been few studies examining the effects of temperature [53–55] or rate [54, 56] on delamination behavior and even fewer studies examining the combined effects of rate and temperature [57]. Furthermore, while delamination onset data are available in the literature [19], there have been virtually no published studies of fatigue delamination onset and propagation at elevated temperatures in PMCs.

Miyano et al. have done extensive work studying the relationship between quasi-static neat resin and composite behavior and composite fatigue behavior [36, 58]. They discovered that shifts in composite elastic and strength properties with temperature and loading rate were the same as the neat resin viscoelastic shift factors [58]. They also showed that the phenomenon holds for flexural composite strength [36]. Tsai and Kuraishi have recently extended application of the shift factors to a general durability framework [6].

The same types of studies have not been performed for fracture properties. $G - N$ curves (delamination onset as a function of cycles) are analogous to $S - N$ curves and thus are a natural application of any observed shifts in quasi-static neat resin or composite fracture behavior to fatigue predictions. However, Reeder et al. have measured the Mode I delamination toughness of a carbon fiber/thermoplastic composite as a function of temperature and loading rate. They found that the toughness values did not form a viscoelastic master curve [57], similar to those observed by Miyano.

It is clear from the literature that a study of neat resin and composite quasi-static and fatigue fracture behavior is warranted to determine whether shift factors in one instance may be applied in another instance. In addition, a comparison of neat resin and composite quasi-static and fatigue fracture mechanisms at a variety of temperatures has not been performed in the past and will provide insight regarding the potential to link constituent and composite quasi-static and fatigue properties, in accordance with the objectives of AIM-C.
4.2 Methods

The materials used in these experiments were IM7/977-3, a graphite/epoxy composite, and the composite’s neat epoxy resin, 977-3. The neat resin was provided in the form of approximately 150 x 100 x 3 mm plaques by the manufacturer, Cytec Engineered Materials. The composite material was fabricated in two different locations. Some of the specimens used in the quasi-static tests were fabricated at MIT using material provided by the manufacturer, Cytec Engineered Materials. All of the specimens used in the fatigue tests and some of the quasi-static specimens were fabricated by the Boeing Company. Every manufactured plate contained 28 unidirectional plies and a 13 µm thick Teflon* strip at the midplane that was long enough to produce a 63 mm long artificial delamination in the test specimens. Both locations processed the materials in accordance with manufacturer recommended guidelines, which include an autoclave cure at 0.59 MPa pressure and 179 °C for six hours. Quasi-static delamination tests on materials manufactured at both locations produced similar results (within 5% of each other), indicating that manufacturing location would not have a significant effect on the results of the study.

The neat resin plaques were cut into compact tension (CT) specimens, shown in Figure 4-1, using a milling machine. The dimensions were chosen in accordance with the ASTM standard CT specimen for testing the plane-strain fracture toughness of plastic materials [59], with a W dimension of 12.5 mm for the quasi-static tests and 25 mm for the fatigue tests. (A longer crack length was required in the fatigue tests to obtain a reasonable amount of crack propagation data.) A sharp pre-crack approximately 1.5 mm long was created in all specimens by tapping a 0.2 mm thick razor blade into the machined notch.

The composite material was cut into double cantilever beam (DCB) specimens using a diamond-bladed saw. The specimen and its dimensions are shown in Figure 4-2. The dimensions of the specimen were chosen in accordance with the ASTM standard [45]. Hinges were bonded to the specimens using a high temperature adhesive (Cotronics Duralco® 4525 260 °C Epoxy), with the exception of the specimens
Figure 4-1: Compact tension specimen. $W=12.5$ mm and $a_0=4.8$ mm for quasi-static specimens. $W=25$ mm and $a_0=8.0$ mm for DCB specimens. Thickness is 3 mm and $a=a_0+1.5$ mm in both cases.
used in tests at the highest temperatures (149 °C) where hinge peeling initially was a problem. The hinges were mechanically fastened to these specimens using two screws and tapped holes in these instances. The regions used for gripping in the hinges are longer than the bonded regions because the testing grips are located outside of the temperature chamber. In addition, the two different gripping lengths in the upper and lower hinges were required to allow the hinges to be inserted through the slots in the temperature chamber. When the specimen was placed in the chamber and the lower hinge was inserted into the access slot, the upper hinge could only be placed in the upper slot if it was shorter than the lower grip.

All tests were conducted using an Instron® 8552 servohydraulic testing machine. Load and displacement were recorded from the load cell and LVDT, respectively, using a LabView® data acquisition program at a frequency of 5 Hz. The program also allowed the user to click a button and electronically “mark” a specific instance in time.
All experiments were performed in a custom-built temperature chamber, shown in Figure 4-3, with interior dimensions measuring 400 mm long, 100 mm high, and 75 mm deep. The most important feature of the chamber is that the door is made of glass, allowing crack length to be measured during a test at an elevated temperature. A resistance heater provides heat and one thermocouple controls temperature and another thermocouple records temperature in another location of the chamber. The two temperature measurements indicated that there was acceptable temperature uniformity within the chamber (readings within 5 °C of each other). The CT tests were performed using clevis pins that were inside the chamber, while the DCB tests used hydraulic grips to clamp the hinges outside the chamber.

Quasi-static compact tension tests were performed in displacement control at 24, 66, 107, and 149 °C and at loading rates of 0.05, 0.5, and 2.5 mm/min following the ASTM standard [59]. Four specimens were tested at each test condition. The Mode I plane-strain critical stress intensity factor (fracture toughness), $K_{IC}$, was calculated using the relationship defined in the standard:

$$K_{IC} = \left( \frac{P_c}{B \sqrt{W}} \right) f(x)$$

(4.1)
where $P_C$ is the critical load, $B$ is the thickness of the specimen, $W$ is the width of the specimen, as defined in Figure 4-1, $x = a/W$ ($a$ is the crack length), and $f(x)$ is:

$$f(x) = \frac{(2 + x)(0.886 + 4.64x - 13.32x^2 + 14.72x^3 - 5.6x^4)}{(1 - x)^{\frac{3}{2}}} \quad (4.2)$$

In nearly all the tests the critical fracture load was clear because the fracture was quite brittle and hence there was a sharp drop in the load-displacement plot. However, at the highest temperature, there was some nonlinearity in the plot prior to the test reaching a maximum load. In these cases, the critical load was determined by drawing a line on the load-displacement plot that was identical to the linear load-displacement behavior but had a compliance 5% greater than the compliance in the test. The intersection of this line with the test data was considered the critical load. In all cases, $P_{max}/P_C < 1.1$, in accordance with the standard. Furthermore, all tests met the standard’s criteria for a valid plane strain test.

Critical Mode I strain energy release rates (toughness), $G_{IC}$, were calculated from the measured $K_{IC}$ values:

$$G_{IC} = \frac{(1 - \nu^2)K_{IC}^2}{E}. \quad (4.3)$$

The AIM-C resin module calculates Young’s modulus, $E$, and Poisson’s ratio, $\nu$, as a function of temperature using a model described in Reference [9] and experimental data. 977-3 is an epoxy with a $T_g$ of 150 °C and the mechanical properties do not change significantly with temperature until temperatures near the $T_g$. The resin module exemplified this behavior by calculating a modulus of 3.72 GPa and a Poisson’s ratio of 0.369 at the three temperatures below 149 °C and a modulus of 3.34 GPa and a Poisson’s ratio of 0.381 at 149 °C. These values were used in all quasi-static and fatigue $G_{IC}$ calculations.

The Mode I interlaminar toughness of the composite was determined from quasi-static displacement controlled tests performed in accordance with the ASTM standard [45] and at the same temperatures and loading rates used in the resin tests. Three to four specimens were tested at each test condition. Crack propagation was monitored using a Questar® traveling microscope. One side of each specimen was polished to
a 0.5 μm particle size polishing solution finish and was then coated with a semi-transparent reflective solution to improve the contrast between the crack and the surface. Fiducial marks were made using a fine ink pen every 1 mm ahead of the artificial crack tip for 10 mm and then every 5 mm after that up to a total of 45 mm ahead of the crack tip. The width of the marks and the accuracy of their placement was approximately ± 0.2 mm. All specimens were dried at 80 °C for thirty hours and then stored in a desiccator prior to testing.

As mentioned in Section 4.1, nine values of initiation toughness may be calculated from the ASTM standard. The experimental results showed that the nonlinear initiation criterion and the modified beam theory (MBT) calculation method provided the most conservative values. Audible cracking was often heard when the load-displacement behavior became nonlinear, which was prior to visual observation of crack growth. This is most likely due to the aforementioned tendency of crack growth to occur in the interior of the specimen prior to visual observation [46]. Thus, all SERR values presented here were calculated using the MBT method and all GIc values (Mode I initiation toughness) were determined from the load at which the load-displacement plot deviated from linearity.

The modified beam theory method treats the DCB specimen as if it contained a slightly longer delamination to correct for the rotation that is occurring in the beam at the delamination front. A correction factor, Δ, is added to the crack length, a, and is determined by plotting the cube root of the compliance, \( C^{1/3} \), as a function of the crack length. The compliance is the ratio of the total load point displacement, \( \delta \) (shown in Figure 4-2), divided by the load, \( P \). This plot should be linear and would normally begin at \( C^{1/3} \) and \( a \) values of zero, but in actuality \( C^{1/3} \) will be zero at a crack length value less than zero. \( \Delta \) is the distance on the x-axis from the plot’s x-intercept \( (C^{1/3}=0) \) to the origin. The SERR calculation is then:

\[
G_I = \frac{3P\delta}{2b(a + |\Delta|)}
\]

where \( b \) is the specimen width. Experiments were conducted until the crack had
propagated to the final marking on the side of the specimen (crack propagation of 45 mm). Load and displacement levels were recorded continuously and were “marked” when the crack reached each fiducial mark on the side of the specimen. This allowed for the \( C^{1/3} \) vs. \( a \) plot to be created in addition to a \( G \) vs. \( a \) plot (R-curve).

Mode I neat resin fatigue tests were performed following the ASTM standard for fatigue crack growth measurement [60]. Two tests were performed at 24 °C and two tests were performed at 149 °C, with the intention of providing a set of bounds for comparison with the composite tests. The experiments were performed in load control with an \( R \) ratio of 0.1 (\( P_{\text{min}}/P_{\text{max}} \)), a frequency of 5 Hz, and involved a fatigue precracking phase and a crack growth measurement phase. The final \( K_{\text{max}} \) of the precracking phase had to be less than the \( K_{\text{max}} \) used in the data collection phase, but a relatively high \( K_{\text{max}} \) was required to initiate the fatigue crack from the crack created by the razor blade. Thus, load shedding was used in the precracking stage by decreasing the maximum load by no more than 10% every 0.25 mm of crack growth until crack propagation rates averaged below \( 10^{-8} \) m/cycle. During the actual test, a constant maximum load was applied and crack growth rates were measured every 0.25 mm until the crack propagated in an unstable fashion. \( K \) and \( G \) calculations were made in the same manner as the quasi-static tests.

Mode I delamination onset in fatigue was measured in the composite specimens in accordance with the ASTM standard [52]. Tests were performed in displacement control at an \( R \) ratio of 0.1 (\( \delta_{\text{min}}/\delta_{\text{max}} \)), a frequency of 5 Hz, and at the same four temperatures used in the quasi-static tests. Four or five specimens were tested at each temperature at applied displacement ratios in the range of 0.1 to 0.8, depending on the test temperature. (The applied displacement ratio is defined as \( \delta_{\text{max}}/\delta_{\text{IC}} \), where \( \delta_{\text{IC}} \) is the average displacement value at initiation for tests conducted at a particular temperature and at the 0.5 mm/min displacement rate.) MBT calculations for \( G \) were made using average \( \Delta \) values from the 0.5 mm/min tests at each temperature. Tests were stopped regularly to monitor the maximum load and delamination growth onset was defined as an increase in compliance of 5%. Onset values were verified by visually checking the specimen for observable crack growth within a reasonable
amount of time after the indication of onset from the change in compliance.

Once delamination onset occurred, fatigue crack propagation rates were measured in all specimens using the traveling microscope to monitor crack growth. The accuracy of the crack length measurements was approximately ± 0.05 mm. Measurements were taken periodically (approximately every 0.5 mm of crack growth) until (in most cases) crack propagation rates reached below $10^{-8}$ m/cycle or the test reached $10^{5}$ cycles. Testing beyond $10^{5}$ cycles is extremely time consuming and typically yielded few data points.

4.3 Results

4.3.1 Neat Resin Quasi-Static Tests

Typical load-displacement behavior for the neat resin quasi-static tests is depicted in Figure 4-4. As mentioned above, nearly all tests exhibited brittle fracture characteristics with a clear load at which fracture occurred, as shown in the “brittle” plot in Figure 4-4. However, experiments at the highest temperature displayed nonlinear load-displacement behavior prior to fracture. Tests at the highest load rate showed nonlinear behavior and then brittle fracture, whereas tests at the slower load rates exhibited slow crack growth depicted by the “ductile” plot in Figure 4-4.

Neat resin quasi-static toughness as a function of loading rate and temperature is plotted in Figure 4-5. Average values are plotted along with the standard deviation. Lines on the plot are logarithmic fits through the data. The correlation coefficient, $R^2$, is a measure of the goodness of fit, with a value of 1 being a perfect fit. $R^2$ values for each fit were 0.99. The data for $K_{IC}$ and $G_{IC}$ are also shown in Tables 4.1 and 4.2, respectively. Although the trend is slight, there is a consistent increase in toughness with increasing loading rate at all temperatures. The toughness does not depend on temperature for the first three temperatures, but there is a measurable decrease in toughness at the highest temperature.

The difference between the brittle and ductile fracture mechanisms is clear in
Figure 4-4: Neat resin quasi-static brittle and ductile load-displacement behavior.

Figure 4-5: Neat resin quasi-static Mode I toughness as a function of displacement rate and temperature. Lines on the plot are logarithmic fits through the data. $R^2$ values for each fit are 0.99.
Table 4.1: Neat resin quasi-static Mode I $K_{IC}$ (MPa m$^{0.5}$) as a function of displacement rate and temperature. The coefficient of variation is in parentheses.

<table>
<thead>
<tr>
<th>Rate (mm/min.)</th>
<th>24 °C</th>
<th>66 °C</th>
<th>107 °C</th>
<th>149 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>0.75  (7.5%)</td>
<td>0.72  (2.9%)</td>
<td>0.74  (9.5%)</td>
<td>0.59  (5.2%)</td>
</tr>
<tr>
<td>0.5</td>
<td>0.77  (3.4%)</td>
<td>0.76  (13.1%)</td>
<td>0.75  (12.3%)</td>
<td>0.66  (5.7%)</td>
</tr>
<tr>
<td>2.5</td>
<td>0.77  (7.3%)</td>
<td>0.80  (11.6%)</td>
<td>0.78  (1.5%)</td>
<td>0.69  (10.2%)</td>
</tr>
</tbody>
</table>

Table 4.2: Neat resin quasi-static Mode I $G_{IC}$ (J/m$^2$) as a function of displacement rate and temperature. The coefficient of variation is in parentheses.

<table>
<thead>
<tr>
<th>Rate (mm/min.)</th>
<th>24 °C</th>
<th>66 °C</th>
<th>107 °C</th>
<th>149 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>130.3 (14.9%)</td>
<td>120.3 (5.7%)</td>
<td>127.0 (18.4%)</td>
<td>88.1 (10.4%)</td>
</tr>
<tr>
<td>0.5</td>
<td>136.9 (6.7%)</td>
<td>135.6 (25.3%)</td>
<td>133.8 (23.6%)</td>
<td>110.7 (11.6%)</td>
</tr>
<tr>
<td>2.5</td>
<td>139.5 (14.9%)</td>
<td>150.7 (23.3%)</td>
<td>140.6 (2.9%)</td>
<td>123.1 (20.8%)</td>
</tr>
</tbody>
</table>

micrographs of the fracture surfaces shown in Figure 4-6. The brittle fracture surfaces away from the crack tip were always smooth with no surface features, whereas the ductile fracture surfaces displayed a great deal of surface roughness. The mechanisms are clearly different in the two cases and have a significant effect on the initiation and propagation behavior.

### 4.3.2 Composite Quasi-Static Tests

The load-displacement behavior in the quasi-static composite tests was also dependent on temperature. Figure 4-7 demonstrates the difference in initiation behavior at the lower two temperatures and the higher two temperatures. Tests at 24 and 66 °C exhibited gradual nonlinearity in the load-displacement curves, followed by a small increase in load, and gradually finishing with a decreasing load. Some tests displayed a slight decrease in load after the nonlinearity that preceded the load increase. In contrast, many of the tests (but not all) at 107 and 149 °C reached a relatively high load and then suddenly decrease to a much lower load, followed by a gradual increase in load, and once again finished with a decreasing load.

The Mode I quasi-static delamination initiation toughness for the composite as a function of loading rate and temperature is shown in Figure 4-8 and the data are listed in Table 4.3. Once again, average values are plotted in addition to the
Figure 4-6: Neat resin static fracture surfaces at (a) 24 °C and 0.5 mm/min and (b) 149 °C and 0.005 mm/min.
Figure 4-7: Composite quasi-static load-displacement behavior at all temperatures.

Table 4.3: Composite quasi-static Mode I $G_{IC}$ (J/m²) as a function of displacement rate and temperature. The coefficient of variation is in parentheses.

<table>
<thead>
<tr>
<th>Rate (mm/min.)</th>
<th>24 °C</th>
<th>66 °C</th>
<th>107 °C</th>
<th>149 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>176.1 (10.3%)</td>
<td>188.1 (1.4%)</td>
<td>196.6 (10.0%)</td>
<td>187.5 (11.1%)</td>
</tr>
<tr>
<td>0.5</td>
<td>187.4 (3.0%)</td>
<td>177.8 (8.6%)</td>
<td>218.8 (4.0%)</td>
<td>217.6 (1.8%)</td>
</tr>
<tr>
<td>2.5</td>
<td>176.7 (2.8%)</td>
<td>177.6 (3.1%)</td>
<td>188.2 (4.4%)</td>
<td>233.5 (7.6%)</td>
</tr>
</tbody>
</table>

standard deviation and lines on the plot are logarithmic fits through the data. $R^2$ values for the 24, 66, 107, and 149 °C fits are 0.15, 0.92, 0.17, and 0.99, respectively, indicating that the 24 and 107 °C lines are poor fits of the data. It is difficult to discern any clear trends with respect to loading rate, but there does appear to be a slight increase in toughness at the higher temperatures. The temperature effect is particularly pronounced at the highest temperature.

Although the initiation toughness did not exhibit clear trends, the propagation behavior was markedly different at the four temperatures. Figure 4-9 depicts $G_R$ versus propagated crack length for the 0.5 mm/min tests, where $G_R$ is the strain energy release rate during propagation and the collection of these values makes up
Figure 4-8: Composite quasi-static Mode I interlaminar toughness as a function of displacement rate and temperature. Lines on the plot are logarithmic fits through the data. $R^2$ values for the 24, 66, 107, and 149 °C fits are 0.15, 0.92, 0.17, and 0.99, respectively.
Figure 4-9: Composite R-curves for the 0.5 mm/min displacement rate as a function of temperature. Each curve is an average of the third-order polynomial fits from all the tests at a particular temperature. On average, a particular $G_R$ value calculated from the curves had a coefficient of variation of 6% for 24 °C, 13% for 66 °C, 9% for 107 °C, and 12% for 149 °C.

In theory, the energy required to propagate a crack should be constant and independent of crack length. However, in these experiments fiber bridging in the propagated crack played a significant role in increasing the amount of energy required to propagate the crack. The higher R-curves at higher temperatures are indicative of
the larger role fiber bridging played at these temperatures. The bridging also explains
the higher propagation loads observed in tests at higher temperatures, as depicted in
Figure 4-7.

The fracture surfaces at nearly all temperatures and loading rates were virtu-
ally indistinguishable, demonstrating that the fracture mechanisms at initiation were
fairly independent of the test conditions. Figure 4-10 shows fracture surfaces near
the crack tip at the lowest and highest temperatures and at two different loading
rates. Brittle fracture appears to be the dominant fracture mechanism because none
of the surfaces appear similar to those seen in the neat resin ductile surfaces, shown
in Figure 4-6b. There also seem to be locations of fracture in resin rich regions where
relatively smoother surfaces are present.

Microscopy of the side of the specimens showed that there was a resin rich re-
gion ahead of the crack tip, shown in Figure 4-11. The fracture surfaces in Figure
4-10, particularly Figure 4-10a, appear to reinforce that crack propagation most likely
occurred in a resin rich region near the crack tip. There are large regions of resin de-
formation that do not show the “corrugated roof” surface indicating fibers embedded
in the surface.

4.3.3 Neat Resin and Composite Quasi-Static Fracture Mech-
anisms

The increase in initiation toughness as a function of loading rate exhibited by the
neat resin is most likely related to the fact that as loading rate increases deformation
resistance also increases [61]. Molecular chain relaxation occurs more at slower rates,
thereby decreasing deformation resistance. The decrease in toughness at the highest
temperature is explained by the decrease in yield strength that occurs at higher
temperatures [61]. It was mentioned in Section 4.2 that the modulus does not change
significantly until the temperature approaches the \( T_g \). This behavior is exemplified
in the results of a dynamic mechanical analysis (DMA) experiment performed using
977-3, which are shown in Figure 4-12. The tests were performed at the Kanazawa
Figure 4-10: Composite quasi-static fracture surfaces at (a) 24 °C and 0.5 mm/min and (b) 149 °C and 0.05 mm/min.
The plot shows the storage modulus and the loss tangent (loss modulus/storage modulus) as a function of temperature. It is evident from the plot that the modulus does not vary significantly until near the $T_g$ of 150 °C. Since yield strength typically scales with modulus, it comes as no surprise that the toughness does not change with temperature until the tests performed at 149 °C, nearly at the $T_g$.

The decreased yield strength and modulus help to explain why the initiation toughness decreased at higher temperatures, but they also help to explain why the behavior was more ductile. Indeed, even though the initiation toughness was less, the total work of fracture expended to propagate the crack was much higher in the 149 °C specimens. This is exemplified by the significantly larger area under the load-displacement curves for the ductile fracture specimens.

The load-displacement curves from the composite tests can be explained as a combination of initial propagation in a resin rich region followed by increasing amounts of fiber bridging, shown in Figure 4-13. The initial propagation causes the deviation from linearity and the drop in load, which precede the fiber bridging that increases the load. All effects appear to be magnified at higher temperatures. It is not clear how much of an effect the resin rich region has on initiation toughness since a resin
The rich region is found between most plies. The literature seems to indicate that the effect is minimal [37].

It is interesting to note that although the neat resin showed a decrease in toughness at higher temperatures, the composite showed the reverse trend of an increase in toughness at higher temperatures. The obvious key difference between the resin in the two materials is that in the composite the resin is constrained in an extremely thin resin rich interply layer. This constraining effect can have a significant effect on in situ resin deformation behavior, which will be explored further in Chapter 5. The decreased resin yield strength at higher temperatures presumably led to more plastic deformation and/or damage in the in situ constrained resin prior to initiation, and a consequently higher toughness.

The increase in bridging behavior with temperature is also presumably related to the decrease in resin yield strength at higher temperatures. The lower yield strength increases the likelihood that a crack will initiate ahead of a propagating crack. In addition, the fiber/resin interfacial properties may change with temperature and thereby affect the bridging properties.
Although both the resin and the composite initiation toughness showed shifts in behavior with temperature, the shifts were not clear enough to create a viscoelastic-type master curve, as has been done in Miyano's work for strength [58]. In addition, the variation in the composite experimental toughness data made it difficult to draw decisive conclusions. Indeed, the neat resin shifts in toughness could not be used to predict the composite shifts because trends of toughness with respect to temperature were opposite for the two materials.

4.3.4 Neat Resin Fatigue Tests

As mentioned in Section 4.2, the neat resin fatigue tests were performed merely as a set of bounds for comparison with composite fatigue crack growth rates. The crack propagation rates for all four tests (two at 24 °C and two at 149 °C) are shown in Figure 4-14 with a power law fit through the data at each temperature. \( R^2 \) values for each fit were 0.99. This is an indication of the consistency amongst the tests at each temperature.
Fatigue crack growth is typically described in terms of a power law relationship:

\[
\frac{da}{dN} = A (G_{\text{max}})^m
\]  \hspace{1cm} (4.5)

where \( da/dN \) is the crack growth rate and \( A \) and \( m \) are fitted parameters. On a log-log plot, \( m \) is the slope of a line and \( A \) is the \( y \)-intercept. The \( m \) values for the neat resin experiments are 4.9 and 3.3 for the 24 and 149 °C experiments, respectively, and are similar to those reported in the literature for epoxies [63].

It should be noted that fatigue crack propagation curves for most materials are shown as \( da/dN \) versus \( \Delta K \). However, crack propagation experiments using composites are typically measured using \( G \). In addition, fatigue experiments performed at an \( R \) ratio of 0.1 typically use \( G_{\text{max}} \) as the independent variable instead of \( \Delta G \) because of uncertainties surrounding \( G_{\text{min}} \) values [47]. These uncertainties are related to facial interference that occurs when the DCB faces come together during unloading. This interference is caused by a number of effects including fiber bridging, a plasticity zone...
wake, rough surfaces, and debris. Thus, the effects lead to uncertainties relating to $G_{\text{min}}$ values when an $R$ ratio of 0.1 is used and because of this $\Delta G$ is considered to be nearly equivalent to $G_{\text{max}}$. As such, all composite crack propagation data is plotted as $da/dN$ versus $G_{\text{max}}$. The neat resin data is also plotted in this fashion for comparison purposes.

The neat resin fatigue fracture surfaces at the two temperatures in Figure 4-15 are nearly indistinguishable and lack any significant features. There are no striations or signs of ductile fracture, which appears to indicate that brittle fracture is the dominant mechanism.

### 4.3.5 Composite Fatigue Tests

$G - N$ curves for the composite at the four test temperatures are plotted in Figure 4-16, which depicts Mode I delamination onset as a function of cycles. Lines on the plot are logarithmic fits through the data. $R^2$ values are 0.96 for 24 °C, 0.85 for 66 °C, and 0.99 for 107 and 149 °C. The maximum strain energy release rate values in the fatigue tests are normalized by the critical initiation values from the 0.5 mm/min tests, but since $G_{\text{IC}}$ values at different temperatures were quite close in the quasi-static tests, the relationships amongst the curves at different temperatures on the plot are representative of an unnormalized plot as well. The lines fit the data well for most of the temperatures and the trend with temperature is remarkably clear - delamination onset occurs at far fewer cycles for a given maximum applied strain energy release rate. This behavior was immediately clear from a test performed at 149 °C and an applied displacement ratio of 0.8. Propagation occurred almost immediately for the test and after this experience the applied displacement ratios ranged from 0.1 to 0.4 for the 149 °C tests, whereas values as high as 0.8 could be used for the 24 °C tests.

Crack propagation data for three to four composite specimens per temperature are shown in Figure 4-17. Data for specimens at the same temperature but different applied displacement ratios are shown using the same symbol and a power law fit through the data at each temperature. $m$ and $R^2$ values for each temperature are listed in Table 4.4. There is certainly a great deal of scatter in the data, particularly
Figure 4-15: Neat resin fatigue fracture surfaces at (a) 24 °C and (b) 149 °C.
Figure 4-16: Composite Mode I fatigue delamination onset as a function of cycles and temperature. Lines on the plot are logarithmic fits through the data. $R^2$ values are 0.96 for 24 °C, 0.85 for 66 °C, and 0.99 for 107 and 149 °C.

Table 4.4: Composite delamination propagation power law exponents and fit measures from all tests at each temperature.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$m$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>9.7</td>
<td>0.68</td>
</tr>
<tr>
<td>66</td>
<td>4.7</td>
<td>0.48</td>
</tr>
<tr>
<td>107</td>
<td>8.2</td>
<td>0.59</td>
</tr>
<tr>
<td>149</td>
<td>6.8</td>
<td>0.61</td>
</tr>
</tbody>
</table>

in the data from different tests at the same temperature, indicating that the applied displacement ratio has an effect on the test data. The scatter is represented by the relatively low $R^2$ values.

One can see the effect of applied displacement ratio more clearly in Figures 4-18 and 4-19, which each plot crack propagation rates at two temperatures for three separate applied displacement ratios per temperature. Once again, lines on the plots are a power law fit through the data. The data from each applied displacement ratio are quite linear, but there are clear shifts between the tests at the same temperature. Power law exponents and $R^2$ values for individual tests are listed in Table 4.5. The
Figure 4-17: Composite delamination propagation rates as a function of maximum strain energy release rate for different temperatures. Lines are a power law fit through the data. $m$ and $R^2$ values for each temperature are listed in Table 4.4.
Figure 4-18: Composite delamination propagation rates as a function of maximum strain energy release rate at 24 and 66 °C and three applied displacement ratios per temperature. Lines are a power law fit through the data.

fits for the individual tests are quite good and the slopes of the plots are much higher than the values for all tests at the same temperature. This indicates that the average values for each temperature do not adequately account for the applied displacement ratio effect.

As with the neat resin fatigue tests and quasi-static composite tests, the fracture surfaces for the composite fatigue tests at different temperatures and different applied loading ratios were nearly indistinguishable from one another. Fracture surfaces from tests at 24 °C and 149 °C are shown in Figure 4-20. There are no striations and as in the other tests, brittle fracture appears to be the dominant mechanism.
Figure 4-19: Composite delamination propagation rates as a function of maximum strain energy release rate at 107 and 149 °C and three applied displacement ratios per temperature. Lines are a power law fit through the data.

Table 4.5: Composite delamination propagation power law exponents and fit measures from fatigue crack propagation tests.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$\delta_{max}/\delta_{IC}$</th>
<th>$m$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>0.8</td>
<td>17.0</td>
<td>0.96</td>
</tr>
<tr>
<td>24</td>
<td>0.6</td>
<td>14.8</td>
<td>0.80</td>
</tr>
<tr>
<td>66</td>
<td>0.8</td>
<td>16.9</td>
<td>0.97</td>
</tr>
<tr>
<td>66</td>
<td>0.6</td>
<td>11.1</td>
<td>0.87</td>
</tr>
<tr>
<td>66</td>
<td>0.5</td>
<td>17.3</td>
<td>0.97</td>
</tr>
<tr>
<td>66</td>
<td>0.4</td>
<td>12.9</td>
<td>0.95</td>
</tr>
<tr>
<td>107</td>
<td>0.6</td>
<td>13.6</td>
<td>0.95</td>
</tr>
<tr>
<td>107</td>
<td>0.5</td>
<td>10.2</td>
<td>0.94</td>
</tr>
<tr>
<td>107</td>
<td>0.4</td>
<td>10.2</td>
<td>0.99</td>
</tr>
<tr>
<td>149</td>
<td>0.8</td>
<td>9.1</td>
<td>0.91</td>
</tr>
<tr>
<td>149</td>
<td>0.4</td>
<td>12.2</td>
<td>0.96</td>
</tr>
<tr>
<td>149</td>
<td>0.3</td>
<td>9.0</td>
<td>0.86</td>
</tr>
</tbody>
</table>
Figure 4-20: Composite fatigue fracture surfaces at (a) 24 °C and 0.4 applied displacement ratio and (b) 149 °C and 0.4 applied displacement ratio.
4.3.6 Neat Resin and Composite Fatigue Fracture Mechanisms

The fatigue crack propagation in the neat resin is interesting in the respect that the quasi-static experiments showed unstable brittle crack propagation at ambient temperature and stable ductile crack propagation at high temperatures, yet the fatigue experiments exhibited stable crack propagation and did not show either the smooth, glassy fracture surface characteristic of brittle propagation or the rough fracture surface characteristic of ductile propagation. Rather, the fatigue surfaces appeared to have different characteristics without any clear striations.

Mechanisms of fatigue crack propagation are quite complex and the exact processes are for 977-3 are unclear from the microscopy. However, one may hypothesize that the fatigue propagation mechanism was ductile fracture because the propagation proceeded in a stable manner. If ductile fracture is occurring at the crack tip, one may make an estimate of the cyclic plastic zone size, $r_c$ [64]:

$$r_c = \frac{1}{\pi} \left( \frac{K_{\text{max}}}{2\sigma_y} \right)^2$$  \hspace{1cm} (4.6)

where $\sigma_y$ is the yield strength. The cyclic crack opening displacement ($CCOD$) is merely the cyclic plastic zone size multiplied by the strain at yielding, $\varepsilon_y$. If one uses the assumption of a nonhardening material, then $\varepsilon_y$ is merely $\sigma_y/E$, where $E$ is the modulus, and $CCOD$ becomes:

$$CCOD = \frac{1}{4\pi} \frac{K_{\text{max}}^2}{\sigma_y E}.$$  \hspace{1cm} (4.7)

Equation 4.3 may be substituted into Equation 4.7 to obtain $CCOD$ as a function of $G$. The crack growth per cycle, or $da/dN$ is directly related to and on the order of $CCOD$ and hence, we can use the expression for $CCOD$ to understand $da/dN$ [64]:

$$\frac{da}{dN} = O(CCOD) = \frac{1}{4\pi} \frac{G_{\text{max}}}{(1 - \nu^2)\sigma_y}.$$  \hspace{1cm} (4.8)
One may now make an estimate of the expected crack growth rate for a given SERR. Figure 4-14 shows that for a $G_{\text{max}}$ of 50 J/m$^2$, $da/dN$ is approximately $10^{-7}$ m/cycle. Using a $\sigma_y$ value of 131.7 MPa (determined from measurements described in Section 5.2) and a Poisson’s ratio of 0.37, one calculates a $da/dN$ value of 0.35 $10^{-7}$ m/cycle, or 35 nm/cycle.

The results of this first order analysis provide insight on several key issues. First, the calculated $da/dN$ is on the same order of magnitude as the measured $da/dN$, indicating that the assumption of ductile fatigue propagation is consistent. If the fatigue propagation is indeed ductile, this would explain the stable crack propagation. Second, the calculation and the measurements indicate that any striations would be on the order of hundreds of nanometers, which is below the visible range of the surfaces depicted in the micrographs in Figure 4-15. The combination of these tiny striations, however, would give the fatigue surfaces the non-smooth appearance. Finally, the relationship in Equation 4.8 shows that as the yield strength decreases at higher temperatures, the fatigue crack propagation rates increase, which was observed experimentally.

Composite delamination onset values are clearly affected by temperature and as in the quasi-static tests this is directly related to resin yield strength decreasing as the temperature increases. A lower resin yield strength will allow more resin deformation per cycle, which increases the likelihood of delamination onset from an existing flaw. The resin behavior at high temperatures also affects the crack propagation rates. For a given maximum applied SERR, crack propagation rates are definitely higher at higher temperatures in the neat resin and appear to be higher at higher temperatures in the composite, although the scatter in the composite data prevents statistical significance being assigned. The lower resin yield strength at high temperatures and the higher levels of resin deformation per cycle mean that more crack propagation is likely to occur at high temperatures, as shown previously in the ductile crack growth first order analysis.

The scatter in the composite fatigue crack propagation data, exemplified by the low $R^2$ values, is undoubtedly due to the fiber bridging that occurred during the
crack propagation. The bridging was visually observed, but it also manifested itself in the slope of the crack propagation plots from each test. Values for $m$ in metals are typically below 5 [65]. In these experiments, values for $m$ from individual tests ranged from 9 at the highest temperature to over 17 at the lower temperatures. These high values lead to the aforementioned uncertainties related to predictions of crack propagation rates.

An alternative method has been proposed to account for the bridging in fatigue crack propagation by normalizing $G_{I_{\text{max}}}$ values by $G_R$ values in the power law relationship [51]. The $G_R$ values are derived from the quasi-static R-curves for a specified crack length. Since $G_R$ values take into account excess bridging energy, the normalization theoretically eliminates the bridging contribution. This normalization was done for these tests using the 0.5 mm/min quasi-static test data and the results are plotted in Figure 4-21, which includes power law fits for the data at each temperature. Table 4.6 lists $m$ and $R^2$ values for all tests at each temperature and Table 4.7 lists values from individual tests. The scatter in the data is less than the unnormalized data ($R^2$ values in Table 4.6 are much higher than those in Table 4.4) and the trend of higher growth rates at higher temperatures is easier to see. The collapsing of the data from different tests at the same temperature onto a single curve by accounting for the fiber bridging indicates that the bridging has a significant impact on the fatigue crack propagation. In addition, the slopes of the normalized curves in Table 4.7 are less than the unnormalized curves' slopes (Table 4.5) and are much more representative of structural, or unbridged, crack propagation. The major drawback of the $G_R$ normalization method is that it has no physical basis because the derived $G_R$ curve is specimen dependent and therefore not a material property.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$m$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>9.5</td>
<td>0.91</td>
</tr>
<tr>
<td>66</td>
<td>4.5</td>
<td>0.73</td>
</tr>
<tr>
<td>107</td>
<td>5.1</td>
<td>0.91</td>
</tr>
<tr>
<td>149</td>
<td>4.2</td>
<td>0.89</td>
</tr>
</tbody>
</table>
Figure 4-21: Composite delamination propagation rates as a function of maximum strain energy release rate normalized by R-curve values and temperature. Lines are a power law fit through the data at each temperature.

Table 4.7: R-curve normalized composite delamination propagation power law exponents and fit measures from individual fatigue tests.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$\delta_{\text{max}}/\delta_{IC}$</th>
<th>$m$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>0.8</td>
<td>10.6</td>
<td>0.97</td>
</tr>
<tr>
<td>24</td>
<td>0.6</td>
<td>8.4</td>
<td>0.80</td>
</tr>
<tr>
<td>66</td>
<td>0.8</td>
<td>7.3</td>
<td>0.94</td>
</tr>
<tr>
<td>66</td>
<td>0.6</td>
<td>5.0</td>
<td>0.89</td>
</tr>
<tr>
<td>66</td>
<td>0.5</td>
<td>7.6</td>
<td>0.95</td>
</tr>
<tr>
<td>66</td>
<td>0.4</td>
<td>5.1</td>
<td>0.94</td>
</tr>
<tr>
<td>107</td>
<td>0.6</td>
<td>5.5</td>
<td>0.94</td>
</tr>
<tr>
<td>107</td>
<td>0.5</td>
<td>3.6</td>
<td>0.92</td>
</tr>
<tr>
<td>107</td>
<td>0.4</td>
<td>4.9</td>
<td>0.99</td>
</tr>
<tr>
<td>149</td>
<td>0.8</td>
<td>4.4</td>
<td>0.89</td>
</tr>
<tr>
<td>149</td>
<td>0.4</td>
<td>4.8</td>
<td>0.96</td>
</tr>
<tr>
<td>149</td>
<td>0.3</td>
<td>3.0</td>
<td>0.84</td>
</tr>
</tbody>
</table>
The fiber bridging helps to explain some of the observed characteristics in the fatigue crack propagation plots. Figure 4-19 shows that tests with smaller applied displacement ratios shift to the left. That is, for a given $G_{\text{max}}$, crack propagation rates will be higher for smaller applied displacements. This is due to the fact that bridging forces are lower for smaller applied displacements, thereby allowing higher crack propagation rates.

Figure 4-22 shows a comparison of the neat resin and composite fatigue crack propagation data at 24 and 149 ºC. The actual SERRs that cause particular crack propagation rates are not suitable for comparison because of the different constraints on the resin in the neat resin and the composite tests. However, it is useful to compare the slopes of the curves, which are shown in Figure 4-14 for the neat resin and Tables 4.4 and 4.5 for the composite. Values for $m$ in the neat resin tests are 4.9 for the 24 ºC tests and 3.3 in the 149 ºC tests, whereas $m$ values for the unnormalized composite data are near 15 for the individual 24 ºC tests and approximately 10 for the individual 149 ºC tests. (The line in the plot for the 149 ºC composite data is obviously a poor fit because it includes several tests at different applied displacement ratios. It has an $R^2$ value of 0.61 for that temperature.) Values for $m$ from the $G_R$ normalized data are approximately 9 for the 24 ºC tests and 4 for the 149 ºC tests, which are closer to the neat resin values.

### 4.4 Conclusions

The objectives of this work were to compare the neat resin and composite quasi-static and fatigue fracture behavior to determine whether links could be made between the lengthscales in terms of qualitative mechanisms and quantitative predictions. The experimental results clearly indicate that the mechanisms in the neat resin experiments directly explain the observed behavior in the composite. The neat resin quasi-static toughness slightly increased with increasing loading rate, but was independent of temperature until near the $T_g$ where the initiation toughness decreased. However, the fracture toughness decreased and fracture was more ductile at the highest tem-
Figure 4-22: Neat resin and composite crack propagation rates as a function of maximum strain energy release rate at two temperatures.
perature, as opposed to the brittle fractures observed at all other temperatures. The composite quasi-static toughness did not exhibit any clear trends with respect to loading rate or temperature, but it appeared that toughness was slightly higher at higher temperatures, the opposite shift from the neat resin. The constraint on the resin in the composite most likely combines with the ductility of the matrix to produce this increased toughness at elevated temperatures.

Delamination fatigue behavior in the composite was heavily dependent on temperature. This was exemplified by the delamination onset curves, which indicated that higher temperatures significantly decrease the number of cycles to delamination onset for a given applied maximum strain energy release rate. The fatigue crack propagation curves also appeared to indicate that higher temperatures caused higher levels of fatigue crack propagation for a given $G_{\text{max}}$ as shown by the higher $m$ values and shifted curves, although no statistical significance can be ascribed to this observation because of the scatter in the data. Normalization of the data using $G_R$ information reduced the scatter in the data and reinforced the hypothesis that fiber bridging was affecting the tests. The drawback of the $G_R$ method is the lack of a physical basis. The problems associated with bridging in fatigue motivate the need for a model that will separate bridging and crack tip propagation contributions. This model was created as part of this work and is presented in Chapter 6 where this fatigue data will be examined again.

The quantitative composite strength predictions that can be made using resin viscoelastic parameters are not possible for fracture because the neat resin and composite quasi-static data do not follow viscoelastic-type master curves and the shifts in behavior are different for the two materials. Since the relationship cannot be established for quasi-static cases, quantitative information cannot be used in the fatigue case. However, the neat resin quasi-static behavior and mechanisms were crucial in explaining composite quasi-static and fatigue behavior. In particular, the trends of resin toughness with temperature were particularly important for describing the trends in composite fatigue behavior. Composite fatigue tests are quite expensive, so an understanding of the mechanisms that determine fatigue shifts with temperature
can be an important contribution to determining the crucial experiments that need to be conducted. Given that the neat resin tests are quite simple to perform, future composite material insertion programs would benefit from knowing the neat resin fracture behavior.

An interesting outcome of these experiments is that they point to the need to perform quasi-static and fatigue experiments at a range of temperatures. For example, the quasi-static composite tests indicated that fracture properties at elevated temperature were better than those at ambient conditions. This could lead one to assume that fatigue tests should be performed at ambient conditions because this would be the critical fracture condition. These experiments indicated that that is certainly not the case. Indeed, the elevated temperature fatigue fracture behavior was significantly worse than the ambient fracture behavior, making the high temperature condition more critical for durability estimation.
Chapter 5

Delamination Initiation Model

An important outcome from the previous chapter is that the constrained resin in the composite interlaminar region does not necessarily behave the same as the unconstrained neat resin. Indeed, there were significant differences between neat resin and composite critical SERRs as a function of temperature and there were even different mechanisms observed at the highest temperature. The constraint on the resin in the composite is the most likely cause of these differences.

This chapter describes an attempt to develop a predictive tool that can determine the delamination initiation toughness of a composite material based on the constituent properties. Based on the experimental studies, this model particularly analyzes the constrained resin layer in between plies in a double cantilever beam specimen as a key determinant of toughness. A finite element model of a DCB is created to analyze the behavior of the composite at the global and local (constituent) level. The results of the numerical analysis must then be coupled with an appropriate fracture criterion.

5.1 Background

Epoxies have been used in composite materials because of their relatively high stiffness and stable behavior in hot/wet conditions. However, their Achilles’ heel has been their poor fracture toughness, which often manifests itself in delamination or off-axis ply cracking. Many experimental studies have been conducted that have a
particular emphasis on examining the role of the matrix on the composite fracture process. Figure 5-1 shows a collection of Mode I interlaminar toughnesses for several composites versus their associated matrix toughnesses [66]. It is interesting to note that composites that contain brittle resins have fracture energies higher than those of their neat resins while composites with tougher matrices are more fracture resistant but do not benefit from the increase in neat matrix toughness to nearly the same extent.

Many studies were performed to determine which mechanisms played a key role in the interlaminar fracture process. Several fracture mechanisms were discovered through delamination examinations including fiber bridging, fiber debonding, fiber pullout, cohesive resin fracture, microcracking ahead of the crack tip, and resin crack tip plastic deformation [67, 68]. However, it is clear that plastic deformation and/or damage at the crack tip of the matrix is the dominant mechanism in interlaminar fracture, assuming adequate fiber/matrix interfacial strength, because these processes
require the most energy. The energy consumed in the inelastic crack tip deformation is much larger than the energy required to create a matrix crack surface cohesively and the energy involved in separating a fiber/matrix interface [69]. Improving fiber/matrix interfacial strength improved toughness in several studies because poor interfacial bonding tends to “short-circuit” the resin deformation process and induces premature fracture [70, 71]. In systems with well characterized fiber/matrix interfaces, debonding should not be a critical failure mechanism.

Several hypotheses were proposed based on the experimental evidence to explain the change in composite toughness with increasing matrix toughness. Explanations for the higher composite toughness than the associated constituent brittle resin included the increased surface area of the composite fracture surface (analogous to a “corrugated roof”) over a neat resin fracture surface, the impinging of cracks on misaligned fibers, and the stress state ahead of the crack tip decaying more slowly than that in the neat resin, causing stresses to be more evenly distributed [67]. Other analyses of the constrained resin stress state have indicated that in composites the fibers can create stress concentrations in the resin and increase the extent of matrix yielding and therefore the fracture toughness [72], whereas in adhesive joints a decrease in thickness causes the local tensile stresses ahead of the crack tip to act over a much longer distance than in the bulk material, creating a much longer plastic zone [73].

Although there is little consensus as to what mechanism is primarily responsible for composites with brittle resins, there is general agreement on the explanation for the decreased toughness of a composite with a ductile matrix when compared to the neat matrix toughness. The argument is that the fibers reduce the amount of matrix volume available to deform or microcrack and thus absorb energy that would otherwise be applied to matrix damage in the relatively large plastic zone created in a ductile matrix [67]. Thus, even though the composite toughness is greater than that of composites with brittle resins, the potential of the tougher matrix is not fully realized.

In other related experimental work, Chai attempted to correlate the toughness
of resins in adhesive bonds to composite interlaminar fracture toughness [41]. The work has shown that the composite toughness coincides with the adhesive toughness, given that the thickness of the adhesive layer is sufficiently small (on the order of the thickness of a resin rich region in a composite). Other adhesive bond studies examined the effect of the bond thickness on the bond toughness [74]. Thicker bonds had a toughness equal to that of the bulk adhesive while thinner bonds had a significantly reduced toughness. These results indicate that the examination of a crack constrained by rigid adherends can provide critical information relating to composite fracture toughness.

There has been little work in the area of theoretical predictions of composite fracture toughness based on constituent properties. Two models have been created to make such predictions, but they fall short of the goal because they rely on parameters that cannot be directly measured [69, 75]. Crews et al. created a finite element model using discrete fibers and resin at the crack tip to examine the constraint that the fibers have on the resin, but they did not actually make toughness predictions [72]. The analysis was purely elastic and made simple estimates on plastic zones to investigate the likelihood that the fibers were inducing yielding in the resin and thereby increasing toughness. Crews' modeling work has also shown that a two-dimensional plane strain model of a double cantilever beam specimen is an appropriate approximation of the fully three-dimensional model [76].

5.2 Description

The review of the literature and the experimentally observed fracture mechanisms clearly indicate that plastic deformation and/or damage at the crack tip in the matrix of a composite is the primary energy dissipative mechanism in interlaminar fracture. Hence, an FEM was created that could specifically account for the inelastic deformation in the resin at the crack tip. This is best accomplished by discretely analyzing the resin and fibers at the crack tip in a manner similar to that used by Crews [72]. The fibers create the appropriate constraint on the resin, while the resin in the re-
A global homogeneous composite model determines the applied displacements for the local model. The applied displacements for the local model determine the deformation state at the crack tip and from this the $J$-integral is calculated. The global $J$-integral represents the strain energy release rate that would be determined experimentally. The local $J$-integral accounts for the inelastic deformation in the resin and this value is compared with a resin-critical fracture criterion. The global $J$-integral that results in the local value that meets the resin fracture criterion is the interlaminar fracture toughness of the composite.
The model is depicted schematically in Figure 5-3, which also shows the dimensions for the global and local models, and was generated and run using the finite element software ABAQUS®. The global model is a 2-D symmetric plane strain DCB that contains elastic homogeneous composite properties. A point load is applied to the tip of the global model while the displacements for the local model are calculated and applied to the local model automatically by the ABAQUS® *SUBMODEL command. The local model is also 2-D, plane strain, and symmetric and is one ply thick. It contains a resin-rich layer at the crack tip beneath four layers of fibers alternating with three layers of resin. A homogeneous composite region surrounds these layers. Fibers are elastic and the resin is elastic-plastic; perfect bonding is assumed. Eight-noded, biquadratic quadrilateral plane strain continuum elements were used in both models. The meshes near the crack tips in each model are shown in Figures 5-4 and 5-5. (Sample input files for the global and local models are included in Appendices A.2 and A.3, respectively.)

The J-integral is evaluated in the global and local models using the ABAQUS® *CONTOUR INTEGRAL command. This calculates the J-integral in successive contours around the crack tip until a final value is converged upon. If the model is elastic the ABAQUS® command produces the same results as the virtual crack closure technique (VCCT), but without any post-processing. In this situation, however, the VCCT cannot be used due to the inelastic nature of the resin deformation. The global model is elastic and hence its J-integral coincides with the closed form solution for the strain energy release rate in a DCB specimen determined by Bao et al. [77], which includes a numerically determined correction factor to the elementary beam theory that accounts for shear effects and orthotropy.

The material properties used in this model are a combination of experimental results presented in the previous chapter, calculated properties from micromechanical models, and an additional experimental calibration performed specifically for this model. The properties are listed in Table 5.1. The fiber and resin properties were obtained from an AIM-C database, which included information from the manufacturer. Transverse fiber properties were estimated from composite properties \( (E_2, G_{23}, \nu_{23}) \).
Figure 5-3: Global and local FEM schematic.
Figure 5-4: Global crack tip mesh.
Figure 5-5: Local crack tip mesh. The resin-rich layer in this mesh is 5 μm thick.
Table 5.1: Material Properties used in the FEM.

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_1$ (GPa)</th>
<th>$E_2$ (GPa)</th>
<th>$G_{12}$ (GPa)</th>
<th>$G_{23}$ (GPa)</th>
<th>$\nu_{12}$</th>
<th>$\nu_{23}$</th>
<th>$G_{IC}$ (J/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M7/977-3</td>
<td>168.0</td>
<td>9.55</td>
<td>4.35</td>
<td>3.42</td>
<td>0.26</td>
<td>0.40</td>
<td>190</td>
</tr>
<tr>
<td>IM7 Fibers</td>
<td>277.0</td>
<td>17.2</td>
<td>18.6</td>
<td>4.83</td>
<td>0.20</td>
<td>0.25</td>
<td>-</td>
</tr>
<tr>
<td>77-3 Resin</td>
<td>3.8</td>
<td>3.8</td>
<td>1.39</td>
<td>1.39</td>
<td>0.37</td>
<td>0.37</td>
<td>140</td>
</tr>
</tbody>
</table>

Lamina properties were calculated using standard micromechanics equations [4] in order to maintain the AIM-C methodology of determining composite properties from constituents. Calculated composite properties were similar to those reported by the manufacturer [32]. The dimensions of the model including DCB size, ply thickness, and fiber spacing were determined from the experimental specimens, whereas the diameter of the fibers was provided in the material properties.

An important material definition in the FEM is the inelastic behavior of the resin. This was not available, so compressive tests were performed using the neat resin in order to obtain a measure of the material’s inelastic deformation behavior. Compression tests were performed, as opposed to tensile tests, because tensile tests on the brittle material would have resulted in fracture before any meaningful inelastic deformation data could be obtained. The tests were conducted in accordance with the ASTM standard for rigid plastic materials [78]. Right prism specimens with a rectangular cross-section measuring 3 mm x 6 mm x 12 mm were machined from the resin plaques and tested using the long axis in compression. The specimens were tested in a servohydraulic testing machine by inserting them in between compression discs with smooth polymer faces and placing Teflon® in between the specimen ends and the disc faces to reduce friction. One disc mated into a ball and cup joint to minimize misalignment effects. Tests were performed using a loading rate of 0.1 mm/min.

The stress-strain plots were always nonlinear, as shown in Figure 5-6. The non-linearity appeared to be due to inelastic deformation because unloading was linear, did not follow the same path as the loading, and completed at a finite displacement, indicating permanent deformation. Average results for five specimens are shown in
Table 5.2: 977-3 Neat Resin Compressive Properties using Ramberg-Osgood model. Numbers in parentheses are standard deviation.

<table>
<thead>
<tr>
<th>(\sigma_y) (MPa)</th>
<th>(\sigma_m) (MPa)</th>
<th>(\alpha)</th>
<th>(n)</th>
</tr>
</thead>
<tbody>
<tr>
<td>91.4 (2.6%)</td>
<td>131.7 (5.0%)</td>
<td>0.063 (4.8%)</td>
<td>4.64 (3.5%)</td>
</tr>
</tbody>
</table>

Table 5.2 with standard deviation in parentheses. \(\sigma_y\) is the 0.2% offset yield stress, \(\sigma_m\) is the maximum stress, and \(\alpha\) and \(n\) are fitted Ramberg-Osgood parameters, as defined by the plasticity model (\(E\) is modulus, \(\sigma\) is stress, and \(\varepsilon\) is strain):

\[
E \varepsilon = \sigma + \alpha \left( \frac{|\sigma|}{\sigma_y} \right)^{n-1} \sigma. \tag{5.1}
\]

Although many polymers used as matrices in composite materials, such as epoxies, behave in a brittle fashion in bulk form, epoxy crack tip fracture processes are typically ductile [79, 80]. In addition, the constraint on the resin in the composite can increase the maximum strain levels the material can withstand prior to failure [67]. However, it is difficult to determine the precise inelastic constitutive behavior of the resin in this constrained layer, although some form of hardening is likely involved. Thus it is
appropriate to examine a variety of resin inelastic material definitions.

The model was analyzed using a total of five different inelastic constitutive models, three of which are depicted schematically in Figure 5-7: perfect plasticity, deformation plasticity (Ramberg-Osgood behavior), Drucker-Prager behavior (the material exhibits pressure dependence in plasticity), and two types of hardening behavior of the form shown in the figure, which is typical for many polymers. The Drucker-Prager plasticity model was chosen to represent a material behavior that exhibits extensive amounts of inelastic deformation and would therefore act as an upper bound on plasticity levels.
The yield strength ($\sigma_y$) from Table 5.2 was used in all models. Deformation plasticity was defined using the parameters in Table 5.2. The Drucker-Prager model used pressure sensitivity parameters of $K = 1$ and $b = y = 20$ degrees, which are typical for a polymer. Finally, both hardening models used a $\sigma_{max1}$ value (shown in Figure 5-7) equal to the maximum stress listed in Table 5.2, but the first model used a $\sigma_{max2}$ value equal to 260 MPa and a $\varepsilon_{max}$ value of 0.225, whereas the second model used a $\sigma_{max2}$ value equal to 1000 MPa and a $\varepsilon_{max}$ value of 0.235. ABAQUS® assumes that the material is perfectly plastic once the maximum stress and strain are reached in the two hardening models. That is, for all plastic strains higher than $\varepsilon_{max}$, the stress in the material remains equal to $\sigma_{max2}$.

5.3 Results

A parametric study was conducted to determine the sensitivity of the model to the resin-rich region thickness and the incorporation of plasticity to the resin behavior. Figure 5-8 shows the results of the analyses in which a 200 N load was applied to the global model, resulting in a global $J$-integral of 332.0 $J/m^2$. The plot compares resin behavior defined as elastic, perfectly plastic, or by deformation plasticity. Varying the resin plastic behavior in the model serves to examine the model’s applicability to materials with different plastic behavior. Note that the values listed for the resin-rich region in the model are actually half the thickness of the resin-rich region in the actual composite. A 2 $\mu$m gap is a typical interfiber spacing while a 30 $\mu$m region exists immediately after the insert in Figure 4-11.

For this load a variation in thickness can change the $J$-integral by over 10% and it is clear that increasing the constraint on the crack tip (i.e. decreasing the thickness) increases the $J$-integral. This is expected because the crack tip stresses increase as the constraint is increased. Other studies in which the stiffness of the constraining layer was increased validated this behavior. As the stiffness increased the stresses increased and hence the $J$-integral increased. Plasticity has the effect of relieving the stresses at the crack tip and hence produces lower $J$-integral values. Perfect plasticity
Figure 5-8: Computed local $J$-integrals for various resin-rich thicknesses and resin behaviors.
creates more yielded material than deformation plasticity and thus it has more of a stress relieving effect.

One can clearly see the effect that the constraint has on the resin in Figure 5-9. The resin constitutive behavior is perfectly plastic and all contour plots show equivalent plastic strain and depict the undeformed state. Figure 5-9a and Figure 5-9c are of the local composite model while Figure 5-9b and Figure 5-9d are of a crack tip in a neat resin compact tension model. The dimensions are the same in all the pictures and the globally applied load is the same for the composite models, 200 N. The neat resin models have the same J-integral values as their corresponding composite models. The areas in black at the crack tip are regions where the equivalent plastic strain is greater than 0.1, whereas other non-white shaded regions denote equivalent plastic strain less than 0.1 but greater than zero. Note that the regions containing fiber and composite properties have zero plastic strain because they are defined as purely elastic.

Table 5.3 shows the amount of black area and the total amounts of plastically deforming area for all the plots. The black areas in the resin-rich region for both composite models are approximately the same - slightly over 8 \( \mu m^2 \). However, the total yielded area in the model with the thinner region is 12 \( \mu m^2 \) while it is 169 \( \mu m^2 \) in the model with the thicker region. This is compared with 174 \( \mu m^2 \) and 116 \( \mu m^2 \) in the associated CT models. Furthermore, the magnitude of the highest plastic strains is much higher in the model with the thinner region. (The actual values of the strains are unrealistically high because of the perfectly plastic material assumption.) Two important conclusions can be drawn from these comparisons. The intensity of the strains is higher for a thinner resin-rich region, but the total amount of yielded area is less. Furthermore, the intensity of the crack tip strains is much higher than the resin in the upper layers, indicating that the energy dissipation at the crack tip is most likely the largest contribution to the work of fracture.

Another way of examining the intensity of the plastic strains is by investigating the amount of energy dissipated through plastic deformation. This value is calculated by ABAQUS® for the entire model and is associated purely with the resin inelastic deformation since the resin is the only material plastically deforming. Values of
Figure 5-9: Equivalent plastic strain in local composite models with different resin-rich region thicknesses and neat resin compact tension models at the same $J$-integral levels. The resin constitutive behavior is perfectly plastic. The areas in black at the crack tip are regions where the equivalent plastic strain is greater than 0.1, whereas other non-white shaded regions denote equivalent plastic strain less than 0.1 but greater than zero. (a) Composite model, $t=1 \, \mu m$, $J=335.0 \, J/m^2$, (b) Neat resin model, $J=335.0 \, J/m^2$, (c) Composite model, $t=15 \, \mu m$, $J=299.7 \, J/m^2$, (d) Neat resin model, $J=299.7 \, J/m^2$. 
Table 5.3: Plastically deforming area in Figure 5-9. Black area is regions at the crack tip where the equivalent plastic strain is greater than 0.1. Total area is all plastically deforming area (non-white). Plot letters correspond to letters in Figure 5-9.

<table>
<thead>
<tr>
<th></th>
<th>a</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>c</th>
<th>d</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st layer</td>
<td>8</td>
<td>28</td>
<td>18</td>
<td>8</td>
<td>14</td>
<td>15</td>
</tr>
<tr>
<td>all layers</td>
<td>12</td>
<td>86</td>
<td>174</td>
<td>169</td>
<td>227</td>
<td>116</td>
</tr>
</tbody>
</table>

plastic energy dissipation and maximum crack tip equivalent plastic strains are listed in Table 5.4 for four different constitutive models and two resin-rich region thicknesses per model. The energy dissipation values are given on a per unit resin area basis since there is nearly five times as much resin area in the thicker model. The maximum crack tip equivalent plastic strain is the highest strain value calculated by the model, which occurs at the crack tip. The maximum strains are unrealistically high in some cases for four main reasons. First, in the case of the perfectly plastic model, there almost certainly will be hardening in the material, which would increase the stresses and decrease the associated strains. Second, the hardening models may not be accurately capturing the stress-strain behavior in the constrained layer. Third, the actual surface constraining the crack tip is similar to a corrugated roof shape rather than a flat plate, which will decrease the overall strains. Finally, the high strains may be an indication that the material has already failed for this applied load. However, it was mentioned previously that constrained resin crack tip failure strains may be much higher than bulk failure strains.

The results in Table 5.4 reinforce the aforementioned observation that the crack tip strains in the thinner layer are higher and they also show that plastic energy dissipated is significantly higher in the thinner model than it is in the thicker model. The greater plastic energy dissipation is noteworthy given that there is less resin volume available for deformation and there is less resin actually deforming (shown in Table 5.3). As expected, the constitutive models with more hardening decrease the maximum crack tip strains and plastic energy dissipation. However, there is not a wide variance in values for $J$.

It is informative to compare the local $J$-integral that results from an applied global
Table 5.4: Maximum crack tip strains and plastic energy dissipation for four inelastic material constitutive models and two resin-rich region thicknesses. The four constitutive models are perfect plasticity (PP), deformation plasticity (DP), hardness plasticity 1 (H1), and hardness plasticity 2 (H2). "Thick." is the thickness of the resin-rich region in the model and \( J \) is the calculated J-integral for the model. “Max CT PEEQ” is the maximum crack tip equivalent plastic strain and “PED” is the plastic energy dissipated by the model, per unit resin area. The globally applied load is 200 N.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thick. (( \mu )m)</th>
<th>( J ) (J/m(^2))</th>
<th>Max CT PEEQ</th>
<th>PED (kJ/m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP</td>
<td>1</td>
<td>335.0</td>
<td>88.7</td>
<td>791</td>
</tr>
<tr>
<td>PP</td>
<td>15</td>
<td>299.7</td>
<td>3.8</td>
<td>153</td>
</tr>
<tr>
<td>DP</td>
<td>1</td>
<td>344.9</td>
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</tr>
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<td>306.7</td>
<td>1.1</td>
<td>65</td>
</tr>
<tr>
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<td>20.6</td>
<td>416</td>
</tr>
<tr>
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<td>1.6</td>
<td>89</td>
</tr>
<tr>
<td>H2</td>
<td>1</td>
<td>343.7</td>
<td>6.7</td>
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</tr>
<tr>
<td>H2</td>
<td>15</td>
<td>305.3</td>
<td>0.3</td>
<td>87</td>
</tr>
</tbody>
</table>

load and the associated global \( J \)-integral. Figure 5-10 compares these values over a wide range for a model with a 5 \( \mu \)m thick resin-rich layer and for three different types of plastic behavior: perfect plasticity, deformation plasticity, and Drucker-Prager plasticity. It is clear that the more plasticity that exists at the crack tip, the lower the local \( J \)-integral will be. Furthermore, the difference between global and local values is more pronounced at higher load levels. It is important to note that this plot is comparing calculated \( J \)-integrals for a particular stress state, as opposed to critical fracture parameters. However, if the neat resin toughness was used as the fracture criterion at the local level then the plot would show the neat resin toughness versus composite toughness. This is obviously incorrect because it indicates that the more plastic behavior a neat resin exhibits the more likely it is to have a toughness less than that of the composite. Figure 5-1 with experimental data shows that this is not the case. Thus, another fracture criterion is needed.
Figure 5-10: Computed global vs. local $J$-integral. The resin-rich layer in the local model is 5 μm thick.
5.4 Discussion

While these mechanics analyses provide insight into the stress state that leads to fracture, they do not specifically indicate when the resin will fracture. As mentioned previously, using a neat resin fracture toughness is inadequate. It is useful to examine the results of adhesive bond toughness experiments that vary the thickness of the bond [74]. Work of this sort exhibits the behavior shown in Figure 5-11, where the brittle resins exhibit no thickness effect while the ductile resins show a marked thickness effect. The explanation for this behavior is usually that the thicker bonds have the toughness of the bulk adhesive, but there is an increase in toughness with decreasing thickness as the constraint causes an increase in the amount of material that is plastically deforming. Further decreasing thicknesses have the effect of decreasing the amount of material that is plastically deforming, thereby decreasing the toughness. This results in a substantial loss in toughness for the bond when compared to the bulk adhesive.

Although the thickness of the bonds in Figure 5-11 are not on the scale of the resin-rich region in a composite, Chai has tested such bonds and discovered that the
toughness of the bond was almost identical to that of the composite with the same resin [41]. The results of his work are shown in Figure 5-12 with the tested resins listed near the data points. The plot shows how the constrained resin toughness is nearly the same as that of the composite for a range of materials with different toughnesses. Johnson and Mangalgiri have performed similar experiments on a different material and obtained the same result [81]. They maintain a bond thickness on the order of an interlaminar resin-rich region thickness by using a Teflon® insert in between the adherends that also acts as a starter crack, in an analogous manner to the composite DCB test.

The adhesive bond thickness studies have implications for the current work because they indicate that the constraint on the resin causes a change to occur in its critical fracture level that cannot be simply explained by comparing global and local $J$-integrals. The model developed in this research may still be used as a tool to determine the extent of plastic deformation at the crack tip, which is the largest contributing factor to the work of fracture, and it may also be used to determine the intensity of crack tip strains. The latter of these phenomena may explain why fracture occurs in a constrained layer with reduced plastic area. The local $J$-integral,
however, must be compared with a fracture criterion that accounts for the change in constraint on the material and the decrease in volume that is available to deform plastically. At the moment, this fracture criterion must be determined experimentally and the constrained resin adhesive bond test shows the most promise for providing the data for the criterion.

For a brittle material with a low critical SERR, the model indicates that there is little difference between the local and global $J$-integrals. The plastic zone size of the neat resin may be small enough that the composite actually enlarges the volume of plastically deforming material, which would increase the toughness of the constrained resin. The model describes this behavior in the difference between the global and local $J$-integral. The increase in available fracture area from the corrugated roof shape of the fracture surface may also explain the increase in toughness for a brittle resin.

If a relationship between the volume of plastically deformed material and fracture levels in constrained ductile resins can be determined, then the model will be of great use in determining fracture toughness. However, unless the fracture criterion is known \textit{a priori}, a prediction of toughness cannot be made from bulk constituent data alone. Data from adhesive bond tests using neat resins with thin layers on the order of the interply resin thickness could be used as an estimate of composite toughness and a fracture criterion for the model. The model could then be used to explain the differences between the neat resin and composite toughesses. This combined process of neat resin experiments, in the form of adhesive bond tests, and analysis would certainly be beneficial in the evaluation of candidate resin systems and would accelerate the insertion of new materials. The composite fracture values could be used in design studies before the actual composite is fabricated.

5.5 Conclusions

A finite element model has been created to examine the difference between the global and local behavior in a composite fracture specimen with the goal of predicting composite interlaminar delamination toughness from constituent properties. The model
indicates that an increase in the constraint on the crack tip increases the stress state at the crack tip and hence the $J$-integral as well. Furthermore, plasticity relieves stresses at the crack tip and therefore reduces the local $J$-integral and increases the difference between the global and local $J$-integral. The most important conclusion of the model is that the intensity of the strains in a plastically deforming crack tip are higher in a more highly constrained crack tip, but the total plastic volume is less. This may serve as the chief explanation as to why the toughness of a composite is less than its more ductile resin. The neat resin toughness is inadequate as a fracture criterion, but a constrained toughness value at an appropriate thickness level is more appropriate. Neat resin adhesive bond tests could be used as an estimate of composite toughness and the model could be used to elucidate the mechanisms that change the resin toughness in the composite.
Chapter 6

Fiber Bridging Model for Fatigue Delamination

Fiber bridging is a phenomenon that is commonly observed in fracture of nearly all types of fibrous composites. In general, bridging is a positive attribute of fracture in composites because more energy must be applied to the system in order to propagate the bridged crack. However, damage mechanisms in structural laminated polymer-matrix composite materials do not typically involve a great deal of fiber bridging. Off-axis ply cracking, or intralaminar fracture, is an example of a fracture mechanism that is commonly observed in PMCs that does not involve bridging. Interlaminar delaminations are another common damage mechanism that involve minimal fiber bridging because the delaminations form as a result of interlaminar shear stresses between plies of different orientations; fiber bridging does not typically occur between plies of different orientations.

Experiments that characterize PMC interlaminar fracture toughness are typically performed using unidirectional lay-ups [45]. Propagating cracks in between layers of the same orientation often develop fiber bridging; this behavior was observed in the delamination experiments described in Chapter 4. Thus, the crack propagation measurements are not representative of behavior in an actual structure. This is of concern for several reasons, but primarily because the measurements do not act as the basis for conservative design. Interlaminar crack propagation measurements in quasi-
static or fatigue experiments using unidirectional specimens that experience fiber bridging overestimate the strain energy release rate that is required to propagate a crack between plies of different orientations. Furthermore, the rate of change of the fatigue crack propagation rate in a specimen with fiber bridging as a function of the maximum SERR is very high compared to unbridged systems. Thus, any uncertainty in the SERR will lead to large uncertainties in the crack propagation rates.

Bridging issues in PMC delamination experiments have historically been dealt with in several ways. Initiation values in quasi-static interlaminar delamination toughness tests that are based on an artificial unbridged crack are considered to be preferred values for static design [45]. Thus, even though the experimental propagation measurements are used to determine the compliance of the system, the propagation values are effectively ignored. In fatigue, the delamination onset and fatigue threshold methods mentioned in Section 4.1 are recommended methods for determining the end of fatigue life. Once again, these techniques effectively ignore crack propagation data.

The need for a fiber bridging model that can separate the intrinsic response of an unbridged crack from the bridging contributions was motivated by the fatigue propagation experiments described in Chapter 4. There was a great deal of scatter in the data from the tests at different applied displacement ratios and this was shown to be due to the fiber bridging observed during the test. In addition, the bridging caused the exponents in the power-law relationships for the tests to be quite high, leading to the aforementioned uncertainties in crack propagation rate predictions. Rather than casting this data aside as unusable, a model was needed that could effectively remove the bridging contributions and determine the SERR in the crack tip that propagates the crack in the resin.

The fiber bridging model presented here is based on a cohesive zone law that describes the traction-separation behavior in the bridged region. The cohesive zone law is determined from quasi-static R-curves and allows one to calculate the bridging SERR for a given applied SERR. The difference between the applied and bridging contributions is the crack tip SERR in the resin. The model is used to calculate the
crack tip SERR in fatigue propagation for the data shown in Figure 4-17, examine the effect bridging has on fatigue crack propagation, and determine the trends of the constrained resin crack tip propagation behavior as a function of temperature. Calculated bridging SERR results are verified by measuring crack opening displacement profiles for a specimen using a scanning electron microscope at the same strain energy release rate applied during the fatigue tests.

Fatigue crack propagation in PMC structures does not necessarily immediately result in structural failure. Thus, the ultimate goal of the model is to allow crack propagation measurements to be used as part of a damage tolerant design philosophy that provides an accurate representation of delamination growth within structures.

6.1 Background

It was mentioned in Section 4.1 that numerous studies have determined that the fiber bridging observed during a DCB test is an artifact of the test specimen [37, 38]. Fiber bridging occurs when the crack switches from one fiber/matrix interface to another and leaves behind the unbroken fiber to bridge the crack, as shown in Figure 6-1. It is generally agreed that the bridging behavior in PMCs is a result of fiber “nesting”, which refers to the migration of fibers during the consolidation of the composite [81]. Figure 6-2 is a schematic depicting fiber migration and the wavy delamination plane that results.

Fiber bridging experiments have been performed extensively for polymer [81–84], metal [85], and ceramic matrix composites [82, 86–88]. The experiments typically observe fiber bridging in delaminations or in cracks propagating perpendicular to the fiber direction. A great deal of modelling of fiber bridging effects has also been conducted [85, 87–94]. The majority of the models tend to separate the applied strain energy release rate into a bridging component, $G_{br}$, and a tip component, $G_{tip}$ (i.e. the SERR in the matrix material at the crack tip). Many of the models that are specifically examining R-curve behavior assume that $G_{tip}$ is constant and is equal to the initiation toughness of the material (initial propagation from an unbridged
Figure 6-1: Schematic of fibers bridging a delamination in a composite and a micrograph of bridging in IM7/977-3. Grid on side of specimen is used for displacement measurements described in Section 6.4. Bridge in micrograph is a group of bonded fibers.

Figure 6-2: Schematic of nesting between layers in a composite.
crack). The bridging SERR is then determined in a variety of ways. Experimentally, the bridging SERR is merely \(\sqrt{G_{\text{tip}}}\) subtracted from the applied, or measured, strain energy release rate, \(\sqrt{G_{\text{appi}}}\). (Square roots of these quantities must be used because only stress intensity factors, \(K\), are additive and \(K \propto \sqrt{G}\).) However, the measured R-curve depends on the specimen geometry and thus, is not a material property. Therefore, models are often distinguished from one another in how they calculate \(G_{\text{br}}\) using material properties.

The most common method of determining the bridging SERR as a function of material properties is through the use of a cohesive zone law. This is a traction-separation law that defines the stresses at a particular location in a prescribed cohesive zone as a function of the opening displacement of the zone in that location. The law is independent of specimen dimensions and hence, is a material property. In the case of fiber bridging, the fibers act as the cohesive force that is preventing the crack from opening and the cohesive zone is the region that contains fibers bridging the crack. The SERR in the cohesive zone can be determined by integrating the cohesive stresses over the bridged region. However, the stresses can only be calculated if the displacements within the region are known. Since the displacements in the region are dependent on the applied load and the amount of bridging, the displacements can generally only be determined numerically or through some form of a self-consistent scheme.

The distinguishing characteristics among various models relate to the cohesive laws implemented and the methods for determining the displacements in the cohesive zone and the associated bridging energy. Suo et al. created solutions for delamination R-curves that are easy to use, but are only applicable to a few simple linear cohesive laws [91]. Jacobsen and Sørensen have implemented a more complicated nonlinear cohesive law, but their method requires numerical solutions [90]. An objective of this work is to create a model that can use a nonlinear cohesive law without a computationally intensive framework.

Most of the work dealing with bridging models is applied to quasi-static R-curves. Cox and co-workers have modeled bridging behavior in fatigue for cracks propagating
perpendicular to the fibers in ceramic matrix composites [94], but to the authors’ knowledge, there has been no modelling applied to bridged cracks for interlaminar delamination fatigue.

It has been suggested that the maximum cyclic applied SERR in a fatigue test with fiber bridging could be normalized by the R-curve SERR at the same crack length in the crack propagation rate plot [51]. This effectively accounts for the bridging contribution in the fatigue tests and gives an idea of the crack tip SERR during the test. Implementation of this method into the fatigue data in Figure 4-17 dramatically reduced the scatter and gave more consistent results for tests performed at the same temperature, indicating that fiber bridging was having a significant effect on the test results. The disadvantage of this technique is that it has little physical basis and relies on bridging data that is specimen dependent. Furthermore, the opening displacements in the quasi-static tests are likely to be greater than the opening displacements in the fatigue tests because of the higher loads required to propagate a quasi-static crack, which would result in different bridging behavior. This work aims to alleviate these problems by calculating \( G_{\text{tip}} \) from the applied and bridging SERRs and using this as the foundation for the crack propagation rate plots.

### 6.2 Description

The bridging model presented here uses the major elements mentioned above that are common to most bridging models. A schematic for the general implementation method is shown in Figure 6-3. \( G_{\text{tip}} \) is calculated as the difference between the applied and bridging contributions. \( G_{\text{app}} \) is determined for a given load and crack length from a standard DCB model that includes corrections for shear effects and rotations that occur near the crack tip. The uniqueness of the model lies in the calculation method for \( G_{\text{br}} \), which is performed using Timoshenko beam theory and the cohesive zone law in an iterative self-consistent manner. Once the strain energy release rates have been calculated, they are transformed into stress intensity factors, \( K \), using standard transformation equations for anisotropic materials, because stress intensity factors
can be linearly superimposed, whereas SERRs cannot. Finally, $K_{\text{tip}}$ is calculated as the difference between the applied stress intensity factor and the bridging stress intensity factor and then transformed into $G_{\text{tip}}$. The entire process has been coded in a Mathematica® program. (A sample code is provided in Appendix B.)

The equations used in the model for calculating the strain energy release rate in a DCB were developed by Bao et al. [77]. The relationship contains an elementary beam theory component modified by a correction for the length and thickness of the beam:

$$G_I = \frac{12(Pa)^2}{E' h^3} \left(1 + Y_I \lambda^{-\frac{1}{2}} \left(\frac{h}{a}\right)^{\frac{1}{2}}\right)^2.$$

(6.1)

$P$ is the applied load per unit width, $b$, of the beam, $a$ is the crack length, $h$ is the thickness of one of the beams (i.e. half the thickness of the entire specimen), and $E'$...
is the longitudinal material modulus, corrected for plane strain (defined below). $Y_I$ and $\lambda$ are:

$$Y_I(p) = 0.677 + 0.146(p - 1) - 0.0178(p - 1)^2 + 0.00242(p - 1)^3$$  \(6.2\)

$$\lambda = \frac{E_2'}{E_1'}$$  \(6.3\)

where $p$ is:

$$p = \frac{(E_1'E_2')^{\frac{1}{2}}}{2G_{12}} - (\nu_{12}'\nu_{21}')^{\frac{1}{2}}.$$  \(6.4\)

The elastic constants corrected for plane strain are:

$$E_1' = \frac{E_1}{(1 - \nu_{13}\nu_{31})}, \quad E_2' = \frac{E_2}{(1 - \nu_{23}\nu_{32})},$$

$$\nu_{12}' = \frac{(\nu_{12} + \nu_{13}\nu_{32})}{(1 - \nu_{13}\nu_{31})}, \quad \nu_{21}' = \frac{(\nu_{21} + \nu_{23}\nu_{31})}{(1 - \nu_{23}\nu_{32})}.$$  \(6.5\)

There is no change in the shear modulus, $G_{12}$. Bao also provides the relationship between strain energy release rate and stress intensity factor in terms of the same parameters:

$$G_I = \left(\frac{1 + p}{2E_1'E_2'}\right)^{\frac{1}{2}} \frac{K_I^2}{\lambda^4}. \hspace{1cm} 6.6$$

Given the dimensions, material properties, applied load, and crack length of a DCB specimen, one can calculate $G_{app}$ from the above equations. The calculation method for $G_{br}$ is more complicated and is shown schematically in Figure 6-4, which also includes a pictorial definition of the axis, displacements, crack lengths, and loads within the cohesive zone and for the entire system. The process begins with a known cohesive zone law (the process for determining of this law is described later), which describes the stress in the region, $\sigma$, as a function of opening displacement, $\delta$. Since the displacement is a function of position, the stress is also a function of position (i.e. $\sigma(\delta(x))$). An initial displacement profile is assumed, $\delta_1(x)$, which is calculated using Timoshenko beam theory. This theory accounts for shear deformations that are important in short beams, which come into play during the calculation of loads.
Wang et al. have developed a simple method to calculate Timoshenko beam displacements by using properties of Euler-Bernoulli beam theory [95]. To calculate Timoshenko beam displacements for a clamped-free cantilever beam, $\delta_T(x)$, one merely needs the Euler-Bernoulli beam displacements, $\delta_{EB}(x)$, and the Euler-Bernoulli beam moments, $M_{EB}(x)$:

$$
\delta_{EB}(x) = \frac{P}{6E_1^i I} \left(3a(x^2) - (x^3)\right)
$$

(6.7)

$$
M_{EB}(x) = -P(a - x)
$$

(6.8)

where $I$ is the standard second moment of inertia ($I = bh^3/12$). $\delta_T(x)$ for a clamped-free cantilever beam can then be defined:

$$
\delta_T(x) = \delta_{EB}(x) + \frac{1}{K_S G_{13} A_b} (M_{EB}(x) - M_{EB}(0))
$$

(6.9)

where $G_{13}$ is a shear modulus, $A_b$ is the cross-sectional beam area ($A_b = bh$), $M_{EB}(0)$ is the moment at $x=0$ (the crack tip), and $K_S$ is a Timoshenko beam shear coefficient:

$$
K_S = \frac{10(1 + \nu_{13})}{12 + 11\nu_{13}}.
$$

(6.10)

The initial displacement profile, $\delta_1(x)$, is calculated from $\delta_T(x)$ using the globally applied load, $P$. This allows for a distributed load within the cohesive zone, $q_1(x)$ (shown in Figure 6-4), that is acting to close the crack faces to be calculated using the cohesive zone law:

$$
q_1(x) = \sigma(\delta_1(x))b.
$$

(6.11)

The displacements within the cohesive zone are governed by the distributed load, in addition to the globally applied load. Thus, a second displacement profile within the bridged region, $\delta_{br}$, is calculated from the distributed load. Since $\delta_{br}$ is calculated using Equation 6.9, the Euler-Bernoulli moments and displacements must be calculated first through standard beam theory integrations and the appropriate boundary conditions.
Figure 6-4: Calculation method for determining bridging SERR.
for a clamped-free cantilever beam:

\[
\frac{d^2 M_{EB,br}}{dx^2} = -q_1(x) \tag{6.12}
\]

\[
E'_I \frac{d^4 \delta_{EB,br}}{dx^4} = q_1(x). \tag{6.13}
\]

The total beam displacement is assumed to be the difference between the applied displacement, \(\delta_{app}\), which is merely \(\delta_T(x)\) calculated with the globally applied load, and the displacement in the bridging region, \(\delta_{br}\). This calculation to determine \(\delta(x)\), which is \(\delta_2(x)\) in the current calculation process, is depicted graphically in Figure 6-4. The current displacement profile, \(\delta_2(x)\), is compared with the original displacement profile, \(\delta_1(x)\), by calculating displacements at \(x = \Delta a\), where \(\Delta a\) is the length of the cohesive zone. If the difference between the two values is less than a prescribed error value, in this case \(\delta_T(\Delta a)/1000\), then \(\delta_2(x)\) is accepted as the displacement profile of the beam. If the difference between the calculated displacement values is greater than the error value, then the process iterates and begins again by calculating a new distributed load, \(q_2(x)\), from the current displacement profile, \(\delta_2(x)\). The process continues until the error criterion is met, indicating that the distributed load profile and displacement profile are self-consistent.

Once the distributed load and displacement profiles, \(q(x)\) and \(\delta(x)\) respectively, have been calculated, an effective load is calculated so that the bridging energy can be determined from Equation 6.1. In addition, the location where the effective load acts must be determined. The effective load in the bridging region, \(P_{br}\), is calculated by integrating the distributed load over the cohesive zone length:

\[
P_{br} = \int_0^{\Delta a} q(x) \, dx. \tag{6.14}
\]

The location where the load acts, or the effective crack length, \(a_{br}\), is determined by calculating the “area” of the distributed load, \(A_q\), and the first moment of area, \(Q\):

\[
A_q = \int_0^{\Delta a} \int_0^{q(x)} dy \, dx \tag{6.15}
\]
Figure 6-5: Cohesive zone law used in bridging calculations.

\[ Q = \int_{0}^{\Delta a} \int_{0}^{q(x)} x \, dy \, dx. \]  

(6.16)

The centroid of the area, or the effective crack length \( a_{br} \), is merely \( Q \) divided by \( A_q \). \( G_{br} \) can finally be determined using Equation 6.1 and the calculated \( P_{br} \) and \( a_{br} \) values. Experimental validation of calculated \( G_{br} \) values is presented in Section 6.4.

Determining the appropriate traction law that best fits the experimental R-curves is a matter of trial and error, although the shape of the R-curve can give some indication of the shape of the cohesive zone law. Several different cohesive zone laws were used in an attempt to find the best fit to the R-curves from the quasi-static tests for the material tested in Chapter 4. The laws examined included linear softening, power law softening, a binding energy relationship [85], a combination of perfectly plastic and power law softening, and a combination of linear softening and power law softening. The traction law that provided the best fit to the data is a combination of linear softening and power law softening and is based on a similar law used by Jacobsen and Sørensen [90]. The cohesive zone law is shown in Figure 6-5. Four parameters define the shape of the curve: \( \sigma_m \), the maximum stress, \( \delta_c \), the maximum critical displacement, \( \delta_0 \), the displacement at the transition between linear and power law behavior (\( \sigma_0 \), the stress at the transition can be calculated from the other parameters), and \( m \), the shape of the curve in the power law region.

The two important parameters in the cohesive zone law are \( \sigma_m \) and \( \delta_c \) and hence,
determining the appropriate values for a set of data focuses on these two parameters. For an R-curve, $G_{tip}$ should remain equal to $G_{IC}$, so this is used as the primary measure of "goodness of fit". In addition, the opening of the crack at the end of the cohesive zone, $\delta(\Delta a)$, is equal to $\delta_c$ when the R-curve becomes flat, indicating that the cohesive zone has reached its ultimate length. Thus, the program is run for the load and crack length where the R-curve becomes flat using approximate traction law parameters and the two major parameters are adjusted until $G_{tip} = G_{IC}$ and $\delta_c = \delta(\Delta a)$. Then, the program is run using all loads and crack lengths and the other two parameters, $\delta_0$ and $m$, are adjusted to improve the fit such that $G_{tip}$ is close to $G_{IC}$ at all crack lengths.

The nonlinearity of the cohesive zone law made it impossible to use in the integrations steps in Mathematica®. Thus, each calculation involving integration of the distributed load in the cohesive zone, which is calculated from the cohesive zone law, was actually based on a polynomial that was fit to the distributed load for that load and crack length case. An eighth order polynomial was judged to capture accurately the distributed load behavior in all cases.

### 6.3 Results

The material properties used in these analyses are listed in Table 6.1. There are few published sources of material data for IM7/977-3 and even fewer that are a function of temperature [32, 96–98]. Many properties are within a reasonable range, with the exception of Daniel’s measured properties, which are higher than any other published source [96]. This is apparently due to a customized manufacturing process that created a higher fiber volume than is typical for IM7/977-3. The only other variation among other data sources was for the longitudinal modulus, $E_1$, which plays a significant role in calculating the SERR. Some simple tension tests were conducted at room temperature to verify the material behavior of the IM7/977-3 used in these experiments and to compare results with those published in the literature. $E_2$, $\nu_{12}$, and $G_{12}$ values were similar to those measured by Donaldson et al. [97, 98], but $E_1$ was
Table 6.1: IM7/977-3 material properties.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$E_1$ (GPa)</th>
<th>$E_2$ (GPa)</th>
<th>$\nu_{12}$</th>
<th>$G_{12}$ (GPa)</th>
<th>$\nu_{23}$</th>
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<td>0.37</td>
<td>6.1</td>
<td>0.55</td>
</tr>
<tr>
<td>66</td>
<td>149</td>
<td>9.5</td>
<td>0.37</td>
<td>5.9</td>
<td>0.55</td>
</tr>
<tr>
<td>107</td>
<td>149</td>
<td>9.3</td>
<td>0.37</td>
<td>5.7</td>
<td>0.55</td>
</tr>
<tr>
<td>149</td>
<td>149</td>
<td>9.1</td>
<td>0.37</td>
<td>5.5</td>
<td>0.55</td>
</tr>
</tbody>
</table>

measured as 149 GPa, compared to 162 [32] and 172 GPa [97, 98] from other sources. It is not clear what caused this difference in modulus, but $G_{app}$ values calculated using Equation 6.1 were close to experimentally measured values calculated using compliance methods when the 149 GPa modulus was used. Thus, this modulus was used in all calculations. The other properties listed in Table 6.1 and their changes with temperature were estimated from the values and relationships reported in the literature.

The fits of the theoretical R-curves calculated with the cohesive zone law parameters to the experimental quasi-static R-curves are shown in Figure 6-6 and the actual numbers used in the traction laws are listed in Table 6.2. In these plots, the theoretical $G_R$ values are merely the sum of $G_{IC}$ (i.e. $G_{tip}$) and $G_{br}$. Although there are four parameters that describe the cohesive zone law, it is clear that only two parameters, $\sigma_m$ and $\delta_c$, are dominant because the other two parameters, $\delta_0$ and $m$ show little variation. The fit is better at smaller crack lengths, but this is more important because it is where most of the fatigue propagation occurs. The steepness of the R-curves immediately after initiation necessitates the linear softening region of the cohesive zone law. It also indicates that the majority of the bridging contribution to the SERR is provided by short fiber lengths bridging the crack at small opening displacements that most likely provide a great deal of resistance in shear rather than tension.

Once the cohesive zone law parameters were determined, $G_{tip}$ calculations were performed using the fatigue data and the cohesive zone law parameters for each temperature. Calculations were performed for all data points at a particular temperature and displacement ratio using another slightly modified Mathematica® program specif-
Figure 6-6: Comparison of experimental and theoretical R-curves as a function of temperature.

Table 6.2: Cohesive zone law parameters.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$\sigma_m$ (MPa)</th>
<th>$\delta_c$ (mm)</th>
<th>$\delta_0/\delta_c$</th>
<th>$m$</th>
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<tbody>
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<td>0.68</td>
<td>0.04</td>
<td>0.12</td>
</tr>
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<td>107</td>
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<td>0.77</td>
<td>0.07</td>
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</table>
ically tailored to the fatigue calculations. The entire procedure required less than a
minute to complete using a standard personal computer with a 1.4 GHz processor.
The original data from the fatigue tests is plotted again in Figure 6-7a, but in this plot
the $G_{appl}$ values were calculated using Equation 6.1 and the experimentally measured
$P$ and $a$ values, whereas the data in Figure 4-17 were calculated using the modified
beam theory compliance method described in Section 4.2. The $G_{tip}$ values calculated
using the fiber bridging model are shown in Figure 6-7b. Lines in both plots are
power law fits of the data at a particular temperature.

There is clearly a significant difference between the $G_{appl}$ and $G_{tip}$ data. The
$G_{appl}$ data show a great deal of scatter, which is caused by the fiber bridging having
different effects on the tests at various applied displacement ratios but the same
temperature. Furthermore, the slopes of the fits for individual tests at a specific
applied displacement ratio are quite high, leading to the uncertainties mentioned
earlier. In contrast, there is less scatter in the $G_{tip}$ data from all tests at a particular
temperature and the slopes of these curves are much less than those from the $G_{appl}$
data. The $G_{tip}$ data allow one to see that there is a clear increasing shift in fatigue
crack propagation rate with increasing temperature for a given applied maximum
SERR. This shift is greater for smaller applied SERRs. These phenomena were not
clear from the unmodified experimental data.

$G_{tip}$ data for individual tests are shown in Figure 6-8 for 24 and 66 °C and Figure
6-9 for 107 and 149 °C. In comparison to the original experimental data (Figures 4-18
and 4-19) the slopes are significantly decreased and for most of the temperatures the
shift between test data at different applied displacement ratios is less.

Table 6.3 lists the power law exponents, $m$ (defined in the same manner as the
exponent in Equation 4.5), for $G_{appl}$ and $G_{tip}$ data at a given applied displacement
ratio and temperature. It is important to note that the $G_{appl} m$ values are different
than the experimental values presented in Chapter 4 because of the different methods
used to calculate the SERR. The bridging clearly has a significant effect on the power
law exponents because the $G_{tip} m$ values are significantly less than the $G_{appl} m$ values.
This reinforces the supposition that crack propagation should occur at faster rates
Figure 6-7: (a) Experimental crack propagation rates and (b) predicted tip crack propagation rates. Lines on the plot are power law fits through the data.
Figure 6-8: Predicted tip crack propagation rates for individual tests at 24 and 66 °C. Lines on the plot are power law fits through the data.
Figure 6-9: Predicted tip crack propagation rates for individual tests at 107 and 149 °C. Lines on the plot are power law fits through the data.
Table 6.3: $G_{\text{app}}$ and $G_{\text{tip}}$ power law exponents and fit measures from individual fatigue tests.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$\delta_{\max}/\delta_{IC}$</th>
<th>$G_{\text{app}}$ m $R^2$</th>
<th>$G_{\text{tip}}$ m $R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>0.8</td>
<td>18.3</td>
<td>0.94</td>
</tr>
<tr>
<td>24</td>
<td>0.6</td>
<td>18.4</td>
<td>0.82</td>
</tr>
<tr>
<td>66</td>
<td>0.8</td>
<td>24.3</td>
<td>0.96</td>
</tr>
<tr>
<td>66</td>
<td>0.6</td>
<td>14.2</td>
<td>0.79</td>
</tr>
<tr>
<td>66</td>
<td>0.5</td>
<td>31.0</td>
<td>0.91</td>
</tr>
<tr>
<td>66</td>
<td>0.4</td>
<td>24.6</td>
<td>0.91</td>
</tr>
<tr>
<td>107</td>
<td>0.6</td>
<td>20.4</td>
<td>0.91</td>
</tr>
<tr>
<td>107</td>
<td>0.5</td>
<td>12.6</td>
<td>0.86</td>
</tr>
<tr>
<td>107</td>
<td>0.4</td>
<td>7.3</td>
<td>0.99</td>
</tr>
<tr>
<td>149</td>
<td>0.8</td>
<td>14.3</td>
<td>0.62</td>
</tr>
<tr>
<td>149</td>
<td>0.4</td>
<td>14.9</td>
<td>0.95</td>
</tr>
<tr>
<td>149</td>
<td>0.3</td>
<td>10.9</td>
<td>0.88</td>
</tr>
</tbody>
</table>

for a given $G_{\max}$ in an unbridged crack. The lower values also reduce the uncertainty associated with using the crack propagation data in design.

It is also interesting to compare the quality of the curve fits for each temperature before and after applying the bridging model. Table 6.4 lists power law exponents for all data at a particular temperature and correlation coefficients, $R^2$. It is clear that the $m$ values for the $G_{\text{app}}$ data can hardly be considered valid because the fit is so poor at most temperatures (particularly 107 °C), whereas there are drastic improvements for the fits in the $G_{\text{tip}}$ data. This bolsters the argument that the different bridging levels for different applied displacement ratios cause the shift in crack propagation rate curves because crack propagation behavior should theoretically be independent of applied displacement ratio. The reduction of the scatter in the $G_{\text{tip}}$ data appears to indicate that unbridged crack propagation is indeed independent of applied displacement ratio. In addition, the reliability of the data increases with the narrower scatter, thereby improving the likelihood that the data can be used in a design environment.

Since the $G_{\text{tip}}$ values represent crack propagation that is only occurring in the resin, it is a useful exercise to compare the $G_{\text{tip}}$ crack propagation curves with neat resin crack propagation curves. Figure 6-10 presents such a comparison, with the
Table 6.4: $G_{appl}$ and $G_{tip}$ power law exponents and fit measures from all tests at each temperature.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$G_{appl}$ m</th>
<th>$R^2$</th>
<th>$G_{tip}$ m</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>8.1</td>
<td>0.75</td>
<td>8.3</td>
<td>0.86</td>
</tr>
<tr>
<td>66</td>
<td>6.0</td>
<td>0.42</td>
<td>3.5</td>
<td>0.78</td>
</tr>
<tr>
<td>107</td>
<td>3.0</td>
<td>0.16</td>
<td>2.4</td>
<td>0.83</td>
</tr>
<tr>
<td>149</td>
<td>2.7</td>
<td>0.58</td>
<td>1.2</td>
<td>0.70</td>
</tr>
</tbody>
</table>

neat resin data coming from Figure 4-14. It is not reasonable to compare numerical crack propagation rates for a particular SERR because the unconstrained neat resin will behave differently than the constrained resin in the composite. However, it is useful to compare the slopes of the curves. Neat resin $m$ values are 4.9 for the 24 °C test and 3.3 for the 149 °C test, compared with the associated 8.3 and 1.2 values for the same temperatures in the composite $G_{tip}$ data. Considering that $m$ values from the $G_{appl}$ test data were near 18 for individual tests at 24 °C and 12 for individual tests at 149 °C, the $G_{tip}$ $m$ values are much closer to the neat resin values.

6.4 SEM Crack Opening Displacement Measurements

While the results from the fiber bridging model are encouraging because they reduce the scatter in the experimental data, they suffer from the pitfall that they cannot be verified experimentally using the standard DCB experiments. Rather, a separate technique is required to probe the behavior within the cohesive zone. Paris and Poursartip have developed an experimental system and technique that allows one to measure delamination crack tip behavior by using a scanning electron microscope (SEM) [99, 100]. Crack opening displacement (COD) profiles can be directly related to strain energy release rates through an established relationship involving material properties. However, this SERR is a local value, $G_{IL}$. The local SERR should be equal to the global applied SERR, $G_{IG}$, unless there is fiber bridging or some other mechanism that changes the COD profile near the crack tip. In the case of fiber
Figure 6-10: Comparison of experimental neat resin and predicted tip crack propa-
gation rates at two temperatures.
bridging, the applied $G_{IC}$ (equivalent to $G_{appl}$) is merely the square root of the sum of the experimentally measured $\sqrt{G_{IL}}$ (equivalent to $\sqrt{G_{tip}}$) and $\sqrt{G_{br}}$. Thus, it is possible through this method to measure bridging and crack tip SERRs.

This technique was used to measure the bridging properties of a previously tested fatigue specimen as a way to obtain a preliminary validation of the fiber bridging model. Detailed descriptions of the technique are presented elsewhere [99, 100], but the important aspects are listed here. The SEM is modified so that it can accept a standard DCB specimen. Loading is applied by stepper motors and displacement is measured by cantilever beam displacement sensors. The specimen is mounted on a moveable stage and the stage moves within the chamber. Position is monitored by an LVDT. The side of the DCB specimen is polished, coated with carbon, and then coated with a grid of gold squares that are 12.7 $\mu$m apart. Both coatings are applied using vacuum evaporation.

When the specimen is in the SEM, a collection of pictures is taken along the crack length with the crack closed. Then, the specimen is opened to a specified load and another collection of pictures is taken along the crack length while the specimen is held at that load. The pictures are later “stitched” together to form one complete montage. An example of one such montage for an open crack is shown in Figure 6-11, which also shows the gold grid. Software is then used to determine the location of each square in an image with respect to the crack tip. The COD profile is determined by comparing the distance between two squares (one above the crack tip and one below) in the closed crack montage with the distance between the identical squares in the open crack montage. The COD for a particular distance from the crack tip, $r$, is merely the difference between the two distances.

An untested specimen was examined to verify that the technique gave reasonable results for IM7/977-3. The specimen contained only the initial artificial crack from the Teflon® strip and hence contained no bridging. The dimensions of the specimen were identical to those of the quasi-static and fatigue specimens, with the exception of crack length. The SEM loading jig cannot examine cracks longer than 50 mm, which was the standard initial crack length for the quasi-static and fatigue specimens. Thus,
the initial crack length in the specimens tested in the SEM was reduced to 20 mm by cutting with a diamond-bladed saw.

Experimental results for the untested specimen are shown in Figure 6-12. $G_{IC}$ values are calculated using Equation 6.1 and the individual data points are experimental values for a particular $G_{IC}$ level. Two loads were applied to the specimen and hence there are two sets of data corresponding to two applied strain energy release rates. The curve fits are calculated using the relationship between COD and $G_{IL}$:

$$COD = \frac{4}{\sqrt{\pi}} a^{3/2} \left( \frac{2a_{12} + a_{66}}{2a_{11}} + \sqrt{\frac{a_{22}}{a_{11}}} \right)^{1/4} \left(a_{11}a_{22}\right)^{1/4} \sqrt{\frac{G_{IL}}{\gamma}}$$

(6.17)

where

$$a_{11} = \frac{1}{E_1}, \ a_{22} = \frac{1}{E_2}, \ a_{12} = -\frac{\nu_{12}}{E_1}, \ a_{66} = \frac{1}{G_{12}}.$$  

(6.18)

In the case of the untested specimen, the $G_{IL}$ curve with a value equal to $G_{IC}$ fits the experimental data well, indicating that the technique and the material properties are acceptable for IM7/977-3. The deviation of the experimental data from the $G_{IL}$ curve at $r$ values above 400 $\mu$m is because Equation 6.17 is derived considering only the first term of the elastic stress singularity. Far from the crack tip, higher order terms become more important and this is not captured in the curve. However, the fit in the region near the crack tip is good and this is the most important component of determining $G_{IL}$ values.

The fatigue specimen examined in the SEM was originally tested at 66 °C and an applied displacement ratio of 0.5. Crack propagation during the test was nearly 7 mm. All of the 24 °C specimens were completely separated to examine the fracture
surfaces and were not available for testing. Although the initial crack length of the fatigue specimen was reduced to 20 mm by removing 30 mm of the specimen length, the load applied to the specimen in the SEM created a $G_{IG}$ that was equivalent to the applied $G_{max}$ during the fatigue test. Delamination initiation tests were performed as a verification measure on a servohydraulic testing machine using the standard quasi-static test method and a specimen with a 20 mm crack length. The results were within the experimental error of previous tests performed with the 50 mm crack specimens. In addition, the bridging law predicted the bridging behavior of the 20 mm crack specimen relatively well. Thus, the difference in crack length is not expected to affect the results of the SEM experiments.

Figure 6-13 shows the results of the experiment using the 66 °C fatigue specimen. The applied $G_{IG}$ was 83 J/m², but the curve that best fit the data was associated with a $G_{IL}$ of 40 J/m². A curve is plotted that shows the COD profile if there were no bridging (i.e. $G_{IG}=G_{IL}$), whereas another curve shows the COD profile associated with the $G_{IL}$ value of 30 J/m² predicted by the fiber bridging model. The predicted
profile is remarkably close to the experimental profile. While one specimen is not representative of the model at all conditions, the experimental results certainly give credence to the validity of the model.

### 6.5 Conclusions

Experimental data from fatigue crack propagation experiments indicated that fiber bridging had a significant effect on crack propagation rates, making the data virtually unusable for quantitative design purposes. The fiber bridging model presented here separated the strain energy release rate contributions to fiber bridging and to propagation of the crack in the resin. This eliminated the previously observed dependency of crack propagation rates on applied displacement ratio and allowed one to see the clear shifts in crack propagation behavior as a function of temperature. Furthermore, the slopes of the plots decreased and the scatter of the data was reduced, making it more likely that the data could be used in design. The important function of the
model is that it provides representative Mode I crack propagation rates for a crack in an actual structure, as opposed to a bridged test specimen. In the case of this test data, one could certainly observe that crack propagation rates were higher at higher temperatures for a given maximum applied SERR, which was not clear from the unmodified test data. The independent validation performed using the neat resin fatigue tests and the SEM COD measurements were unique verification measures that strengthen the credibility of the model.
Chapter 7

Discussion

Several conclusions have been made in this thesis relating to the experimental and modeling work presented in the previous chapters. It is necessary to explore how those conclusions are connected. The purpose of this chapter is to discuss how the thesis results are tied together by describing methodologies that allow the work to be used in future applications. In addition, the chapter discusses the limitations of the work presented in the thesis and an assessment of linking fracture mechanics properties across length scales.

7.1 Proposed Methodologies

There are two distinct audiences who would apply the results of this work: designers in an industrial setting who are making predictions of structural behavior and researchers in an academic or industrial research and development environment who are evaluating the behavior of a candidate material. Designers are more concerned with the application of the models, whereas the researchers are typically more interested in the damage mechanisms and how these affect a modeling framework. These two paradigms lead to different connections between the areas presented in this thesis.

A designer’s focus will be on the application of models to predict structural behavior. This person is interested in the outcomes of models, rather than how the models were developed or how the predictions are made. Thus, the nanoindentation
Desired Material Method of Determining Property Property

Constrained Matrix and/or Double Cantilever Beam Composite Delamination Quasi-static Experiment

Initiation Toughness

Composite Fatigue Delamination Onset Double Cantilever Beam Fatigue Experiment

Constrained Matrix Fatigue Propagation Fiber Bridging Model

Use properties in structural models

Figure 7-1: Designer methodology.

work, the quasi-static and fatigue fracture mechanisms, and the delamination initiation model will be of little interest to a designer. However, there is a great deal of other information that will be of use in a design environment.

Designers need basic fracture mechanics properties as inputs to structural models; delamination behavior is of concern in this case. Durability properties are also important because they help to set bounds on the critical environmental conditions. Constituent behavior is used by a designer in a framework that is examining the effect of properties at lower length scales on those at higher length scales, such as AIM-C. These requirements set up the methodology shown in Figure 7-1. The basic delamination initiation properties come from constrained matrix experiments or alternatively composite experiments. Delamination fatigue properties are determined from composite properties, but the fiber bridging model must be used to determine the propagation behavior in the constrained matrix between plies. These properties can then be used as fracture criteria in structural analyses, similar to the work of Krueger et al. [19].
Two components of this thesis’ objectives were to examine the possibility of creating models to predict the properties in the first two boxes. Chapter 5 showed that it was not possible to make a prediction of delamination initiation behavior without a constrained resin fracture criterion. Since the experimentally determined constrained resin toughness is equivalent to the composite toughness, then the model is not necessary to make the prediction. In the case of the composite delamination onset behavior, the data in Chapter 4 indicated that it was not possible to predict composite fracture fatigue behavior from constituent properties using viscoelasticity principles for IM7/977-3. Thus, this data must also be determined experimentally. The fatigue crack propagation behavior was an opposite case where the experimental data could not be used until it had been analyzed using the bridging model.

The designer methodology shown in Figure 7-1 is quite similar to methodologies being developed today, but it has two main improvements. First, the fiber bridging model adds a tool that did not previously exist and therefore expands the type of predictions that can be made. Fatigue crack propagation rates are a required input for structural models predicting fatigue. The model can be used to provide Mode I crack propagation rates. Second, the methodology has the advantage of explicitly incorporating constrained matrix properties, which allows for sensitivity studies of the constituent fracture properties on the composite fracture properties to be performed.

The researcher has different motivations than the designer and hence, the researcher’s methodology is different, as shown in Figure 7-2. This person is interested in the assumptions that are used in models, mechanisms involved in quasi-static and fatigue fracture, effects of temperature on fracture behavior, inelastic deformation and associated energy during delamination in the matrix material near the crack tip, and the ability to create new models or improve existing models from observed behavior. Materials insertion for a researcher could certainly involve insertion of an existing material into a new product, but it is also likely that a researcher would be evaluating a candidate material with an unknown set of properties. The latter case would necessitate a more in-depth examination of material properties and damage mechanisms.
Objective

Compare neat and in situ matrix modulus and hardness

Understand neat matrix and composite quasi-static and fatigue fracture mechanisms

Compare shifts in neat matrix and composite quasi-static and fatigue fracture behavior as a function of temperature

Examine inelastic matrix deformation at crack tip in composite DCB specimen

Assess potential for future modeling improvements

Method of Accomplishing Objective

Nanoindentation Experiment

Compact Tension and Double Cantilever Beam Quasi-static and Fatigue Experiments

Compact Tension and Double Cantilever Beam Quasi-static and Fatigue Experiments at Temperature

Delamination Initiation Model

Figure 7-2: Researcher methodology.
Since researchers are typically involved in model development, the nanoindentation experiments will certainly be of interest as a way of gauging the assumption that neat matrix properties used in models are the same as unconstrained *in situ* properties. Depending on the results of these experiments, a researcher may decide to modify matrix properties in the models or proceed with the same initial values while understanding the uncertainty associated with this decision.

The neat resin and composite quasi-static and fatigue fracture experiments are essential to a researcher’s understanding of a material’s behavior for several reasons. First, an understanding of the fracture mechanisms in these processes is key to the application of fracture theories to structural predictions and also to the development of other models. Second, the shifts in behavior with temperature offer the potential to use viscoelasticity principles to predict fatigue properties. Although these techniques were not applicable to IM7/977-3, this may not be the case for all composite materials. Furthermore, the shifts in behavior with temperature are an important method of determining the limiting durability environments. The experiments in this study proved that the quasi-static and fatigue delamination shifts with temperature were not the same and hence, consideration of both quasi-static and fatigue behavior at various temperatures is important in any durability study.

The delamination initiation model will primarily be of use to researchers’ evaluating a new material. When a constrained matrix fracture criterion and the inelastic matrix behavior are known, the model can be used to determine the amount of inelastic deformation in the matrix prior to fracture. This is important for two reasons. First, irreversible damage in the matrix material prior to fracture is the primary mechanism in determining the fracture toughness of the composite. Thus, an understanding of this behavior in a Mode I DCB model could create a foundation for knowledge of the delamination behavior in structural mixed-mode deformation as well. The model will be particularly effective as a comparative tool since known material systems will already have been run in the model and their constrained matrix deformation characteristics will be understood and available for comparison. The second reason the amount of inelastic matrix deformation prior to fracture is important is
that this information can be used in conjunction with similar information from other material systems to develop a fracture criterion that relates the amount of plastically deforming area in the model to matrix critical fracture toughness. This relationship will be a key factor for the model to ever have true predictive capabilities.

The final step in the researcher methodology is essential to further development and improvement of the designer, researcher, and AIM-C methodologies. After completing the researcher methodology, there is a need to assess the potential for future modeling improvements. These improvements are a fundamental component of the AIM-C process because they expand an engineer’s predictive capabilities, thereby potentially reducing the number of experiments required to characterize material properties.

7.2 Limitations

As with any experiment or model, the work presented in this thesis contains some limitations. It is important to discuss these issues because they set parameters on how the experiments or models are applied in the future. Extrapolation of results to a scenario for which they were not developed carries a great deal of uncertainty and this does not instill confidence in the overall AIM-C methodology.

The primary limitation for the nanoindentation test technique is the size of the polymer pocket available in the composite material. The strength of the technique is that the numerical parameters can be calculated to determine whether any test is acceptable, but the modeling work indicated that only relatively large polymer pockets would produce reasonable results. In cases where the polymer pockets are not of adequate size in a composite material, artificial pockets should be created in order apply the test technique.

Another limitation of the nanoindentation experiments is the uncertainty related to the relationship between measurements of material properties using experiments such as tensile tests and measurements of material properties using indentation techniques. For example, the experiments in this thesis determined that there is a dif-
ference between neat and unconstrained in situ matrix properties, but the question remains as to whether the measured unconstrained in situ properties could be used in a model predicting composite behavior. This is a larger question that the nanindentation research community is working to address.

The quasi-static and fatigue fracture experiments do not have any intrinsic limitations because their purpose is to measure very specific properties such as Mode I delamination initiation toughness. The limitations are much more related to how the properties are applied in design. For example, the quasi-static delamination experiment uses a Teflon® insert and a unidirectional layup, neither of which are found in aerospace structures. However, this is the case with many of the strength and fracture mechanics properties measured in a laboratory and then applied to a structural prediction. The uncertainties associated with applying these measured properties to structures analyses must be considered.

The delamination initiation model is limited by the fact that it has only been applied to Mode I delamination initiation. In addition, only one material system has been examined and the results have not been experimentally verified. The extraordinary difficulty of validating the inelastic deformation of the matrix near the crack tip in a DCB specimen makes it unlikely that this limitation will be removed. However, the model could be applied to more material systems, thereby expanding the knowledge base and allowing for comparisons to be made between materials regarding inelastic matrix deformation prior to fracture. In addition, the model could be applied to Mode II and mixed-mode initiation, but the finite element model could not remain a symmetric model due to the asymmetry of these loading types.

Limitations of the fiber bridging model are also related to its application of only Mode I behavior and one material system. Furthermore, experimental validation was only performed on one specimen, so the lack of adequate experimental validation data could also be considered a limitation. Application of the model to other composite materials that experience bridging in Mode I crack propagation would help to examine the generality of the model.
7.3 Linking Fracture Properties Across Lengthscales

There was discussion in Chapter 2 regarding the importance of using mechanism-based models to bridge lengthscales, in conjunction with experimental observations. It is now appropriate to assess the feasibility of using this methodology for fracture properties at the constituent and lamina lengthscales, in light of the thesis results presented in the previous chapters.

The specific niche of this thesis falls at the lower end of the lengthscale spectrum and is focused on the mechanism fracture. The comparison of the fracture mechanisms between the constituent and composite lamina levels indicated that there were significant differences between the fracture behavior. Constrained matrix behavior was found to be the key mechanism in determining delamination initiation and fatigue propagation behavior. Neat matrix behavior, by contrast, was different than unconstrained in situ matrix behavior for elastic properties, shifts in quasi-static toughness with temperature, and even fracture mechanisms in the quasi-static tests at the highest temperature. All of these observations point to the conclusion that it is not possible to use unmodified neat matrix fracture properties to predict directly numerical values of composite delamination toughness. However, the mechanisms that occur in the neat matrix have a direct influence on the constrained matrix behavior and therefore the composite behavior as well. Thus, the process of using mechanism-based models that incorporate the constrained matrix behavior (delamination initiation and fiber bridging models) was a direct result of the experiments performed in conjunction with the modeling.

The observations of the links between these two lengthscales may have implications for links between other lengthscales as well. For example, structural models that incorporate lamina level fracture properties such as $G_{IC}$ and $G_{IIC}$ that are measured using unidirectional materials may require a modification to predict initiation and propagation of delaminations. Experimental observation will be the only method of determining whether the lamina level behavior is applicable in structural behavior and any potential modifications should be based on the observed structural mechanisms.
Empirically modified design allowables are only applicable to the systems on which the experiments were performed and because they do not examine the mechanisms responsible for the change in behavior at different lengthscales, the specific values will not be applicable in a more general design framework.

This thesis is representative of the need to combine experimental work with modeling that bridges lengthscales. These practices accelerate materials insertion by contributing to the overall designer knowledge base, thereby reducing the need for testing in the future and improving predictive capabilities. Some of the existing modeling frameworks mentioned in Section 2.1 that do not combine experimental verification with steps between lengthscales may suffer from the lack of knowledge of the mechanisms involved that can significantly change behavior between lengthscales.
Chapter 8

Conclusions

The objective of this thesis was to examine the issues associated with linking constituent and composite fracture properties. This was explored through a combination of experimental and analytical work that examined the objective from a variety of perspectives. A number of conclusions were formulated as a result of the work with the majority relating to the specific experimental and analytical efforts and several others relating to the overall methodology of implementing fracture properties in a range of lengthscales.

The conclusions are as follows:

- Nanoindentation experiments indicated that unconstrained in situ matrix properties are not necessarily the same as the neat properties. Modulus differences for an epoxy resin were up to 30%. This is most likely due to differences in processing techniques. These results may have a significant effect on models that use neat matrix properties. Comparisons of neat and in situ nanoindentation load-displacement curves using the exponent from a power-law curve fit was necessary to determine which in situ tests were not affected by the constraint of the fibers and were therefore acceptable tests.

- A finite element analysis of the nanoindentation tests showed that the distance across a resin pocket in a composite needs to be fifty times larger than the maximum penetration depth in order to obtain acceptable experimental results.
that were not mechanically affected by the presence of the fiber (assuming the resin has similar properties to the material modelled in the analysis). Some materials that do not have these pockets naturally may require an artificially created polymer pocket in order to conduct the nanoindentation experiments.

- Quasi-static fracture experiments performed on a composite and its neat resin at a range of rates and temperatures indicated that the mechanisms in the neat resin fracture behavior could explain the composite fracture behavior, but the shifts in behavior as a function of temperature were different for the two materials (composite toughness increased, whereas neat resin toughness decreased at elevated temperature). Thus, viscoelasticity principles could not be used to predict durability behavior.

- The mechanisms observed in the neat resin and composite quasi-static experiments were also important in explaining the results of the fatigue tests. Fatigue behavior was heavily dependent on temperature in both materials. Higher temperatures significantly decreased the number of cycles to delamination onset for a given applied maximum SERR. Fatigue crack propagation rates in the neat resin were higher at higher temperatures for a given applied maximum SERR and the same trend appeared to be true for the composite, but the fiber bridging observed in the test created a great deal of scatter in the results. Crack propagation rates in the composite were dependent on the applied displacement ratio, a clear indication that bridging was affecting the results.

- A global-local finite element model created to predict Mode I delamination initiation toughness from neat matrix properties showed that in order for a prediction to be made, a constrained matrix fracture criterion was needed. This fracture criterion could come from an adhesive bond test using the matrix as the adhesive in a layer whose thickness is on the order of an interply layer. Since this value has been shown to be quite similar to the interlaminar fracture toughness of the composite, the model’s value lies in being able to analyze the difference between inelastic neat matrix and constrained matrix behavior and
how this contributes to composite toughness. Indeed, the plastically deformed volume near the crack tip may serve as a potential fracture criterion in the future.

- A fiber bridging fatigue model was shown to account effectively for bridging effects in composite fatigue delamination propagation. The scatter that was previously observed in fatigue data was significantly reduced once the data had been analyzed using the model. Trends with respect to temperature could then clearly be seen (crack propagation rates increasing with temperature for a given maximum applied SERR) and the power-law exponents were much closer to neat resin values. The implication of this model being used on other fatigue data is that fatigue crack propagation data no longer need to be ignored for reasons of uncertainty. The model is a further example of the need to observe constituent behavior within the composite in order to make predictions of composite behavior.

- Verification of the fiber bridging model was performed using SEM crack opening displacement measurements. The COD profile in a specimen that had been used in a fatigue test and contained fiber bridging was close to the profile predicted by the model.

- Cumulatively, the work presented in this thesis represents a set of tools for design and materials engineers to evaluate and implement Mode I delamination properties of composite materials and thereby accelerate materials insertion. The choice of which tools are used and how they are used together will depend on the paradigm of the person using the tools. Two methodologies were presented to suit the two audiences that are most likely to utilize these results: the designer and the researcher. Designers are more concerned with the application of the models, whereas the researchers are typically more interested in the damage mechanisms and how those affect a modeling framework. The methodologies connect the work presented in the thesis to suit the needs of the two audiences.
• The final, and perhaps most important, conclusion of the thesis was that the work reinforced the need to combine mechanism-based models with experimental observation when linking behavior at two different lengthscales. This observation has been made in the past, but the thesis is a specific example that highlights the importance of incorporating this design methodology into other models that link fracture properties at different lengthscales.

Implementation of the combined technique of mechanism-based models and experimental observations with the proposed methodologies would accelerate materials insertion by reducing the number of tests required to characterize material behavior and reducing the risk of “surprises” occurring during testing and analysis at higher lengthscales. The primary reason for this is that this practice entails a fundamental understanding of the mechanisms involved in composite damage, which leads to a stronger prediction capability of composite failure and a reduced reliance on empirical results.

8.1 Contributions

There are several pieces of original work in this thesis that contribute to the advancement of the field of composite mechanics and the accelerated insertion of materials effort.

• The nanoindentation tests were the first study to compare neat and in situ polymer deformation behavior in PMCs using identical test techniques. This was also the first experimental evidence that the mechanical properties of the neat polymer and the unconstrained cured polymer within the composite were different. The finite element analysis will help to guide future testing in this area.

• The quasi-static neat resin and composite tests were unique in their comparison of the fracture behavior between the two materials and the combined effects of temperature and loading rate. There was no indication from previous research
that the shifts with temperature would be different. Furthermore, the literature contains research on strength properties of neat resin and composite materials that exhibit the same viscoelastic shift characteristics, but no work had been presented on fracture mechanics properties. The finding that fracture mechanics properties do not follow viscoelastic-type behavior, at least in the material system examined here, is a contribution to the field.

- There are several papers available examining fatigue delamination behavior, but few, if any, have studied this behavior at elevated temperatures. The dependence of delamination onset on temperature is significant because the quasi-static tests indicated that the critical design case was at room temperature, whereas the delamination onset tests showed that the highest temperature was the most important case from a durability standpoint. The crack propagation experiments were important for similar reasons. The dependence of the crack propagation tests on applied displacement level in the presence of bridging was also an important result. These findings are of consequence when choosing the crucial durability experiments to perform.

- Micromechanical models of the crack tip in a DCB specimen incorporating discrete resin and fiber properties have been created in the past, but the model presented here was the first to specifically examine the contribution of inelastic matrix deformation at the crack tip and the differences this creates between global and local SERRs. The analysis was also unique in its discovery that the intensity of the strains in a plastically deforming crack tip are higher in a more highly constrained crack tip, but the total plastic volume is less, which may serve as the chief explanation as to why the toughness of a composite is less than its more ductile resin. The model also explicitly motivates the use of a constrained matrix fracture criterion and the potential association of plastic volume with this criterion.

- The fiber bridging model is unique in its examination of fatigue properties and the experimental verification of behavior through neat resin fatigue tests
and the SEM crack opening displacement experiments. Most bridging models only study quasi-static crack propagation, but the analysis of fatigue crack propagation data presented here using the bridging model was of significant value because it explained the test dependence on applied displacement level and showed clear trends with temperature. The experimental verification gives confidence that the model makes accurate predictions.

- Finally, the overall approach applied in the thesis and the resulting methodologies are contributions to the larger effort to accelerate materials insertion. This work has emphasized the importance of a mechanism-based modeling approach combined with experimental observations as a verification and improved understanding measure and the thesis acts as an example of why these techniques are important.

8.2 Recommendations

There are many opportunities available to improve upon the work presented in this thesis. The biggest recommendation for future work in nearly all areas is the application of the experimental and analytical techniques to other material systems. This will help to clarify the limitations of the methods and delineate the important mechanisms associated with each class of materials. In addition, more data points always help to improve the statistical reliability of experimental outcomes. A final general recommendation is the incorporation of Mode II and mixed-mode considerations into experimental and analytical investigations. These opportunities are steps that should be taken to expand the applicability of fracture mechanics to design.

Some of the other recommendations for future work include:

- An assessment of the application of measured properties using nanoindentation to models that use bulk properties would help to expand the application of the test method. It would also be useful to develop a reliable technique to create artificial polymer pockets within a composite so that the size of a natural
polymer pocket would no longer be a limiting case in nanoindentation tests.

- An increase in the temperature range examined in the quasi-static and fatigue experiments, particularly an inclusion of temperatures below 0 °C, would increase the applicability of the results to a design environment.

- The delamination initiation model would benefit from an improved fracture criterion, perhaps one related to the amount of plastic volume near the crack tip. A three-dimensional model may be necessary to capture this behavior adequately.

- More experimental verification of the bridging model would help to strengthen the validity of the model. In addition, the technique used to determine the cohesive law parameters could potentially be improved and with this the capability of the bridging law to predict quasi-static bridging behavior in other geometric configurations could be explored.

- The designer and researcher methodologies should be applied numerous times as a means of improving the processes. This will have the added benefit of examining the practice of using mechanism-based models and experimental observation to link lengthscales, thereby continuously advancing the accelerated insertion of materials effort.
References


[33] 2002. Personal communication with Pete George, the Boeing Company.


2003. Personal communication with Akira Kuraishi, Stanford University.


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Appendix A

ABAQUS® Files

This appendix contains the ABAQUS® files used in the finite element analyses of the nanoindentation and delamination initiation models.

A.1 Nanoindentation Model

The input file below is a typical model used in the nanoindentation studies. The dimension \( x_2 \) (from Figure 3-21) is 3.0 mm and \( x_1 \) is 0.1 mm. The dimensions are meant to be 300 times and 10 times the maximum expected displacement of 0.01 mm, respectively, for the applied load of 500 mN. The mesh density values of 50 and 25 refer to the number of elements along the edge in the region \( (x_2-x_1) \) and \( x_1 \), respectively. The mesh was generated using a MATLAB® file that calculated the location of each node based on the desired mesh density and the number of elements along each edge. The actual coordinates of every node are not included for brevity. In this model, the outer edge is constrained.

**

HEADING

9773-500-3000; 977-3; 50,25 x 50,25; 0.1,3 x 0.1,3; 500 mN; constrained
*

*-----Define Nodes-----
*

NODE

1, 0.00000e+000, 0.00000e+000
2, 2.00000e-006, 0.00000e+000
3, 4.00000e-006, 0.00000e+000
4, 6.00000e-006, 0.00000e+000
5, 8.00000e-006, 0.00000e+000

5772, 1.58956e-003, -3.00000e-003
5773, 1.86138e-003, -3.00000e-003
5774, 2.18117e-003, -3.00000e-003
5775, 2.55739e-003, -3.00000e-003
5776, 3.00000e-003, -3.00000e-003
5777, 4.70735e-004, 1.68547e-004

* *----Define Elements---- *

ELEMENT, TYPE=CAX4R
1, 77, 78, 2, 1

ELGEN, ELSET=POLYMER
1, 75, 1, 1, 75, 76, 75

* *----Define Useful Node and Element Sets---- *

NSET, NSET=TIP_REF
5777
NSET, NSET=TOP, GENERATE
5701, 5776, 1
NSET, NSET=LEFT, GENERATE
1, 5701, 76
NSET, NSET=RIGHT, GENERATE
76, 5776, 76

ELSET, ELSET=TOP, GENERATE
1, 75, 1
ELSET, ELSET=LEFTTOP, GENERATE
1, 5551, 75

* *----Element Properties---- *

SOLID SECTION, ELSET=POLYMER, MATERIAL=POLYMER

* MATERIAL, NAME=POLYMER
ELASTIC
3.72e9, 0.369

PLASTIC
9.14e+07, 0
9.50e+07, 0.001258433
1.00e+08, 0.003100407
1.05e+08, 0.005041693
1.10e+08, 0.007095927
1.15e+08, 0.009277852
1.20e+08, 0.01160335
1.25e+08, 0.014089477
1.30e+08, 0.016754493
1.35e+08, 0.019617896
1.40e+08, 0.022700451
1.45e+08, 0.026024224
1.50e+08, 0.02961261
*
*-----Define Boundary Conditions----
*
BOUNDARY
LEFT, 1,1
BOTTOM, 2,2
RIGHT, 1,1
RIGHT, 2,2
TIP_REF, 1,1
TIP_REF, 6,6
*
*-----Define Surfaces and Interactions----
*
SURFACE, NAME=POLYM.URIF
TOP, S3
LEFTTOP, S4
SURFACE, TYPE=SEGMENTS, NAME=TIP.URIF
START, 4.70735e-004, 1.68547e-004
LINE, 0, 0
RIGID BODY, ANALYTICAL SURFACE=TIP.URIF, REF NODE=TIP_REF
SURFACE INTERACTION, NAME=TIP_INTER
1.0
CONTACT PAIR, INTERACTION=TIP_INTER
POLYM.URIF, TIP.URIF
*
*-----Step 1: Loading----
*
STEP, INC=150, NLGEOM
STATIC
0.01, 1.0, 1.0e-12, 0.01
*
*Applied Load
*
CLOAD
TIP_REF, 2, -500e-3
*
*Outputs
*
RESTART, WRITE, OVERLAY, FREQUENCY=5
OUTPUT, FIELD
NODE OUTPUT
U, RF, CF
ELEMENT OUTPUT
S, E, PE, PEEQ, PEMAG
CONTACT OUTPUT
CSTRESS, CDISP
*
OUTPUT, HISTORY
NODE OUTPUT, NSET=TIP_REF
U2, CF2
*
NODE PRINT, NSET=TIP_REF, SUMMARY=NO
U2, CF2
END STEP
*
*----Step 2: Unloading----
*
STEP, INC=100, NLGEOM
STATIC
0.01, 1.0, 1.0e-12, 0.01
*
*Applied Load
*
CLOAD
TIP_REF, 2, 0
*
END STEP
*
*----End----
A.2 Global Delamination Model

This input file is the global model in the delamination initiation model. Once again, the node and element definitions are removed for brevity. The applied global load is 8000 N/m, which is 200 N for a 25 mm wide specimen.

**
Heading
2D composite global model, IM7/977-3, P=8E3 N/m
* Job name: 2DG-8E3
*
*-------Model Definition-------
*
*-----Generate Nodes-----
*
Node
.
.
.
*
*-----Generate Elements-----
*
*Local Master Element
Element, Type=CPE8
1, 3, 1, 81, 83, 2, 41, 82, 43
.
.
.
*
*-----Define Orientation of Material-----
*
Orientation, name=Ori
1, 0, 0, 0, 1, 0.
1, 0
*
*-----Define Section-----
*
Solid Section, elset=allels, orientation=Ori, material=CFRP
*
*-----Define useful node sets-----
*
Nset, nset=load node
3229
Nset, nset=mid_fixed_bc_nodes, generate    
41, 3201, 40
Nset, nset=fixed_bc_nodes
    tip, mid_fixed_bc_nodes  
Nset, nset=crack_tip
    1
*  
Nset, nset=sub_nodes, generate
    561, 593, 1
    601, 633, 1
    641, 673, 1
    681, 713, 2
    721, 753, 1
*  
*VCCT nodes
Nset, nset=vcct_force_tip, generate
    1, 33, 1
Nset, nset=vcct_disp
    73, 113
Nset, nset=vcct_force
    vcct_force_tip, 41, 81
*  
*  
*----Define Material----
*  
Material, name=CFRP
Elastic, type=engineering constants
    1.68e+11, 9.55e+09, 9.55e+09, 0.263, 0.263, 0.397, 4.35e+09, 4.35e+09
    3.42e+09
*  
*  
*------Step Definition------
*  
*----Define Step----
*  
Step, name="Apply Load"
Apply load
Static
    1., 1., 1e-05, 1.
*  
*----Define Boundary Conditions----
*  
Boundary
fixed_bc_nodes, YSYM

*-----Define Load-----
*
Cload
load_node, 2, 8.0e+03
*
*-----Output Requests-----
*
Restart, write, frequency=1
*
*
Output, field
Node Output
U
Element Output
S, E
*
Node Print, Nset=vcct.disp, Summary=No
U2
Node Print, Nset=vcct.force, Summary=No
RF2
*
Contour Integral, Contours=20, Symm
crack_tip, 1., 0
*
Node File, Nset=sub.nodes
U
File Format, Zero Increment
End Step
A.3 Local Delamination Model

This input file is the local model in the delamination initiation model. As in the other models, the node and element definitions are removed for brevity. The inelastic material definition for the resin in this model is perfect plasticity.

**
Heading
2D local, perfectly plastic, P=8E3
* Job name: 2DL-PP-1
* Global model is 2DG-8E3
*
*-------Model Definition--------
*
*-----Generate Nodes-----
*
Node
.
.
.
*
*-----Generate Elements-----
*
*Crack Tip Region Master Element
Element, Type=CPE8
1, 3, 1, 81, 83, 2, 41, 82, 43
.
.
.
*
*-----Define fiber orientation-----
*
Orientation, name=ori
1., 0., 0., 0., 1., 0.
1, 0.
*
*
**---Define Sections---**

* Resin Section
  Solid Section, elset=res.els, material=resin
* Fiber Section
  Solid Section, elset=fib.els, orientation=ori, material=fiber
* Composite Section
  Solid Section, elset=comp.els, orientation=ori, material=composite

* Define useful node sets

* Boundary Conditions and Crack Tip
  Nset, nset=fixed.bc.nodes, generate
  1, 2601, 40

  Nset, nset=crack.tip
  1

* Submodel outer regions
  Nset, nset=top.nodes, generate
  258665, 260665, 40
  258649, 258665, 1
  258649, 260649, 40

  Nset, nset=perim
  out.left, out.right, top.nodes

*------Define Materials------

* Material, name=resin
  Elastic
  3.8e9, 0.369
  Plastic
  91.4e6, 0.0

* Material, name=fiber
  Elastic, type=Engineering Constants
  277.0e+9, 17.2e+9, 17.2e+9, 0.2, 0.2, 0.25, 18.6e+9, 18.6e+9
  4.83e+9

* Material, name=composite
  Elastic, type=Engineering Constants
  1.68e+11, 9.55e+9, 9.55e+9, 0.263, 0.263, 0.397, 4.35e+09, 4.35e+09
3.42e+09
*
*
*----Call Submodel----
* Submodel perim *
* *------Step Definition------ *
* *----Define Step---- *
Step, name="Apply Load"
Apply load Static
1., 1., 1e-05, 1.
*
*----Define Boundary Conditions---- *
* *Fixed nodes on bottom Boundary fixed.bc.nodes, YSYM Boundary, Submodel, Step=1 perim, 1, 2 *
*
*----Output Requests---- *
* Restart, write, frequency=1 *
* Output, field Node Output U Element Output S, E, PE, PEEQ, PEMAG, EE *
* Contour Integral, Contours=36, Symm crack.tip, 1., 0 End Step
Appendix B

Mathematica® File

This appendix contains a Mathematica® file used in the bridging analysis. The file was used to analyze data from quasi-static experiments and determine the cohesive law parameters. Another file was used to calculate $G_{tip}$ values from fatigue data, which was nearly identical to this file, except that it calculated $da/dN$ instead of $G_R$. The inputs to this file are three additional files. The first file contains loads and crack lengths for the specified test at a particular temperature (in the case of the fatigue tests the file also contains the number of cycles for the load and crack length). The second file contains the material properties for the specified temperature and the third file contains the cohesive zone properties for the temperature.
Import file containing crack lengths and loads

\[ \text{In[1]} := \text{SetDirectory["C:\Work\Research\Bridging"]}; \]

\[ \text{Loads} = \text{Import["149static.dat"]}; \]

\[ n_{\text{max}} = 18; \]

\[ G_{\text{IC}} = 191.315; \]

**Beam Dimensions**

\[ \text{In[2]} := b = 0.0254; (* \text{width in m} *) \]

\[ t = 0.0075; (* \text{total thickness in m} *) \]

\[ h = \frac{t}{2}; (* \text{individual beam thickness in m} *) \]

\[ a_0 = 0.0495; (* \text{initial crack length in m} *) \]

\[ I_1 = \frac{bh^3}{12}; (* \text{Moment of inertia in m}^4 *) \]

**Material Properties and calculated parameters**

\[ \text{In[3]} := \text{Props} = \text{Import["149props.dat"]}; \]

\[ E_{11} = \text{Props[[1, 1]]}; (* \text{Long. Modulus in Pa} *) \]

\[ E_{22} = \text{Props[[2, 1]]}; (* \text{Trans. Modulus in Pa} *) \]

\[ G_{12} = \text{Props[[3, 1]]}; (* \text{Shear Modulus in Pa} *) \]

\[ G_{13} = G_{23}; (* \text{Shear Modulus in Pa} *) \]

\[ v_{12} = \text{Props[[4, 1]]}; (* \text{In-Plane Major Poisson's Ratio} *) \]

\[ v_{21} = \frac{E_{22}}{E_{11}} v_{12}; (* \text{In-Plane Minor Poisson's Ratio} *) \]

\[ v_{13} = v_{12}; (* \text{Out-of-Plane Major Poisson's Ratio 1} *) \]

\[ v_{31} = v_{21}; (* \text{Out-of-Plane Minor Poisson's Ratio 1} *) \]

\[ v_{23} = \text{Props[[5, 1]]}; (* \text{Out-of-Plane Major Poisson's Ratio 2} *) \]

\[ v_{32} = v_{31}; (* \text{Out-of-Plane Minor Poisson's Ratio 2} *) \]

\[ \lambda = \frac{E_{11}}{E_{22}}; \rho = \sqrt{\frac{E_{11} E_{22}}{2G_{12}} - \left(1 - v_{12}ight)^2}; \]

\[ Y = \frac{0.677 + 0.146(p - 1) - 0.0128(p - 1)^2 + 0.00242(p - 1)^3}; \]

\[ K = \frac{10(1 + v_{13})}{12 + 11v_{13}}; (* \text{Timoshenko Beam Shear Coefficient} *) \]

\[ A_{\text{beam}} = bh; (* \text{Beam Cross-sectional Area in m}^2 *) \]

**Second Linear Softening and Power Law Mixed Function Properties**

\[ \text{In[4]} := \text{CZProps} = \text{Import["149CZProps.dat"]}; \]

\[ \omega = \text{CZProps[[1, 1]]}; (* \text{Max Bridged Stress in Pa} *) \]

\[ \delta c = \text{CZProps[[2, 1]]}; (* \text{Max Bridged Displacement in m} *) \]

\[ \delta 0 = \text{CZProps[[3, 1]]} \delta c; (* \text{Location of transfer from Perf. Plastic to Power Law} *) \]

\[ m = \text{CZProps[[4, 1]]}; (* \text{Power Exponent} *) \]

\[ \omega 0 = \omega m (1 - \frac{\omega 0}{\delta c}); (* \text{Stress at transfer point} *) \]
Euler-Bernoulli beam theory displacement and moment for a point load at the end of the beam, along with Timoshenko displacement:

\[ \delta \text{EBP}[x.] := \frac{P}{6 E_{11} \lambda_1} (3 a (x^2) - (x^3)) \]

\[ \text{MEBP}[x.] := -P (a - x) \]

\[ \delta \text{TP}[x.] := \delta \text{EBP}[x] + \frac{1}{K G_{13} \lambda_{\text{bend}}} (\text{MEBP}[x] - \text{MEBP}[0]) \]

SERR and Stress Intensity Factor Calculations

\[ \text{In}[6] := G_{\text{calc}}[P., \ a.] := \frac{12 \left( P a \right)^2}{E_{11} h^2} \left( 1 + \frac{E_{12}^2}{E_{11}^2} \frac{h}{a} \right) \]

\[ K_I[G_{\ldots}] := \sqrt{G \lambda_1^4 \sqrt{\frac{2E_{11} E_{12}}{1 + \rho}}} \]

\[ K_{II}[G_{\ldots}] := \sqrt{\frac{1 + \rho}{2E_{11} E_{12}} \frac{K^2}{\lambda_1^4}} \]

\[ K_{IC} = K_I[G_{IC}] \]

Determine bridged zone displacements and tractions through iterative process

\[ \text{In}[7] := \ll \text{NumericalMath}'\text{PolynomialFit}' \]

\[ \text{deferr} = \frac{\delta \text{TP}[\Delta a]}{1000}; \quad (* \text{Defined Error Value} *) \]

\[ \text{fitorder} = 8; \]

\[ \text{Op} = \text{Table}[0, \{nmax - 1\}, \{n\}]; \]

\[ \text{For} \left[ n = 2, n \leq nmax, n++, \right. \]

\[ P = \text{Loads}[[n, 2]]; \Delta a = \frac{\text{Loads}[[n, 1]]}{1000}; a = a0 + \Delta a; \]

\[ \delta n[x.] := \delta \text{TP}[x]; \]

\[ \text{calcerr} = 1; \]
While[calcerr > deferr, 
  dncheck = δn[Δa];
  qn[x_] := Which[δn[x] ≤ δ0, 
    c0 (1 - c0 (δn[x] - δ0)) b, δn[x] > δ0 & δn[x] ≤ δc, 
    c0 (δn[x] - δc) b, δn[x] > δc, 0];
  num = 50; inc = Δa/num; xt = 0; k = 1;
  points = Table[0, {num + 1}, {2}];
  Do[points[[k, 1]] = xt; points[[k, 2]] = qn[xt];
    k++, {xt, 0, Δa, inc}];
  qt = PolynomialFit[points, fitorder];

  MEBbr[x_] = DSolve[{M''[x] == -qt[x], M'[Δa] == M[Δa] == 0}, 
    M[x, x][[1, 1, 2]]];

  δEBbr[x_] = DSolve[{E''[1] E''[Δa] == qt'[x], 
    E'[1] E''[Δa] == E'[1] δ''[Δa] == 
    δ'[0] == δ[0] == 0}, δ[x], x][[1, 1, 2]];

  δThr[x_] = δEBbr[x] + Kg Kbeam (MEBbr[x] - MEBbr[0]);

  δnp1[x_] := δTP[x] - δThr[x];
  dnplcheck = δnp1[Δa];
  calcerr = Abs[dnplcheck - dncheck];
  If[calcerr < deferr, Break[]];
  dnpl[x_] := δnp1[x];
  δ[x_] := δTP[x] - δThr[x];

  q[x_] := Which[δ[x] ≤ δ0, 
    c0 (1 - c0 (δ[x] - δ0)) b, δ[x] > δ0 & δ[x] ≤ δc, 
    c0 (δ[x] - δc) b, δ[x] > δc, 0];
  xt = 0; k = 1; points2 = Table[0, {num + 1}, {2}];
  Do[points2[[k, 1]] = xt;
    points2[[k, 2]] = q[xt]; k++, {xt, 0, Δa, inc}];
  qt2 = PolynomialFit[points2, fitorder];

  Aq = Integrate[qt2[x], {x, 0, Δa}];
  Qq = Integrate[qt2[x] y, {x, 0, Δa}];
  xcent = Qq/Aq;
  Pbr = Integrate[qt2[x] dx, {x, 0, Δa}];
Op[[n - 1, 1]] = Aa;
Op[[n - 1, 2]] = P;
Op[[n - 1, 3]] = δ[Aa];
Op[[n - 1, 4]] = G_{appl} = G_{calc}[P, a];
Op[[n - 1, 5]] = K_{appl} = K_1[G_{appl}];
Op[[n - 1, 6]] = G_{br} = G_{calc}[Pbr, xcent];
Op[[n - 1, 7]] = K_{br} = K_1[G_{br}];
Op[[n - 1, 8]] = K_{tip} = K_{appl} - K_{br};
Op[[n - 1, 9]] = G_{tip} = G_1[K_{tip}];
Op[[n - 1, 10]] = K_{r} = K_{ic} + K_{br};
Op[[n - 1, 11]] = G_{r} = G_1[K_{r}];
Clear[δ, δn, δnnpl, δEBbr, δTbr, M, MEBbr, q, qn, qt, qt2];
Op
(* Output is: Δa P δ[Δa] G_{appl} K_{appl} G_{br} K_{br} K_{tip} G_{tip} K_{r} G_{r} *)
In[8] := Export[out.dat, Op, Table]
Out[8] = out.dat