Characterization of Mixed-Mode Fracture Testing of Adhesively Bonded Wood Specimens

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ABSTRACT

The primary focus of this thesis was to investigate the critical strain energy release rates ($G_c$) for mixed-mode (I/II) fracture of wood adhesive joints. The aims of the study were: (1) quantifying the fracture properties of two material systems, (2) analyzing the aspects that influence the fracture properties of bonded wood, (3) refining test procedures that particularly address layered orthotropic systems in which the layers are not parallel to the laminate faces, of which wood is often a particular case, and (4) developing testing methods that enhance the usefulness of performing mixed-mode tests with a dual-actuator load frame. The material systems evaluated experimentally involved yellow-poplar ($Liriodendron tulipifera$), a hardwood of the Magnoliaceae family, as adherends and two different adhesives: a moisture-cure polyurethane (PU) and a phenol/resorcinol/formaldehyde (PRF) resin. The geometry tested in the study was the double cantilever beam that, in a dual-actuator load frame, can be used for testing different levels of mod-mixity. The mixed-mode loading condition is obtained by applying different displacement rates with the two independently controlled actuators of the testing machine.

Characteristic aspects such as the large variability of the adhesive layer thickness and the intrinsic nature of many wood species, where latewood layers are alternated with earlywood layers, often combine to confound the measures of the critical values of strain energy release rate, $G_c$. Adhesive layer thickness variations were observed to be substantial also in specimens prepared with power-planed wood boards and affect the value of $G_c$ of the specimens. The grain orientation of latewood and earlywood, materials that often have different densities and elastic moduli, limits the accuracy of traditional standard methods for the evaluation of $G_c$. The traditional methods, described in the
standards ASTM D3433-99 and BS 7991:2001, were originally developed for uniform and isotropic materials but are widely used by researchers also for bonded wood, where they tend to confound stiffness variations with $G_c$ variations. Experimental analysis and analytical computations were developed for quantifying the spread of $G_c$ data that is expected to be caused by variability of the adhesive layer thickness and by the variability of the bending stiffness along wooden beams.
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Chapter 1

Introduction

1.1 Preface

The goal of mechanical design is to define structures appropriate for the external loads expected, meeting safety criteria without being cumbersome in terms of materials and costs. In particular, researchers with various backgrounds have been investigating fracture mechanics, this area being of great interest for design and reliability of structures. For fastening and connecting parts, adhesives are used as assembly techniques in many structural and non-structural applications [1, 2]. In the wood industry, adhesives have been widely used for producing construction materials (plywood, flakeboards, architectural doors and windows) and for fabricating structural components and assembling structures [3].

For bonded systems, the failure mechanisms are more complex due to the presence of interfaces and different materials [4] that influence the initiation and propagation of cracks that are often responsible for failure. Stress singularities can arise both because of geometry, such as notches, or because of the discontinuity of elastic properties, such as at the interfaces between adhesive and adherend. In a natural material like wood, the anisotropic stiffness matrix, the presence of heterogeneities and the specimen to specimen variability combine in influencing the fracture behavior of the bonded joint. Moreover, mixed-mode fracture is likely to happen in real adhesively bonded applications, due to the nature of both structural configuration and loading scenarios, but is not considered in most common test standards.

The focus of this study is on bonded wood structures subjected to fracture modes that have both mode I and mode II components. The experimental part focuses on evaluating the critical strain energy release rate that characterizes mode I, mode II and mixed-mode
I/II fracture of different adhesives for wood systems. The purpose of this part of the study is to obtain fracture energy envelopes that can be used in design practice and to determine which fracture mode results in the smallest critical fracture energies. Another part of the thesis is the analysis of the characteristics of the loading frame that was used for the tests, with evaluation of the nonlinear effects associated with the configurations of specimens and frame. Analyses of the factors that influence the fracture test results in bonded wood were also developed. The considered factors include the adhesive layer thickness and the variability of the local bending stiffness of the beams that are bonded to form the specimens.

1.2 Fracture mechanics background

Pioneering studies regarding fracture mechanics were developed by Griffith and Irwin. Almost a century ago, Griffith [5] developed a theory for an infinite plate containing a crack and subjected to uniform stress. Drawing upon the concept of potential energy, Griffith showed that for every level of applied stress, there is a critical value for the crack length. At this value, the system is at a maximum of potential energy, meaning that, if from this condition the stresses or the crack length are increased, the system will reduce its potential energy by increasing the crack length, basically developing a failure. A few decades later, Irwin [6] introduced the concepts of stress intensity factor (K) and strain energy release rate (SERR or \( G \)), which are still used in fracture mechanics. Critical values of stress intensity factor and strain energy release rate are material attributes that characterize the resistance to elastic fracture.

The two concepts are widely used to analyze the correlation among crack growth, material properties, and input test parameters, which include the imposed displacements or loads [7, 8]. In a monolithic isotropic and homogeneous material with elastic modulus \( E \) and Poisson’s ratio \( \nu \), the two parameters \( G \) and \( K \) can be calculated as in Equation 1, where index \( i \) refers to the in-plane loading modes: I and II.

\[
G_i = \frac{K_i^2}{E} \quad \text{where} \quad \begin{cases} E & \text{plane stress condition} \\ E/(1-\nu^2) & \text{plane strain condition} \end{cases}
\]

The concept of the SERR comes from an energetic approach with elastic deformation hypotheses [6, 9]. When external loads are applied to a cracked system and the crack
propagates, part of the work given by the loads is stored in the system as elastic energy and part is spent in propagation of the crack. The available energy for crack propagation equals:

$$\mathcal{G} \cdot dA = dW - dU$$

where $\mathcal{G}$ is the applied or available SERR, $dA$ the infinitesimal propagation of crack area, $dW$ the work of the internal forces and $dU$ the variation of stored elastic energy. The critical SERR, $\mathcal{G}_c$, is the amount of energy per unit of area required to create new crack area. If the crack growth is driven by a constant external load and in a system with linear force vs. displacement relation, $\mathcal{G}_c$ can be expressed as follows:

$$\mathcal{G} = \frac{P^2}{2} \frac{dC}{dA}$$

where $P$ is the applied external load and $C$ the specimen compliance.

A large amount of literature has described fracture in homogeneous and isotropic materials. Comprehensive reviews of the classical papers of Griffith and Irwin, newest approaches, and a description of state of the art testing techniques can be found in [10-13]. Fracture often occurs with the nucleation of a crack that then grows and can lead the material to complete failure. Nucleation may occur at points with stress concentrations or singularities in the material; material properties, discontinuities and presence of voids, flaws and other irregularities also influence the nucleation and propagation phases. Moreover, mixed-mode loading conditions are likely to occur in real applications, due to the nature of both structural configuration and loading scenarios [14]. Other issues related to fracture mechanics during the crack growth, such as the direction of crack propagation, have been developed by Erdogan [15] and Cotterell [16].

The critical SERR and $\mathcal{G}$ often depends on the fracture mode that is present at the crack tip, where mode I, mode II, mode III, or combinations of these can cause the crack to propagate. The applied SERR can have components associated with each of the three fracture modes [17]. This study focuses on in-plane loading conditions, as a first step for the characterization of generalized mixed-mode behaviors. In-plane loading conditions are defined by the presence of only mode I and mode II loads. In this case, the angle of mode-mixity is indicated as in [18] with the letter $\Psi$ and defined in the following:
\[ \Psi = \text{ArcTan} \left( \frac{G_{II}}{G_{I}} \right) \]

The critical SERR values referring to different pure mode fractures generally have different numerical values [19].

1.3 Fracture analysis in bonded joints

Ripling et al. [20] was one of the first papers that investigated the fracture properties of adhesively bonded joints applying the concept of \( G \). In that paper, aluminum-epoxy systems were tested for obtaining critical values of the parameter \( G \). Ripling et al. also found that the critical value of \( G \) is not just a function of the material system but also of some geometrical features, such as the bondline thickness. Bennett et al. [21] showed experimentally that \( G_c \) is a property of the material (or of the material system in bonded joints) and \( G_c \) is, within some limits, independent of the particular geometry of the specimen.

A complete fracture characterization of an adhesively bonded system may require evaluating the critical SERR for the pure modes as well as a range of mixed-mode loading conditions. This activity typically necessitates multiple tests and specimen configurations to produce the various fracture modes and combinations. One of the most common geometries used for fracture characterization of bonded specimens is the double cantilever beam (DCB) type specimen [17, 22]. This geometry typically consists of two equal beams bonded together, having one of the beams of the specimen connected to the fixed grip of the load frame and the other pulled by the testing machine’s actuator, thus resulting in an applied fracture mode that is nominally pure mode I. The symmetric DCB specimen can also be connected to traditional testing frames in different ways and other discrete mode mixities can be obtained. In these cases the specimens are traditionally referred to with different names corresponding to the particular loading mode, although the specimen geometry can be essentially the same. The illustrations in Figure 1 show how different levels of mode mixity are traditionally obtained in adhesively bonded joints. Pure mode II is achieved with end notched flexure (ENF) and end loaded split (ELS) specimens, pure mode I with the DCB specimen, and a fixed mode mixity level of \( G_{II}/G_{I} \) equal to \( \sqrt{(3/4)} \) with the single leg bend (SLB) specimen.
Figure 1: Test applications of specimens with DCB-like configuration (ENF: end notched flexure, ELS: end loaded split, DCB: double cantilever beam, SLB: single leg bend).

1.4 Wood material

Few materials have been used throughout history as much as wood. This is due to the relative abundance of the material in many areas of the world and the properties of this natural material. Wood has unique characteristics among natural materials in terms of accentuated anisotropy, providing good strength to weight and stiffness to weight ratios in the grain directions. The combination of these characteristics has facilitated, for example, wood’s use in construction: it is relatively easy to split or saw wood parts from a large block or a tree trunk. Considering also the general low density and the renewable nature of wood, the crucial role of this material in human civilization becomes evident. Still today wood is prized for various applications, ranging from the paper industry to building construction [3, 23]. Relatively new population growth in developing countries and ecological issues in developed countries have further increased the use of this renewable material worldwide [24].

Structurally, all wood is composed of basic units called cells, which are long, narrow, and aligned with the trunk, forming the characteristic wood grain. From the chemical point of view, three basic components form wood: cellulose, hemicelluloses and lignin. Cellulose is produced within a newly formed wood cell and consists of a linear chain of glucose monomers ($C_6H_{10}O_5$). The chain has a degree of polymerization of the order of $10^3$-$10^4$ and is a stable component of the cell walls. Hemicelluloses are formed from other sugars and are branched polymers with a degree of polymerization of the order of $10^2$. Hemicellulloses are usually easily hydrolyzed by acid or base solutions. Lignin is an amorphous polymer formed from different alcohols, and it fills the spaces between the other two components in the cell walls. It provides mechanical strength to the cell wall,
being present within the different cells. Some other components such as gums, resins and waxes may be present in small quantities and usually do not significantly influence the mechanical properties of the wood [25].

Adhesive constructions are used as assembly techniques in many structural and non-structural applications [1, 2]. The technique of adhesive bonding has played an important role in the wood products industry. Adhesives are used for manufacturing building materials, such as plywood particleboards and flakeboards, and also in structural applications such as framing, I-joists and glued laminated timbers.

1.5 Fracture in wood

From a mechanical standpoint, wood can be described as an orthotropic material [26-29]. The principal axes of the stiffness matrix are associated with three directions indicated in Figure 2: the longitudinal (L) axis is parallel to the grain, the radial (R) axis is perpendicular to the growth ring and the grain, and the tangential (T) axis is tangent to the growth ring and perpendicular to the grain. In a natural material like wood, the presence of heterogeneities and specimen to specimen variability combine in influencing mechanical properties.

![Figure 2: Principal axes of wood with respect to grains and growth rings.](image)

Analytical and experimental analysis of fracture mechanics properties in orthotropic materials was performed by Sih et al. [30] and Wu [31], who independently found that, comparing orthotropic with isotropic materials, the stress distributions at the crack tip have singularities of the same order and also the stress intensity factors are similar. A review of fracture propagation in orthotropic materials, such as wood, composites and rock, is presented by Boone et al. in [32], where numerical analyses are performed.
focusing on the effects of the independent orthotropic natures of stiffness and toughness in the crack path selection.

For wood, structural heterogeneities and hygroscopic nature are also factors that influence the mechanical properties of the material [33-35]. With numerical analysis and experimental work on beech, Triboulot et al. [36] confirmed the orthotropic nature of wood, pointing out the intrinsic problem that the high variability in wood properties requires researchers and engineers to deal with extensive statistical analysis when critical values of fracture toughness are to be evaluated. Other important contributions were given by Schniewind and Centeno [37], who measured the fracture toughness along different directions in Douglas-fir wood. They found that the critical stress intensity factors along the longitudinal and the radial directions differ by one order of magnitude. In another study, the same author and Yen and Schniewind [38] applied the J-integral, investigating the effects of moisture content and temperature on the critical values of strain energy release rate and stress intensity factor in Douglas-fir. Porter [26] focused on the critical strain energy release rate and concluded that in western white pine the critical value of $G$ along the tangential direction was higher than in the radial direction. A review of the principal elastic constants and critical strain energy release rates along the different axes for oak, birch, spruce and pine is given by Tomin [39]; in this study the variability of $G_c$ among the different axes was determined to be around a factor equal to 2 for oak and equal to 10 for the other species. Other interesting works on wood fracture, focusing on maritime pine, were developed by de Moura and colleagues that, with numerical analysis and experimental verifications, implemented cohesive zone models for tests in mode I [40], mode II [41], and mixed-mode [42]. A major achievement of these works is that data reduction schemes that allow the evaluation of critical strain energy release rates without the need for crack length measurements were developed for the different loading cases. The same group of researchers [43] also performed an inverse method that, starting from fracture data for maritime pine and Norway spruce and finite element simulations, evaluated the characteristics of the cohesive zone in the two species for three-point bending tests.

Other studies focused on the factors influencing the fracture properties of bonded wood construction. Walters [44], for example, found that in southern pine veneer glued
with phenol-formaldehyde the critical loads at which a crack propagates increase when the moisture content of the wood substrates decreases. Koran and Vasishth’s [45] work gave some useful indications on fracture properties of Douglas-fir plywood bonded with phenol formaldehyde. They showed that critical fracture properties have a maximum for moisture content of 12% and also that a level of roughness of the wood surface before bonding that maximizes the fracture properties should be expected. The work of Ruedy [46], focusing on compact tension specimens made with yellow-poplar and polyvinyl acetate, measured $G_c$ corresponding to the bonded area of specimens loaded in mode I. He measured a value of $G_c$ varying from values around 850 J/m$^2$ for specimens with grain orientation parallel to the bondline, to 2000 J/m$^2$ for specimens with grain perpendicular to the bondline. Ruedy also experienced the typical problem of the crack propagating from the adhesive layer into the adherend, especially when the grain was parallel to the bondline. He observed very consistent results about $K_c$, which was largest when the crack was propagating completely within the adhesive bondline. Ruedy described the dependence of the $K_c$ value on the adhesive layer thickness, suggesting that in his material system the value of $K_c$ peaks to a maximum value for adhesive layer thickness close to 225 µm. 

Shupe et al. [47] conducted a series of tests for determining the angle of wettability for 22 species of hardwood, on the surfaces perpendicular to the three directions R, T and L, with phenol formaldehyde resin. In this study contact angles were measured to be highly variable and depended on drying method, wood species, and wood surface orientation. Much of the variability was attributed to the different roughnesses of the surfaces in the different situations. Mechanical methodologies for the fracture characterization of bonded wood have been studied and presented by de Moura et al., as done for fracture in wood; data reduction schemes based on compliance measurements that do not require the measure of crack length were developed and applied in mode I [48], mode II [49] and mixed-mode [50].

1.6 Mixed-mode fracture

The loading conditions encountered in real structural applications of wood can seldom be considered as pure modes, but are likely to combine two or more fracture modes. Mixed-mode loading conditions develop when a flat crack is not perpendicular to
the axis of principal stress or the system is subjected to a multi-axial stress state. Critical values of strain energy release rate and especially stress intensity factors are usually referred to the pure modes in the literature and standards [12, 13, 51, 52]. Under the hypotheses of elastic deformation, the stress fields resulting from mixed-mode loadings can be obtained as a linear superposition of the stress fields resulting from the pure modes. In particular, the applied strain energy release rate \( G \) is a scalar quantity and it should be noted that its applied value can be obtained as the sum of the applied pure mode components. Thus, for the applied value of strain energy release rate:

\[
G = G_I + G_{II} + G_{III}
\]

Techniques for partitioning the applied strain energy release rate into the pure mode components of Equation 5 have been developed by Williams [53], Schapery and Davidson [54] and Hutchinson and Suo [18]. These techniques apply to geometries such as beams and plates subjected to different loading conditions resulting in mixed-mode. The techniques describe how to compute the pure mode components of the applied strain energy release rates. Tay [55, 56] and Hashemi [57] addressed mixed-mode delamination in fiber composites, developing approaches and physical interpretations that are completely relevant to the fracture in bonded joints and orthotropic materials. Extensive investigations of the effects of mixed-mode fracture were developed by Liechti et al. [58-61]. Effects of materials, geometry and external loads on the fracture of bonded specimens have been studied by Chen and Dillard [62-64]. They were able to predict directional stability of the crack by analyzing the energy balance of the bonded specimens. The fact that a crack can follow a non-straight path during its growth is also a factor influencing the effective mode mixity at the crack tip. This effect can modify the real stress state at the crack tip from the expected pure mode to mixed-mode, as shown by Leivers et al. for quasi-static [65] and fatigue [66] tests in PMMA, by Streit and Finnie [67] for aluminum and by Cotterell [68] for various materials. A number of other studies [69-71] have also focused on elastic and elasto-plastic fracture, finding that a single parameter, the stress intensity factor \( K \), is not sufficient for properly characterizing the stress state at the crack tip. These studies utilized a second parameter, called the T-stress, that was first introduced by Williams [72]. In particular, the T-stress not only influences
the crack path selection as described in [64] but also the crack onset. Reviews of fracture mechanics in different materials and loading conditions are presented in [73, 74].

The critical value of $G_c$ for a material system cannot generally be calculated, but is usually evaluated with experiments. In real applications mixed-mode loading conditions are more common than pure modes and $G_c$ is also generally dependent on the fracture mode that is present at the crack tip, where mode I, mode II, mode III, or combinations of these can cause the crack to propagate. A large number of publications address the experimental techniques that can be used for measuring mixed-mode fracture properties of materials and bonded joints. Researchers used Brazil-nut specimens [75], Arcan specimens [19, 76], bonded beams tested with the use of special fixtures [77-82], or bonded beams tested in different configurations [63, 64, 81, 83] for imposing conditions of different levels of mixed-mode. A characteristic that is common to all of these applications is that the angle of mode mixity cannot be easily changed during a test. A review of fracture mechanics experimental techniques in different materials and loading conditions is described in [22].

1.7 The novelty of the experimental equipment

The dual-actuator load frame [84, 85] is a servo-hydraulic testing machine capable of providing variable mode mixity for fracture mechanics studies by imposing asymmetric displacements or loads on symmetric bonded beam specimens, such as used for DCB specimens.

In the dual-actuator load frame, each actuator has a load cell and a displacement sensor (LVDT) that provide information for quantitative evaluation of specimen behavior. The novelty of this testing frame can be summarized in the following points:

1. mode mixity is not obtained by a variation of the elastic properties of the adherends, but by the imposition of asymmetric loads or displacements;
2. the mode mixity can be varied throughout the test without the need of repositioning the specimen.

Details on the dual-actuator load frame that was used for this research are provided in Chapter 2.
1.8 Problem statement

Mixed mode loading conditions are more common than pure mode conditions in real applications. The characterization of bonded joints under mixed mode loadings is traditionally limited by the need of performing tests on specimens of different geometry for different mode mixity level, or the use of special experimental fixtures. The dual-actuator load frame permits flexibility in designing the tests and collecting data referred to different levels of mode mixity. The advantages of this testing frame include the fact that all the levels of mode mixity, from pure mode I to pure mode II, can be obtained by working with specimens of constant geometry.

The initial task of this study is to characterize the mixed mode fracture properties of different adhesively bonded material systems using a novel testing frame. This activity has to deal with the development and optimization of the testing procedure and with the analytical analysis of the experimental data. The aims of the dissertation are:

1. obtaining graphs that illustrate the fracture properties of adhesively bonded specimens over a range of applied mode mixity;
2. analyzing the aspects that influence the fracture properties of bonded wood;
3. implementing testing procedures that can enhance efficient use of the dual-actuator;
4. implementing analysis techniques that facilitate the evaluation of fracture properties of bonded specimens obtained with anisotropic materials, such as wood.

These objectives are intimately interconnected, so they will be developed in parallel, leveraging one with another since, for example, testing procedures can necessitate adjustment of the data analysis process or vice-versa.

1.9 Research contribution and objectives

The experimental part of this research focuses on evaluating the critical strain energy release rate that characterizes mode I, mode II, and mixed-mode I/II fracture of different adhesives for wood systems. The purpose of the study is to obtain the dependence of the fracture energy on the mode mixity. The results of this kind of analysis can be useful in design practice. An important aspect is, for example, that mixed-mode analysis gives the
possibility to determine which fracture mode mix results in the smallest critical fracture energy. In fact, standards [86, 87] and industrial practice [2] usually focus on pure loading modes, especially mode I, but studies of Singh et al. [88] and Dillard et al. [81] showed that for certain material systems, the critical fracture energies for some levels of mode-mixity are lower than pure mode critical fracture energies.

This work opens new horizons for the usability of the dual-actuator load frame in fracture testing. The test procedures that are developed are not limited to the material systems that are tested, but are general and can be applied to a broad range of problems. These testing procedures, for example, permit combining the advantages of the load frame with the advantages of some classical analysis techniques that are developed for pure mode tests.

Moreover, given the choice of wood as the material for running most of the tests, analysis of peculiarities of this material, regarding fracture of bonded joints, is performed. The particular characteristics of bonded wood include issues such as the variability of the adhesive layer thickness and the variability of elastic properties, mainly elastic modulus, of the adherends. The dependence of the measured values of $G_c$ on the values of adhesive layer thickness and on the method applied for the evaluation was investigated.

1.10 Dissertation outline

The dissertation is organized as a collection of journal papers that have been or are about to be submitted for publication. Each chapter is one of these papers, excluding this first chapter. Information about intended journal and co-authors of the papers are indicated as footnotes at the beginning of each chapter.

The dissertation is structured into the following chapters:

- Chapter 1 provides background information, reviews relevant literature for the topics of fracture mechanics in wood and bonded wood, and finally presents the objectives of this dissertation.

- Chapter 2 provides a description of the experimental effort for measuring mixed-mode (I/II) fracture properties of bonded yellow-poplar. The chapter also addresses the issue of adhesive layer thickness variability in bonded wood.
• Chapter 3 provides a description of experimental techniques that were developed for measuring the mixed-mode (I/II) fracture properties of bonded specimens with a dual-actuator load frame. The focus of the chapter is on the aspects that are characteristic of tests that are performed with this loading frame and on the evaluation of the nonlinear effects that arise from the experimental set-up.

• Chapter 4 analyzes the strengths and the weaknesses of current methods for evaluating the fracture properties of adhesively bonded joints when the adherends are orthotropic layered materials, such as wood. In particular, the chapter evaluates the bending stiffness variability that can be expected along wood beams and how this variability alters the measured fracture properties of the bonded specimen.

• Chapter 5 provides the analytical basis of an experimental and analytical method that is proposed for measuring the local bending stiffness along beams.

• Chapter 6 presents a final summary of the research with the relevant conclusions and proposes future work directions.

• Appendix A is a journal paper, already submitted, describing a project on durability of bonded joints. This work, although not directly related to the main focus of the thesis on bonded wood, depicts how an analysis based on simple and relatively inexpensive tests can direct research work on assessing properties of more complex and expensive structures.
Chapter 2

Characterization of mixed-mode I/II fracture properties of adhesively bonded yellow-poplar¹

2.1 Abstract

The focus of this paper is to present the results of an experimental effort addressing fracture under in-plane mixed-mode loading conditions in bonded wood. In particular, the study considers mode I, mode II, and mixed-mode I/II fracture. The material systems considered involved yellow-poplar (*Liriodendron tulipifera*), a hardwood of the Magnoliaceae family, as adherends and two different adhesives, a moisture-cure polyurethane (PU) and a phenol/resorcinol/formaldehyde (PRF) resin. A dual testing machine with two actuators is used for testing and permits a fine scanning of fracture behaviors at different mixed-mode I/II levels. The tests were performed on double cantilever beam (DCB) geometry specimens over the full range of in-plane mode mixities.

The results of the tests are shown in graphs of the two components (mode I and mode II) of the critical strain energy release rate. In these graphs, the two components are plotted against the angle of mode mixity and as fracture envelopes. All these graphs show the limiting values of energy release rate for different levels of mode-mixity. The experimental data are, as usual for wood, quite scattered. Some measurements are presented for describing a factor that can influence the variability of the results; this effect is the thickness of the bondline. Measurements of adhesive layer thickness were

¹ Prepared for submission to: Holtzforschung. Co-authors: Charles E. Frazier, Audrey Zink-Sharp, David A. Dillard
performed with microscope analysis of samples cut from DCB specimens. The variation of the adhesive layer thickness was on the order of 1 to 100 µm for specimens bonded with the PU resin and 10 to 50 µm for specimens bonded with the PRF resin.

2.2 Keywords

Adhesive joints, bonded wood, fracture, mixed-mode, bondline thickness, yellow-poplar.

2.3 Experimental equipment

For this research a dual-actuator testing frame has been used. The dual-actuator load frame [85, 88] is a servo-hydraulic testing machine capable of providing variable mode mixity for fracture mechanics studies by imposing asymmetric displacements or loads on symmetric bonded beam specimens, such as used for DCB specimens. In principle, the use of asymmetric specimens is also possible; however, having a dual-actuator, it is more practical to keep the specimen geometry constant and to impose asymmetric loading conditions. In the dual-actuator load frame, each actuator is independently controlled and has a load cell and a displacement sensor (LVDT) that provides information for quantitative evaluation of specimen behavior. The novelty of this testing frame can be summarized in the following points: (1) mode mixity is not obtained by a variation of the elastic properties of the adherends, but by the imposition of asymmetric loads or displacements; (2) the mode mixity can be varied throughout the test without the need of repositioning the specimen.

The dual-actuator testing machine illustrated in Figure 3 was built to our specifications before the beginning of the present research by McGaw Technologies Inc. (Fairview Park, OH), with support from the National Science Foundation under contract DMR-0415840. The dual-actuator is characterized by the presence of two actuators that can impose loads or displacements independently to the ends of the two beams of the bonded specimen. Unless the test is performed in pure mode I, the specimen is clamped in a vise at the lower end and loads are applied to the debonded ends of the beams by pins and clevises attached to the load cells of the actuators. A controller drives the two actuators; displacements and forces of the two actuators, as well as time as the test
progresses, are collected by a computer equipped with a data acquisition card (DAQ PCI 6229, National Instruments Inc., Austin TX). The crack length values of the specimen are read periodically by the operator running the test. The reading is performed using a magnification lens and facilitated by white correction fluid and a paper ruler applied on a side of the specimen. The values of crack length are entered into the data acquisition system. The control design allows imposing different combinations of displacement rates to the two actuators as described in [85]. The combination of different displacement rates results in different levels of in-plane mode mixity with a standard DCB specimen.

![Figure 3: Dual-actuator load frame and detail of specimen connection.](image)

### 2.4 Materials and specimens

The study focuses on yellow-poplar (*Liriodendron tulipifera*), a hardwood of the Magnoliaceae family, and two adhesives, a moisture-cure polyurethane (PU) and a two components phenol/resorcinol/formaldehyde (PRF) resin. Specimens consisted of 10mm-thick wooden adherends bonded together with one of the two adhesives. Preparation of the specimens started with two power-planed 10mm-thick boards of wood; the other dimensions of the boards were approximately 250x140mm. The boards were carefully cut from larger stock so that the orientation of the wood grain was between 3° and 6° with respect to the intended bond plane [89]. There are at least two reasons for the choice of maintaining the grain orientation fairly constant. First, this configuration is beneficial
in preventing the crack from moving from the bonded layer into the adherend substrate in the material systems that were considered. Second, a fairly constant grain orientation provides more consistent stiffness characteristics, limiting the sources of variability in the experiments. For the same reason, boards containing knots or macroscopic defects were not used for specimen construction.

A 50mm (2 inches) wide region of the board near one of the edges was colored with a wax crayon to limit adhesion and provide a pre-cracked area in the final bonded specimens. One board is represented in Figure 4.

![Figure 4: Wooden board for specimen construction.](image)

In order to bond these boards, one of them was placed on a precision balance and, after zeroing the load, the amount of adhesive equal to the upper limit indicated on the technical sheet was poured on the board. Then, removing the board from the balance, the adhesive was spread to obtain a uniform distribution and the second board was placed on the top of the adhesive layer. Finally, the assembly was placed in a cold press and, during this pressing phase, correct alignment of the boards to be bonded was facilitated by means of lateral constraints. Adhesive quantity, applied pressure and curing time that were imposed are those indicated in the technical sheets of the adhesives.

Sometimes multiple stacks of bonded boards were placed in the press and compressed at the same time. Particular care was taken to complete the bonding procedure without exceeding the prescribed handling time, which was on the order of a few minutes for both adhesives. The adhesive cured at room temperature inside the press while the prescribed pressure, 1 MPa (150 psi) for the PU resin and 0.68 MPA (100 psi) for the PRF resin, was applied. The boards were kept under pressure for 12 hours. After curing, the bonded pieces were cut into 20-mm-wide specimens and conditioned for at least two weeks in a
chamber at 60% relative humidity and 21°C (70°F). Additional operations were performed on the specimens to prepare them for testing. A hole was drilled through the width direction on the debonded end of each adherend. These holes provided a way to connect the specimens to loading clevises in the testing machine by using pins (see testing equipment paragraph). Next, one side of the bonded specimens was painted with typewriter correction fluid to enhance the ability to detect the crack tip and, finally, a paper ruler was bonded on the same side of the specimen to facilitate crack length measurements. As already mentioned, attention regarding the orientation of the wood grain was given throughout the assembly procedure. The orientation of the wood grain in the final specimen was between 3° and 6° with respect to the bondplane, as illustrated in Figure 5. The definition of axis and section directions in wood for our specimen is illustrated in Figure 6. Note that the radial and longitudinal axes are not parallel to the specimen cut, because of the 3-6° rotation just described.

![Figure 5: DCB specimen; note the grain orientations.](image1)

![Figure 6: Direction of axes in the wood beam used for DCB specimen.](image2)

**2.5 Experimental technique and data analysis**

With the dual-actuator load frame, the DCB specimen geometry can be tested over the full range of in-plane mode-mixity. By simultaneously applying different displacement rates with the two independently controlled actuators, different levels of
mode-mixity can be induced at the crack tip. For tests reported herein, the imposed displacement rates on the two beams were constant during each of several test segments. In these tests the mode-mixity angle is a function of the mode I and mode II components of the imposed forces and the crack length. It was observed during the tests that the angle of mode-mixity increases while the test proceeds and the crack grows. This is an interesting result, since during a single test not only a single value, but a range of mode-mixity angles, is scanned and consequently the number of test runs can be limited. A possible downside of this aspect is that the fracture process zone is changing in a single test and that the R-curve may not stabilize during crack growth. However, no R-curve trend was found in the test results when the tests were run at pure mode I or II (example shown later in Figure 15).

Another aspect to be considered comes with the data analysis. The test involves imposing different displacement rates on the two beams and measuring the forces on the left and right actuator. In the analysis of DCB and other standard tests, several improved approaches can be used for analyzing the data, including the corrected beam theory (CBT) and the experimental compliance method (ECM) described by Blackman, Kinloch, and their colleagues [90-92]. These techniques address some issues of the DCB test adapting the simple beam theory for the geometry of the DCB, where the two beams are connected through an adhesive layer. These issues include the root rotation at the crack tip, shear deformation of the beams, presence of load blocks, effects of beam thickness, and inaccuracies in crack length readings. These issues have been extensively described for mode I in the literature [90, 93-95]. The CBT evaluates the real compliance of the beams of the tested DCBs and corrects the crack length reading using a linear fit of the data of compliance and crack length. The ECM similarly evaluates elastic characteristics of the tested specimens with a differently arranged linear fit. ECM and CBT for DCB specimens are both traditionally based on mode I tests and require experimental data from pure mode I tests, although adaptations of the techniques to pure mode II have also been developed [92, 96-98]. One goal of the study is to test mixed-mode conditions, but it is chosen to have each of the specimens partially tested at the same level of mode-mixity. This gives the possibility to apply ECM and CBT for the same loading condition in all of the specimens. In particular, the choice is to perform
pure mode I testing on part of the procedure in all of the specimens. With this condition, the test has 3 separate phases; the first and the third run in mode I to obtain data points for the CBT and ECM, and in the second run the mixed-mode condition is obtained by imposing different displacement rates to the two beams. Note that both CBT and ECM assume that the effective stiffness EI of the beams is constant along the beam length, as is usually true for a homogeneous material. For wood this assumption is not always correct, as explained by Liswell [99]. Nevertheless ECM and CBT have been traditionally applied for measuring the fracture properties of bonded wood [89]. This assumption is revised and discussed in reference [100].

2.6 Experimental results

The tests were performed on a total of 33 specimens prepared with the PRF adhesive and 28 specimens prepared with the PU adhesive. Some tests were run in pure mode I or pure mode II, but typically mixed-mode tests, following profiles such as illustrated in Figure 7, were performed. The results are presented in terms of \( G_c \) components vs. the angle of mode mixity \( \Psi \). The curves, in Figure 8 to Figure 13, show the results for the two material systems, keeping track of data points coming from different samples by highlighting them in different colors. The graphs show the trend of the \( G_c \) components.
for mode I and mode II and of the total $G_c$, being the sum of the two components shown in Equation 6.

$$G_c = (G_c)_I + (G_c)_II$$

With the tests performed at constant displacement rates, the data in a single specimen were collected from left to right, since the angle of mode-mixity was automatically increasing during the test, as described in reference [101].

The graphs from Figure 8 to Figure 10 show that the results have significant data scatter, as is often common for wood and bonded wood fracture tests [36, 102, 103]. In particular, the fracture envelopes in Figure 10 show that the individual specimens are relatively consistent, but that specimen to specimen variability is considerable. In particular, within the individual specimen the scatter seems, in most cases, to have some trends, which are generally not consistent with the trends of other specimens tested at the same levels of mode-mixity. This aspect suggests that the scatter is probably not related to random variability or some noise-like disturbances during the test, but should depend on some variability of the specimen or of the bond. Moreover, the variability seems also not to be related to R-curve effects that are seen in several material systems [104, 105], since the observed trends of $G_c$ are not, for example, always monotonic (i.e., always increasing with $\Psi$ or always decreasing with $\Psi$).

The graph of Figure 9 shows $G_c$ for the PRF-bonded specimens. In particular, the value of $G_c$ associated with small mode-mixity angles ($\Psi<30^\circ$) is fairly constant, while for higher values of $\Psi$ the value of $G_c$ increases rapidly and $G_c$ for mode II loading is approximately eight times larger than $G_c$ for mode I loading. The fracture envelope of Figure 10 shows that the data scatter is relatively more accentuated for tests performed close to mode I loading. This is an unexpected outcome since usually mode II loading can introduce instability in the crack path selection, facilitating the increase of data scatter, but it has to be noticed that the x and y axes of the graph have different scales.
Figure 8: Critical SERR components for PRF bonded specimens (x mode I component, • mode II component of critical SERR).

Figure 9: Critical SERR for PRF bonded specimens.
Figure 10: Fracture envelope for PRF bonded specimens.

The graphs from Figure 11 to Figure 13 show the results obtained for the PU-bonded specimens. Also in this case, most of the observations that can be drawn are similar to the results of the PRF-bonded specimens. Also for PU-bonded specimens the data scatter is appreciable and occurs because of the values of \( G_c \) have different trends in the specimens. Also in this case the value of \( G_c \) is almost constant for angle of mode-mixity lower than 30° and then increases, as can be seen in Figure 12.

The values of \( G_c \) associated to pure mode I loading are quite similar for the two material systems, while for mode II loading the \( G_c \) of the PRF-bonded DCBs is roughly doubled over the \( G_c \) of the PU-bonded specimens. One positive outcome of the results was that the grain orientation has successfully prevented the crack from moving into the beams even for tests performed with mode II loading.
Figure 11: Critical SERR components for PU adhesive bonded specimens (x mode I component, • mode II component of critical SERR).

Figure 12: Critical SERR for PU bonded specimens.
A comparison between the fracture properties for mixed-mode I/II of the two material systems is shown in Figure 14, where the values of $G_c$ of the PRF- and PU-bonded specimens are plotted against the angle of mode mixity $\Psi$. The two material systems have very similar behaviors for values of $\Psi$ lower than 70-75°. For loading mode close to pure mode II, the $G_c$ of PRF-bonded specimens doubles the $G_c$ of PU-bonded specimens.
Figure 14: Critical SERR for PRF and PU bonded specimens.

2.7 Visual analysis of tested specimens

Visual inspection of failure surfaces is a common practice in fracture studies of adhesive joints, permitting, for example, one to recognize regions associated with adhesive or cohesive failure in a failed specimen. This simple procedure is not particularly useful in bonded wood samples, where the adhesive has a layer thickness generally smaller than 100 μm and often a color that makes it difficult to detect adhesive traces on the adherends. Other traditional surface analysis techniques, such as the X-ray photoelectron spectroscopy, may not be as helpful in studying bonded wood samples because the chemical compositions of the adhesive and adherends are similar.

In this study it is usually observed that after the tests the failure surfaces present nonuniform characteristics and sometimes alternate between smooth and rough areas, which suggest different failure mechanisms. Situations in which the adhesive plastically deforms before failing require more energy than situations in which the failure is brittle. In our case, especially for specimens prepared with PU adhesive, a connection was observed between the roughness of the bonded surface after failure and the recorded $G_c$. 

\[ G_c = G_I + G_{II} \]
Figure 15 shows an example on how $G_c$ varies in a test performed on one of the specimens prepared with the PU adhesive. $G_c$ varies between 300 and 700 J/m$^2$, when one would expect a fairly constant value or a value monotonically changing as a function of the crack length. The constant value for $G_c$ is expected considering that the critical SERR is a property of the tested material system. A monotonic trend for the failure of $G_c$ can, on the other hand, be an indication of the dependence of $G_c$ on the crack growth rate, considering that the crack growth slows down as the crack develops, when the tests are performed at constant imposed displacement rates. In this case, other specimens have similar behavior, presenting considerable variations of $G_c$ during the single test, but have different trends of the $G_c$. The only aspect that consistently correlates with the level of $G_c$ is the roughness of the surface after failure: areas of high $G_c$ values correspond to sections of failed surface with accentuated roughness and whitening of the adhesive, while reduced $G_c$ values correspond to smooth portions of the surface. The example illustrated in Figure 15 shows a specimen tested in pure mode I, where particularly rough failure surface areas were obtained at different portions of the failed surface. The fact that there is not a single trend of $G_c$ in the graph suggests that $G_c$ is in our case not influenced by other factors such as the crack length or speed, which change monotonically during the test.

Figure 15: Failure surfaces and local $G_c$ for PU bonded specimen tested in mode I.
Further analysis was performed on the state of the adhesive layer in the tested DCB specimens to detect possible causes of the scatter of experimental results. The adhesion mechanisms in wood samples are generally quite different from what occurs in metal or composite samples. Adhesive penetrates considerably deeper in the wooden beams and the bondline thickness is very thin [3]. Another example of how the bonding of wood differs from other materials is that, during the production of bonded specimens, pressure applied to the pieces to be bonded is generally controlled rather than the bondline thickness. The bondline thickness is a factor that influences the extension of the plastic zone and the value of $G_c$ [106, 107]. As a result, the variability of this value can lead to scatter of the measured values, as found in the experimental results previously illustrated.

### 2.8 Effect of adhesive layer thickness

The procedure for obtaining the measurements of the bondline thickness was based on the ideas and techniques applied by Frazier and Zheng [108] and Johnson and Kamke [109], and the methods described in [110]. The analysis was performed with a fluorescence microscope working with transmitted light. The microscope is a Zeiss Axioskop (Carl Zeiss, Oberkochen, Germany), the camera for image acquisition is a DS-Fi1 (Nikon Inc., Melville, NY).

For preparing microscope samples, parts of the DCB specimens are cut into microscope samples, which are 40-µm-thick slices of area roughly 5 by 5 mm. The slices were also stained with two different solutions, 0.5% safranin O or 0.5% toluidine blue O, in order to find the combination that would better highlight the presence of adhesive penetrating into the sample. After several initial tests, the combinations of visible light at the microscope, safranin stain for parts bonded with the PU adhesive and no stain for parts bonded with the PRF adhesive gave acceptable results. Some of the samples for microscopic analysis are shown in Figure 16.
Figure 16: Slices for microscope analysis ready to be tested
(from left to right: toluidine stained, unstained, safranin stained).

The transmitted light microscope cannot be directly used to look at bondline penetration in DCB specimens but needs thin samples that permit light to be transmitted through. This aspect complicates the analysis of adhesive penetration and bondline thickness. The required microscope samples can be cut from DCB specimens that either have or have not been tested. In the first case the information regarding the adhesive layer thickness is lost since the already thin layer has been disrupted during the test. In the second case the cutting of the sample destroys the DCB specimen. Consequently, in both cases it is not feasible to compare the local values of $G_c$ and the values of adhesive penetration and layer thickness in the same position. For untested specimens, some of the obtained pictures are shown in Figure 17 and Figure 18. The pictures illustrate the adhesive presence in the bondline (vertical line) and in the vessels around it. For the PU bonded specimens the presence of adhesive is less evident, since with the safranin stain both adhesive and wood exhibit a reddish coloration. The presence of adhesive in the wood vessels can be seen in the pores because of its slightly pink color.
Figure 17: Microscope images for PRF bonded untested DCB.
(Unstained sample, radial-tangential section, visible light).

Figure 18: Microscope images for the PU bonded untested DCB.
(Stained sample, radial-tangential section, visible light).

The analysis of different samples shows that the values of adhesive layer thickness and penetration in the substrate vary considerably, even within a single specimen. The thickness of the adhesive layer varies between 10 and 50 µm and the penetration between 0.1 and 0.75 mm for specimens prepared with the PRF adhesive. The thickness of the adhesive layer varies between 1 and 100 µm and the penetration between 0.3 and 1.5 mm for specimens prepared with the PU resin. The larger variability of $G_c$ data from PU bonded specimens may correlate with the more dramatic adhesive layer thickness variation. As already mentioned, with this kind of analysis both measures of adhesive layer thickness and fracture properties on the same DCB specimen cannot be obtained. Nevertheless, measurements can still be performed in specimens that were adjacent in the
initial board (see Figure 4). These comparisons were performed for specimens tested in mode I, where the additional perturbation given by the change of the angle of mode mixity is not present. In these measurements a direct relation between adhesive layer thickness and measured local value of $G_{lc}$ was apparent, as already seen in different material systems [107, 111]. An example of what was observed in one specimen is shown in Figure 19.

![Image](image1.png)

**Figure 19:** Local adhesive layer thicknesses and $G_{lc}$ values for a PU bonded DCB specimen tested in mode I.

In the case of Figure 19 the value of adhesive layer thickness is lower than 20 µm for sections where the value of $G_{lc}$ is around 200 J/m², while the thickness is around 50 µm in sections where the value of $G_{lc}$ peaks to almost 500 J/m². The dependence of $G_c$ on the thickness of the adhesive layer can be understood considering that in extremely thin layers the interfaces between adhesive and adherends influence the stress distribution at the crack tip and limit the amount of adhesive that is subjected to high stress levels in the area around the crack tip. With an increase of layer thickness, the $G_{lc}$ value is expected to grow, since more adhesive is allowed to plastically deform ahead of the crack tip. The increase of $G_{lc}$ is not indefinite, since when the layer thickness is large enough, compared
to the radius of the plasticized zone, the effect of the interfaces fades and $G_{lk}$ reduces to the value for the bulk polymer. In bonded wood, the thickness of the adhesive layer is particularly small, as the measurements have proved; thus it is very likely that the radius of the plastic zone is larger than the layer thickness, as local increase of layer thickness increases $G_{lk}$ in static tests.

**2.9 Conclusions**

Experimental work for characterizing the mixed-mode I/II fracture properties of bonded yellow-poplar was carried out. Two material systems applied to adhesively bonded double cantilever beams (DCB) specimens were tested. The material of the beams was yellow-poplar wood, while two commercial products were used as adhesives: a moisture-cure polyurethane (PU) and a phenol/resorcinol/formaldehyde (PRF) resin. The experimental procedure that was developed for measuring the mixed-mode fracture properties consists of 3 separate phases, two of which are mode I tests. This approach permits researchers to apply some of the insights of corrected beam theory (CBT) and experimental compliance method (ECM) also to mixed-mode tests. This possibility is particularly important when testing DCBs of non-uniform materials where elastic properties of the adherend are not known.

The curves of the components of $G_c$ vs. $\Psi$ and fracture envelopes $G_{lk}$ vs. $G_{IIc}$ that were obtained for the two material systems show a data scatter that is consistent with what is usually found in bonded wood. One of the possible reasons for the data scatter, the variability of bondline thickness, was investigated with microscope analysis and is presented in this paper. Measurements found that the adhesive layer thickness could vary between 1 and 100 $\mu$m for the specimens prepared with PU resin and between 10 and 50 $\mu$m for specimen prepared with PRF resin. Some comparisons of adhesive layer thickness and mode I critical fracture energy suggest that the thickness variation has a strong influence on the value of $G_c$ that is measured with the DCB samples. In particular, sections of reduced adhesive layer thickness are associated with lower fracture energies, and sections of relatively thick adhesive layer are associated with higher fracture energies.
2.10 Acknowledgments

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Chapter 3

Experimental and improved analysis procedures for mixed-mode fracture of bonded beams with dual-actuator load frame

3.1 Abstract

Numerous common methods and standards for conducting fracture tests of adhesively bonded specimens refer to pure mode measures and to traditional load frames, where only one actuator is present. The critical strain energy release rates that characterize mode I, mode II and mixed-mode I/II fracture of bonded adherends can also be measured using a dual actuator load frame in which there are two degrees of freedom. The geometry that is generally tested in the dual-actuator is the symmetric double cantilever beam (DCB) type. DCB specimens are commonly used in traditional load frames for pure mode I tests, but the independent actuators permit characterization over the full range of in-plane mode-mixity. Tests performed with the dual-actuator give new possibilities and simplify the experimental effort, but this method also introduces different possible issues.

The focus of this paper is to develop and present experimental and analytical aspects that are characteristic of mixed-mode fracture tests performed with a dual-actuator load frame. These aspects include the geometric nonlinearities that are introduced by the dual-actuator and the mode-mixity variability that can be achieved. New testing procedures are presented Goals of these procedures are to enhance the capabilities of the dual-

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actuator frame while maintaining the advantages of other techniques already in use with traditional testing and analysis methods.

### 3.2 Keywords

Adhesive joints, fracture testing, mixed-mode fracture, laminate, double cantilever beam, load frame, dual-actuator, correction factor.

### 3.3 Introduction

Adhesively bonded constructions are used as assembly techniques in many structural and non-structural applications. The advantages of adhesive bonding versus other methods, such as mechanical fastening, include the more uniform stress distribution, the possibility of connecting different materials, and the lighter weight of the final construction. Designers and engineers have traditionally assessed the strength of bonded joints with theoretical analyses, numerical simulations, and experimental work. Methods based on stress analysis give results that strongly depend on the geometry of the construction and are often quite complex due to the presence of two or more materials with different mechanical properties, the adherends and the adhesive, and the interfaces where they join. Methods based on fracture mechanics have proven to be reliable and are commonly applied in the characterization of bonded joints [17]. Fracture mechanics is generally used to analyze the correlation among crack growth, material properties, and input test parameters, which include the imposed displacements or loads [8]. The stress intensity factor ($K$) is commonly applied in stress-based methods, while the strain energy release rate (SERR or $G$) is usually preferred for fracture analysis of bonded joints.

The applied SERR can have components associated with each of the three fracture modes [17]. In particular, with mode I, the opening mode, the crack propagates with the opening of its faces normal to the crack plane due to applied tensile stresses. In mode II, the in-plane shear mode, the crack propagation results from in-plane shear stresses. Finally, in mode III, the out-of-plane shear mode, crack propagation results from out-of-plane shear stress. The applied strain energy release rate $G$ is a scalar quantity and its applied value can be obtained as the sum of the applied pure mode components as shown in Equation 7.
Techniques for partitioning the applied strain energy release rate into the pure-mode components of Equation 7 have been developed by Williams [53], Schapery and Davidson [54] and Hutchinson and Suo [18] for geometries such as beams and plates subjected to different loading conditions. Tay et al. [55, 56] and Hashemi et al. [57] addressed mixed-mode delamination in fiber composites, developing approaches and physical interpretations that are relevant to fracture in bonded joints and orthotropic materials. Extensive investigations of the effects of mixed-mode fracture were developed by Liechti et al. [58-61] and Dillard et al. [81, 83]. In particular, the critical value of $G_c$ for a material system cannot be calculated but is usually evaluated through experiments. $G_c$ also generally depends on the fracture mode that is present at the crack tip, where mode I, mode II, mode III, or combinations of these can cause the crack to propagate.

In real applications of adhesive joints, the loading conditions are seldom pure modes, but mixed-mode loading conditions are more common. A large number of publications address experimental techniques that can be used for measuring mixed-mode fracture properties of materials and bonded joints, including the methods of Wang et Suo [75], Fernlund and Spelt [82], Reeder and Crews [78], Tracy et al. [79], Choupiani [19, 76], Chen and Dillard [64], Park and Dillard [112], Sørensen et al. [77], and Chen et al. [80]. These researchers used different specimen geometries for imposing various levels of mixed-mode loading in specimens such as the Brazilian-nut and Arcan specimens, as well as bonded beams tested with various fixtures. A characteristic that is common to all of these applications is that the angle of mode-mixity cannot be easily changed during a test.

The study reported herein analyzes the aspects of experiments and data analysis that are specific to testing bonded beams with a dual-actuator load frame. The dual-actuator is a custom designed testing frame that imposes mixed-mode loading by applying forces or displacements with two independently controlled actuators. The configuration of the dual-actuator offers several advantages and flexibility during the tests. Papers on experimental results obtained from tests performed with this load frame were presented by Singh et al. [88] and Nicoli et al. [113]. Parts of the present paper are a description of the dual-actuator, a description of the testing procedures that are currently applied, an
estimate of beam foreshortening of the specimen and geometrically nonlinear effects during the test, and finally, the evaluation of mode-mixity variability during the test. Preliminary evaluations of the nonlinear geometric effects in pure mode I and mode II were partially presented by Nicoli et al. [114].

### 3.4 The dual-actuator load frame

The dual-actuator load frame is an instrument that simplifies the experimental effort that is traditionally required for collecting data for different values of mode-mixity. The dual-actuator permits, for example, spanning the complete range of mode mixities between mode I and mode II with the same specimen type and, basically, the same test configuration. The dual-actuator testing machine illustrated in Figure 20 was built to our [85] specifications by McGaw Technologies Inc. (Fairview Park, OH), with support from the National Science Foundation under contract DMR-0415840.

![Dual-actuator load frame and detail of DCB specimen.](image)

Figure 20: Dual-actuator load frame and detail of DCB specimen.

The dual-actuator is characterized by the presence of two actuators that can impose loads or displacements independently to the ends of the two beams of the bonded specimen. The specimens that are tested generally are of the standard double cantilever beam (DCB) configuration. Symmetric DCB specimens consist of two identical beams bonded together and are commonly used for tests performed in pure mode I [87]. In the dual-actuator load frame, unless the test is performed at pure mode I, the specimen is clamped in a vise at the lower end and loads are applied to the debonded ends of the beams by pins and clevises attached to the load cells of the actuators. Each actuator of
the dual-actuator is independently controlled and has a load cell and a displacement sensor (LVDT) that provides information for quantitative evaluation of specimen behavior. A controller drives the two actuators. Displacements and forces of the two actuators, as well as time as the test progresses, are collected by a computer equipped with a data acquisition card (DAQ PCI 6229, National Instruments Inc., Austin TX). The crack length values of the specimen are read periodically by the operator running the test. The reading is performed using a magnification lens and facilitated by white correction fluid and a paper ruler applied on a side of the specimen. The values of crack length are entered into the data acquisition system. The control design allows imposition of different combinations of displacement rates to the two actuators, thus resulting in different levels of in-plane mode-mixity with a standard DCB specimen.

The advantages of this testing frame can be summarized in the following points: (1) mode-mixity is not obtained by a variation of the elastic properties of the adherends, but by the imposition of asymmetric loads or displacements that can readily be controlled while a test is in progress; thus, data can be collected from a single specimen type, the common DCB; (2) the mode-mixity can be infinitely varied between pure mode I and pure mode II; (3) the mode-mixity can be varied during the test without need for repositioning the specimen or adjusting the loading fixture. The consistency of results found with the dual-actuator and traditional load frames was evaluated in [113] for pure mode I, mode II and for $\Psi = 40.9^\circ$. The latest case corresponds to the single leg bending (SLB) test [22] that can easily be obtained also with a traditional load frame, which in the case of the data of Figure 21 is an Instron 4505 (Instron, Norwood MA). The comparison was performed on bonded steel, and the coherence of results found with the dual-actuator load frame (DALF) and traditional techniques is shown in Figure 21.
3.5 Test procedures

Most common methods for testing bonded specimens apply to pure mode I, pure mode II, and SLB tests, and to traditional load frames where only one actuator is present [78, 80, 82]. With mixed-mode tests performed with a dual-actuator load frame, new procedures were implemented. The goal of these procedures is to enhance the capabilities of the frame without losing some of the advantages of other techniques already in use with traditional testing and analysis. The tests are conducted in displacement control to avoid catastrophic failures of the DCB specimens and permit multiple readings of crack length. Tests involve imposing different displacement ramps to the two beams and measuring the forces on the left and right actuator. These forces, $F_L$ and $F_R$, are then combined for evaluating the force $F_I$ that gives the mode I loading component and $F_{II}$ that gives the mode II loading component as:
\[ F_I = \frac{1}{2} |F_R + F_L| \]
\[ F_{II} = \frac{1}{2} |F_R - F_L| \]

These forces are then used for evaluating the values of \( G_I \) and \( G_{II} \) that, if referred to the conditions at which the crack grows, are respectively the mode I and the mode II components of \( G_c \). These components of the SERR can be easily calculated with simple beam theory, and the results are shown in Equation 9.

\[ G_I = \frac{F_I^2 a^2}{EI} \]
\[ G_{II} = \frac{3F_{II}^2 a^2}{4EI} \]

The angle of mode-mixity, \( \Psi \), is defined as in Equation 10.

\[ \Psi = \text{ArcTan} \left( \frac{G_{II}}{\sqrt{G_I}} \right) \]

In common practice in the analysis of DCB and other standard tests, several improved approaches are used for analyzing the data. These approaches are more refined than the simple beam theory and include the corrected beam theory (CBT) and the experimental compliance method (ECM) described by Blackman and Kinloch [91, 92, 115] and in the British Standard 7991 [86]. These techniques address refinements to traditional beam theory, purportedly accounting for root rotation at the crack tip, shear deformation of the beams, the effects of beam thickness, possible errors in estimating the adherend bending stiffness, and systematic inaccuracies in the crack length reading. These issues have been described for mode I in a number of papers [93, 94, 115]. The CBT evaluates the actual compliance of the debonding specimen and corrects the crack length reading using a linear fit of the cube root of compliance vs. crack length. The ECM similarly evaluates elastic characteristics of the tested specimens with a different approach, obtaining a power law fit of the compliance vs. crack length. ECM and CBT for DCB specimens are both traditionally based on mode I tests and require experimental data from pure mode I tests, although adaptations of the techniques to pure mode II have also been developed [96-98]. When mixed-mode fracture tests are performed with the dual-actuator, it is
advantageous to have each of the specimens partially tested at the same level of mode-mixity. In particular, the choice is to grow the crack in pure mode I for a portion of the specimen. This gives the possibility to apply ECM and CBT for the same loading condition for all specimens. With this condition, the test has three separate phases: the first and the last are conducted in mode I in order to get data points for the CBT and ECM, and in the second part, the mixed-mode condition is obtained by imposing different displacement rates to the two beams, as shown in Figure 22.

![Figure 22: Typical test procedure with 3 different phases: the first and the third with mode I loading and the second with mixed-mode.](image)

The use of CBT is particularly important when natural materials with considerable material property variation from specimen to specimen are tested. In the calculation of $G_c$ components from the mixed-mode part of the test, one need not rely on the elastic properties listed in tables or run additional tests, but can use the modulus of the specimen as obtained in the first and third parts of the test with CBT. It should be noted that CBT assumes that the effective stiffness $EI$ of the beams is constant along the beam length, as is usually true for homogeneous materials. Nevertheless, ECM and CBT have been traditionally applied for measuring the fracture properties of bonded wood, which is not a uniform material [89, 116]. An analysis of the use of ECM and CBT in fracture of bonded wood has been presented in [100].

This approach of testing with three different phases provides several other advantages. First, crack growth in tests with mode-mixity angles approaching mode II
are stable only for crack lengths that exceed a certain length. For example, the crack length has to be at least 55% of the total length of the specimen for pure mode II loading [117]. Thus, configurations with short crack lengths can be used for testing mode I or low $\Psi$ loading conditions more effectively than mode II and high $\Psi$ loading conditions. Second, mixed-mode tests require the specimen to be clamped at the bonded end, thus preventing the crack from completely debonding the specimen. The final phase performed as a mode I test on the unclamped specimen permits gathering additional data and eventually obtaining complete debonding of the DCB specimen. This last aspect is quite important, since CBT and ECM are based on fitting of compliance data with crack length data in a mode I testing. Thus, having data associated with both short (first phase with mode I loading) and long (final phase with mode I loading) crack lengths leads to a more accurate evaluation of the parameters defined by the fittings.

### 3.6 Nonlinear geometric effects

As previously described, the DCB specimens tested in the dual-actuator load frame are clamped at the base and the debonded ends of the two beams are connected to the actuators. The two actuators can rotate around two pivot points, thus allowing for beam foreshortening and avoiding damage to the instrument due to an over-constrained configuration. The geometrical changes that the specimen and load frame encounter during a test are shown in an exaggerated scale in Figure 23.

![Figure 23: Schematic view of geometry changes during a test on the dual-actuator (\(\Delta\) is the imposed displacement, \(\theta\) the cylinder rotation and \(\delta\) the beam foreshortening; subscripts L and R refer to left and right beam/actuator).](image-url)
Evaluating the nonlinear geometric effects focused on the beam foreshortening values \((\delta_L \text{ and } \delta_R)\) and the cylinder rotations \((\theta_L \text{ and } \theta_R)\), illustrated in Figure 23. These both affect the actual moments imposed at the crack tip. Foreshortening and cylinder rotation were described as functions of the actuator positions and crack length. The evaluations were carried out for pure mode I and pure mode II and took into account the geometric nonlinear effects in the system. The analysis particularly concentrated on the dual-actuator developed at Virginia Tech. In this testing frame, with reference to Figure 23, the distance between the actuators pivot points, \(2D\), is, in the current configuration, equal to 1400 \(mm\) and the length of the specimen outside the vise, \(L\), is generally 220 mm. Also, the maximum displacement of the two actuators, \(\Delta_L \text{ and } \Delta_R\), is +/- 50 mm. The moment applied at the crack tip changes not only because of beam foreshortening, but, due to actuator rotation, also because the applied force \(F\) has two components \(F_V\) and \(F_H\) that act on the beam, generating moment contributions that may be additive or subtractive.

### 3.7 Beam foreshortening and actuator rotations

The beams in the dual-actuator bend upon application of the forces by the two actuators. The deformed shape can be calculated in each case with the well-known equation 11, where \(M\) is the bending moment and \(EI\) the bending stiffness, assumed to be constant along the beam\(^3\).

\[
\frac{d^2 y}{dx^2} = \frac{M(x)}{EI}
\]

In the DCB specimens, the deformed shape can be calculated with the same equation, considering the correct values of \(M\) and \(EI\) in each section, as the bonded and the debonded portions of the specimen have different bending stiffness. In a pure mode I test, the two debonded legs of the beams are the only parts of the DCB that are bent; in pure mode II or mixed-mode tests, where the specimen is also clamped at the bottom,

\(^3\) The equation assumes small slopes. The accuracy of this assumption will be further investigated later in the paper.
both the debonded and the bonded portions of the specimen are subjected to deformation. This complicates evaluation of the deformed shape, but an estimate through Equation 11 can still be performed, considering the bonded part of the DCB beyond the crack tip as a single beam of double thickness. A schematic of the deformation during mode I and mode II loading tests is shown in Figure 24.

Figure 24: Beam foreshortening, force rotation and moment variation in DCB specimens tested in mode I and mode II loading conditions (the beam on the left is pulled; the beam on the right is pushed).

Given that the $F_V$ component is always small compared to $F_H$, the deformed shape of the beams is calculated considering only the load coming from $F_H$. Once the functions $y_L(x)$ and $y_R(x)$ for left and right beams are known, the foreshortening can be calculated for the respective beams. Direct evaluation of the foreshortening for an individual beam can be obtained with:

$$\delta = L - \int_0^L \sqrt{1 - (y'(x))^2} \, dx$$

where $L$ is the length of the DCB specimen and no rotation at the clamped end is assumed. In particular for mode I, the bonded part of the specimen does not deform and thus $y_L(x)$ and $y_R(x)$ are equal to zero for $x < a$. The beam foreshortening, $\delta_L$ and $\delta_R$, can be described as functions of the crack length $a$ and the imposed displacements, $\Delta_L$ and $\Delta_R$. 

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since also the deformed shape $y(x)$ of the beam can be expressed as a function of the imposed displacements $\Delta_L$ and $\Delta_R$.

With an approach that considers $\Delta_L$, $\Delta_R$ and $a$ as input parameters, values of beam foreshortening can be calculated for each beam. In particular, for mode I $\Delta_L$ is equal to $\Delta_R$ and thus the two parameters can be condensed into one, $\Delta$. Quite similarly, for mode II the magnitudes of $\Delta_L$ and $\Delta_R$ are equal, but the directions of the two are opposite, since one displacement pushes and the other pulls the specimen; also in this case the two parameters can be condensed into a single term $\Delta$. Results of the beam foreshortening for mode I are plotted in Figure 25.

![Figure 25: Beam foreshortening for mode I loading.](image)

For pure mode II loading, the evaluation is performed a bit differently, since the entire specimen, bonded and debonded portions, is bent for an applied displacement. Thus, the length of the deformed part does not change during a mode II test, but only the relative lengths of cracked and uncracked portions. Results of the beam foreshortening for mode II are plotted in Figure 26, where, for consistency with mode I, the represented foreshortening is nondimensionalized with respect to the length of the cracked portion of the specimen. The entire DCB specimen is bent during a mode II test, thus the value of the specimen length $L$ influences the results. For the results that are presented, the foreshortening is calculated imposing the specimen length $L$ equal to 220 mm, as typical for the DCB specimens used in the dual-actuator load frame. The data points of Figure 26 were obtained by evaluating the foreshortening for values of $\Delta$ varying between 0 and 50 mm and values of $a$ varying between 0 and 220 mm.
Figure 26: Beam foreshortening for mode II loading.

A parameter that is more accurate than the beam foreshortening for describing the nonlinearity is the moment at the crack tip. In fact, the foreshortening, along with force rotation, causes a change of the moment applied at the section corresponding to the crack tip, as shown in Figure 24. The moment at the crack tip during a test can be different from the nominal value, $M_{\text{nom}}$, which is equal to the applied force multiplied by the crack length. This effect is important because the moment at the crack causes the crack to grow.

The effect can be quantified by evaluating $M/M_{\text{nom}}$ as a function of displacement $\Delta$ and crack length $a$ as shown in Equation 13. $M$ is the actual moment applied at the crack tip and $M_{\text{nom}}$ the nominal value. It is interesting to note that in mode I loading the two force components, horizontal and vertical, generate opposite moments at the crack tip, while in mode II loading the two force components combine to create moments with the same orientation. Therefore in mode I the moment reduction is more accentuated than in mode II.

$$\frac{M}{M_{\text{nom}}} = \frac{F_H \cdot (a - \delta) - F_V \cdot \Delta}{F \cdot a} \quad \text{Mode I}$$

$$\frac{M}{M_{\text{nom}}} = \frac{F_H \cdot [L - (l_{\text{up}} + \delta)] + F_V \cdot (\Delta - \Delta_{\text{up}})}{F \cdot a} \quad \text{Mode II}$$

The results of Equation 13 do not align on a single curve, since the force rotation depends not only on the values of $\Delta$ and $a$ but also on the distance between the dual-actuator pivot points, which in our case is 1400 mm. The moment reduction for mode I and mode II loading was calculated by imposing discrete changes of $a$ and $\Delta$, in the same
fashion already used for the beam foreshortening. For comparison purposes, a simulation of two material systems was also developed. The material systems that were considered are: (1) aluminum 6061 - T6 bonded with epoxy ($E = 70\, GPa$, $\sigma_y = 240\, MPa$, section of the beam $25 \times 20\, mm$, $G_{Ic} = 5000\, J/m^2$, $G_{IIc} = 8000\, J/m^2$); (2) carbon composite bonded with epoxy ($E = 50\, GPa$, $\sigma_{max} = 500\, MPa$, section of the beam $25 \times 4\, mm$, $G_{Ic} = 1000\, J/m^2$, $G_{IIc} = 2000\, J/m^2$). The results of Equation 13 and of the two cases of bonded aluminum and carbon composite are plotted in Figure 27 and Figure 28 for, respectively, mode I and mode II loading conditions.

![Figure 27](image1)

Figure 27: Moment reduction for mode I loading. The open symbols are the data points for bonded aluminum, the closed squares the data points for bonded composite.

![Figure 28](image2)

Figure 28: Moment reduction for mode II loading. The open symbols are the data points for bonded aluminum, the closed squares the data points for bonded composite. (Note that the lines associated with the different crack lengths are almost superimposed on each other and the scale of y-axis is magnified).
Figure 27 shows that for mode I loading, the moment reduction can be, theoretically, quite substantial, although in real specimens it is likely to be limited to a few percent. Large moment reduction could virtually be reached for short crack lengths and relatively high displacements but would not physically occur because of the limiting $G_{lc}$ value. Figure 28 shows that in mode II loading, the moment reduction is relatively small and almost independent of crack length. It should be mentioned that the graph of Figure 28 plots lines and data points obtained for crack lengths, $a$, larger than 120 mm, since the stability of a mode II test for DCB specimen type can be obtained only for $a/L > 0.55$ [117] and in the presented analysis $L$ is equal to 220 mm. For mode I loading, the moment reduction for the bonded aluminum case is always negligible and does not reach the value 0.1%, while for the bonded composite it is less than 2% for crack lengths up to 160 mm, less than 5% for crack lengths up to 200 mm, and peaks to 7% for crack length equal to the length of the specimen, 220 mm. For mode II loading, the maximum moment reduction is less than 0.2% for bonded aluminum and 4% for the carbon composite.

### 3.8 Accuracy of small slopes assumption

An important verification to be performed is the accuracy of the small deformation and slopes hypothesis, which was assumed for describing the deformed shape of the DCB specimen. The assumption permits one to write Equation 11, which is a differential equation of simple solution, instead of the more complex exact form shown in Equation 14.

$$\frac{d^2 y}{dx^2} = \frac{M(x)}{EI} \left[1 + \left(\frac{dy}{dx}\right)^2\right]^{\frac{1}{2}}$$

The approximation comes from the assumption of the denominator of the left hand term of Equation 14 to be equal to the unity. To some extent, Equation 14 is still an approximation. In fact, the equation does not take into account axial forces required for the solution of an elastica. In the case of the considered dual-actuator, the axial forces are neglected in this paper since the cylinder rotation, which introduces a vertical force
component, is always smaller than 1°. Other possible nonlinearities, such as specimen rotation in at the clamp and shear deformations, are also neglected.

The plots of the term that was neglected with the small slopes assumption, the denominator of the left hand term in Equation 14, were developed for the cases of bonded aluminum and bonded carbon composite that were considered in the previous section of this paper and are shown in Figure 29 and Figure 30.

![Figure 29: Denominator of the left hand term Equation 14 for mode I loading for bonded aluminum (above) and bonded carbon composite (below). The crack lengths are 40, 100, 160 and 220 mm.](image-url)
The plots of Figure 29 and Figure 30 show the value of the denominator of the left hand term of Equation 14 along the deformed portions of the DCB specimen. In the $x$ axis direction of the graphs, the position $x = 0$ corresponds to the crack tip section for mode I loading and to the bottom part of the specimen for mode II loading, as illustrated in Figure 24. Thus, the plots associated with mode I loading show graphs that extend along the $x$ axis from 0 to $a$, since only the cracked portion of the specimen deforms. On the other hand, the plots associated with mode II loading extend throughout the length of the specimen, 220 mm, since the entire specimen deforms.

The plots of Figure 29 and Figure 30 show that the denominator term of Equation 14 is very close to the unity for aluminum DCBs, while the denominator is significantly larger than unity for the more compliant carbon composite DCBs. Nevertheless, for mode I loading the denominator remains fairly close to unity also for the carbon composite.
composite DCB, being for example less than 1.04 for a crack length of 160 mm (Figure 29 right). For pure mode II loading and carbon composite DCB, the denominator exceeds the value 1.10 in part of the specimen, thus suggesting that the small-displacement hypothesis can be less accurate. However, other than the case of \( a = 220 \) mm, the denominator of the left hand term of Equation 14 exceeds 1.10 only for a minor portion of the specimen. These conclusions show that the analyses performed for evaluating beam foreshortening and moment reduction (based on Equation 11, which assumes small displacements and deformations) are still a reasonable approximation of Equation 14 for most of the combinations of crack lengths and displacements that are of physical importance for real specimens.

### 3.9 Geometric nonlinearity correction factors

Papers presented by Williams et al. [92, 118, 119] have already evaluated correction factors that can be used for mode I and mode II loading of DCB-type specimens. In particular, Williams evaluated two correction factors to be included when tests involve large displacements or when loads are introduced through end-blocks. These factors, \( F \) and \( N \), correct the term \( G_i \) (\( i \) indicates the loading mode, I or II) calculated with Equation 9 (or other methods) for accounting respectively for the large displacements at the load point and the presence of end-blocks. The two factors are prescribed, for mode I loading, in the British standard 7991:2001 [86]. With \( F \) and \( N \), the corrected form of Equation 9 is:

\[
G_{\text{I}} = \frac{F^2 a^2}{EI} \frac{F}{N}
\]

\[
G_{\text{II}} = \frac{3F_n^2 a^2}{4EI} \frac{F}{N}
\]

It is important to understand that \( F \) and \( N \) account for the nonlinearities of the specimen itself, but in tests performed using the dual-actuator load frame, there is an additional source of nonlinearity that has been presented in this paper: the rotation of the actuators illustrated in Figure 24. This rotation causes a moment variation, as shown in Equation 13. The results illustrated in Figure 27 and Figure 28 show the combination of the nonlinearities coming from foreshortening and force rotation and give an overall idea of how these nonlinearities affect the results of fracture tests. Now, a development of
Equation 15 for practical use is proposed and, for the nonlinearity given by the actuator rotation, another factor is herein evaluated. The analysis that is developed herein neglects the presence of the loading end-blocks, assuming instead that loads are introduced through the midplane of the adherends.

Since the beam foreshortening has already been accounted for by the factor $F$, the new factor considers only the force rotation variation. Consequently, Figure 24 is still a valid representation of the DCB specimen, but the value of the foreshortening $\delta$ has to be considered equal to 0. Thus, the moments at the crack tip can be described as

$$\frac{M}{M_{\text{nom}}} = \frac{F_h \cdot a - F_v \cdot \Delta}{F \cdot a} \quad \text{Mode I}$$

$$\frac{M}{M_{\text{nom}}} = \frac{F_h \cdot a + F_v \cdot (\Delta - \Delta_{\text{up}})}{F \cdot a} \quad \text{Mode II}$$

With reference to Figure 24, the two components of the force can be calculated with trigonometry as functions of the imposed displacement $\Delta$, the distance between the two pinpoint $2D$, and the beam foreshortening $\delta$ \(^4\), as shown in Equation 17.

$$F_h = \frac{1}{\sqrt{1 + \left(\frac{\delta}{D - \Delta}\right)^2}}$$

$$F_v = \frac{\left(\frac{\delta}{D - \Delta}\right)}{\sqrt{1 + \left(\frac{\delta}{D - \Delta}\right)^2}}$$

Substituting the terms of Equation 17 in the first of Equation 16, performing some simplifications, the factor $M/M_{\text{nom}}$ for mode I loading becomes:

\(^4\) The parameter $F$ takes into account the beam foreshortening as a reduction of the force arm. Here the beam foreshortening is accounted only in the actuator rotation.
\[
\frac{M}{M_{\text{nom}}} = \frac{a - \Delta \left( \frac{\delta}{D - \Delta} \right)}{a \cdot \sqrt{1 + \left( \frac{\delta}{D - \Delta} \right)^2}} = \frac{1 - \Delta \left( \frac{\delta}{D - \Delta} \right)}{\sqrt{1 + \left( \frac{\delta}{D - \Delta} \right)^2}} \tag{18}
\]

The correction factor for \( G_I \) that takes into account the nonlinearity of the rotation of the actuators is equal to the second power of the right hand term of Equation 18, because \( G \) is proportional to the second power of the applied load. Thus, the correction factor for mode I loading is equal to:

\[
T = \left[ \frac{1 - \Delta \left( \frac{\delta}{D - \Delta} \right)}{1 + \left( \frac{\delta}{D - \Delta} \right)^2} \right]^2 \quad \text{Mode I} \tag{19}
\]

In particular, for the dual-actuator \( D = 700 \text{ mm} \) and, \( \delta \) being a function of \( \Delta \) and crack length \( a \), the values of \( T \) can be calculated and are written in Table 1.
Table 1: Values of coefficient $T$ for mode I loading in the dual-actuator ($D = 700$ mm).

<table>
<thead>
<tr>
<th>$a$ [mm]</th>
<th>$\Delta$ [mm]</th>
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</thead>
<tbody>
<tr>
<td>10</td>
<td>0.998</td>
</tr>
<tr>
<td>20</td>
<td>0.999 0.995</td>
</tr>
<tr>
<td>30</td>
<td>1.000 0.998 0.992 0.977</td>
</tr>
<tr>
<td>40</td>
<td>1.000 0.999 0.996 0.990 0.977</td>
</tr>
<tr>
<td>50</td>
<td>1.000 0.999 0.998 0.994 0.987 0.975</td>
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<tr>
<td>60</td>
<td>1.000 1.000 0.998 0.996 0.992 0.984 0.973 0.952</td>
</tr>
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In Table 1 the values of $T$ for $\Delta$ close or larger than $a$ are not calculated, since these configurations are not physically possible. It should be noted that the values of the factor $T$ are quite close to unity, except for the cases of large displacements $D$ and relatively short crack lengths.
For mode II loading the evaluation is, also in this case, more complicated. With a similar substitution of the terms of Equation 17 in the second of Equation 16, the factor $M/M_{nom}$ for mode II loading becomes:

$$\frac{M}{M_{nom}} = \frac{1 + \frac{\Delta - \Delta_{tip}}{a} \left( \frac{\delta}{D - \Delta} \right)}{\sqrt{1 + \left( \frac{\delta}{D - \Delta} \right)^2}}$$

Equation 20 differs from Equation 18 because of the sign “+” between the two terms in the numerator and because of the presence of the factor $\Delta_{tip}$. The correct evaluation of $\Delta_{tip}$ is not trivial because the specimen does not have constant bending stiffness. An approximation is here assumed and shown in Equation 21, where $L$ is the length of the specimen:

$$\frac{\Delta_{tip}}{\Delta} \equiv \left( \frac{L - a}{L} \right)^3$$

Thus, substituting Equation 21 in Equation 20 and considering that also for mode II $G$ is proportional to the second power of the applied load,

$$T = \left\{ \frac{1 + \frac{\Delta}{a} \left( \frac{\delta}{D - \Delta} \right) \left[ 1 - \left( \frac{L - a}{L} \right)^3 \right]}{1 + \left( \frac{\delta}{D - \Delta} \right)^2} \right\}^2$$

In this case the length of the specimen must also be considered. Choosing for $L$ the value 220 mm that is typical for specimens tested on the dual-actuator, the values of $T$ can be calculated and are written in Table 2.
Table 2: Values of coefficient $T$ for mode II loading in the dual-actuator ($D = 700$ mm, $L = 220$ mm).

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For mode II loading in the dual-actuator, the factor $T$ is always very close to the unity, thus the correction is minimal.

The presence of loading end-blocks has been neglected during the analysis. Considering that the dimensions of the end-blocks are small compared to the dimensions of the usual specimens and load frame, and expecting the rotation of the actuators to be minimally influenced by the presence of end-blocks, one can realistically assume in practice that the parameter $T$ is not affected by the presence of end-blocks. Thus, considering the nonlinear effect associated with the rotation of the actuators, a generalized formulation of Equation 15 for tests performed on a dual-actuator load frame is suggested in Equation 23. The values of the coefficients $F$ and $N$ can be found in [118], and the values of $T$ can be calculated with Equation 19 for mode I loading and Equation 22 for mode II loading.
\[ G_1 = \frac{F_i^2 a^2 F}{EI N} \]
\[ G_\Pi = \frac{3F_i^2 a^2 F}{4EI N} \]

3.10 Variability of the mode-mixity angle

There are advantages to conducting fracture tests in displacement control, since this facilitates stable crack growth. Nevertheless, the choice of constant displacement rate control in the tests performed with the dual-actuator leads to a value of mode-mixity angle \( \Psi \) that changes as the crack length grows. In fact, starting from the definition of Equation 10 and beam theory, the angle of mode-mixity \( \Psi \) can be expressed as different combinations of applied forces, displacements and crack length, as shown in Equations 24 and 25.

\[ \psi = \text{ArcTan} \left( \frac{F_R - F_L}{F_R + F_L} \sqrt{\frac{3}{4}} \right) \]

\[ \psi = \text{ArcTan} \left( \frac{2 \sqrt{3} a^3 (a + x_t) \Delta_L - \Delta_R}{(3a^3 + L^3) (a + x_t) \Delta_L + \Delta_R} \right) \]

The angle of mode-mixity is completely defined by the applied forces as expressed in Equation 24. Equation 25 shows that, applying displacement ramps of constant slope, thus having a fixed ratio \( \Delta_L/\Delta_R \), the angle of mode-mixity varies as a function of crack length \( a \). In particular, the angle of mode-mixity grows during the test as the crack propagates. Some trends of mode-mixity change are shown in Figure 31.
Figure 31: Angle of mode-mixity as function of crack length. The numbers in the legend represent different nondimensional imposed displacement rates.

The change of angle of mode-mixity usually permits covering a range of mode mixities during a single typical test. However, some concerns regarding the development of a stable process zone for the stresses ahead of the crack tip may arise, since the angle of mode-mixity continuously changes during the test [82, 120]. Thus, a well-defined value of mode-mixity $\Psi$ may be needed. In this case Equation 25 can be used and the ratio between the right and left displacements can be written as a function of the crack length and of a value of $\Psi$ to be tracked with Equation 26.

$$\frac{\Delta_R}{\Delta_L} = \frac{1 - \frac{(3a^3 + L^3) \tan(\psi)}{2\sqrt{3}a^3}}{1 + \frac{(3a^3 + L^3) \tan(\psi)}{2\sqrt{3}a^3}}$$

In this way, the testing procedure is different than shown in Figure 22, since the second part of the test consists of one displacement ramp for one of the beams (e.g. $\Delta_L$ proportional with time) and the displacement for the other beam calculated with the function of Equation 26. The advantages of having two phases of the tests performed...
with mode I loading remain also with this procedure, thus the test procedure is shown in Figure 32 for the case of a stable crack growth and continuous update of crack length.

![Figure 32: Testing procedure for mode-mixity tracking.](image)

### 3.11 Conclusions

The dual-actuator test frame simplifies the experimental effort required for mixed-mode characterization of adhesively bonded and laminated beam specimens, since a single geometry type of specimen can be used for spanning from pure mode I to pure mode II loading conditions. The dual-actuator introduces the possibility of easily applying different levels of mode-mixity on a single specimen, thus allowing application of some of the positive outcomes of traditional data analysis techniques such as the corrected beam theory (CBT) and the experimental compliance method (ECM) also in mixed-mode tests. The results of CBT can be especially useful in mixed-mode testing and a procedure for increasing the efficiency of each test has been proposed.

The construction characteristics of the testing frame introduce some nonlinear effects such as adherend foreshortening and force rotation during the test. These variations were evaluated analytically and can be accounted for in real tests. In particular, two representative cases of bonded aluminum and bonded carbon composite were described. It has to be pointed out that a problem such as the beam foreshortening is common to most of the techniques that measure fracture properties with DCB type specimens. For the dual-actuator load frame it was shown that the nonlinear geometric effects can
account for a 1% variability of the results when testing relatively stiff materials, such as bonded metals, and can have a larger influence in relatively compliant specimens, such as DCBs obtained from thin layers of carbon fiber composites. Graphs obtained in this paper can be directly used in testing practice with the dual-actuator as correction factors for the mentioned nonlinear effects.

Then, a generalized parameter $T$, accounting for the nonlinearities imposed by the dual-actuator load frame and not anticipated in the classical papers of Williams, was evaluated. This parameter addresses the effects of the rotation of the actuators during mode I and mode II tests and can be used along with the parameters $F$ and $N$ associated with the beam foreshortening and end block stiffening. The computation of the parameter $T$ can easily be achieved in practical cases with equations or look-up tables that are presented in the paper.

### 3.12 Acknowledgments

The authors would like to acknowledge the financial support provided by the National Science Foundation under contract DMR-0415840 for constructing the dual-actuator load frame and by the Wood-Based Composites Center and the Sustainable Engineered Materials Institute at Virginia Tech for supporting the project carried on with the dual-actuator load frame. We also acknowledge the use of facilities in the Engineering Science and Mechanics Department and the interdisciplinary environment fostered by the Macromolecules and Interfaces Institute at Virginia Tech.

We are also grateful to Dr. Hitendra Singh who developed the comparison in Figure 21, and Professor Charles Frazier for helpful discussions.
Chapter 4

Determining adhesive fracture energies in wood double cantilever beam (DCB) specimens: revisiting traditional methods

4.1 Abstract

An analytical approach for investigating the results of fracture tests of adhesively bonded double cantilever beams (DCB) is presented. Motivated by fracture tests involving bonded wood adherends, the analysis focuses on layered beams in which the layers are not parallel to the faces of the beams but are inclined out of the bond plane. When such beams are bonded to form DCB specimens, the geometric and material properties of the layers influence the adherend compliance and the calculated fracture properties of the bonded specimens. The study addresses wood in particular, because grain orientation and earlywood/latewood spacing influence the stiffness characteristic of the beam adherends typically used for DCB specimens. Available data of earlywood and latewood for Douglas fir are used to calculate the equivalent bending stiffness along the length of the beam, which depends on the local bending stiffnesses of the different layers. The orthotropic mechanical properties of the individual layers of earlywood and latewood are also considered.

The behavior of bonded DCB specimens made with such layered materials are analyzed with common methods for mode I fracture testing: simple beam theory (SBT), corrected beam theory (CBT), experimental compliance method (ECM or Berry method),

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and area method (AM). In particular, the first three methods are described in the BS 7991 and ASTM D 3433 standards, while the AM is not suggested by the mentioned standards. SBT, CBT and ECM, although initially developed for characterizing bonds between uniform and isotropic adherends, have also been commonly applied by researchers to other materials, wood included. Nevertheless, these three standardized methods lack precision for determining the critical strain energy release rate, $G_{lc}$, when applied to bonded wood, due to the elastic stiffness variability that occurs along the length of the bonded beams. The area method shows higher precision, although its applicability is not always practical.

Depending on grain orientation, the variability of equivalent elastic stiffness for Douglas fir is found to be on the order of ±6-8% for typical configurations applied to wood DCBs. In the other cases representative for bonded wood beams that are presented, the variability is on the order of ±15-20%. The SBT, CBT and ECM tend to interpret such adherend stiffness variability as $G_{lc}$ variability, thus returning incorrect results. In the developed analytical procedure, the AM yields more coherent results, although practical issues can limit its reliability with experimental results. Another physical problem that arises with the adherend stiffness variation is the onset of a mode II loading component that is not anticipated, nor accounted for, in the traditional data analysis methods.

### 4.2 Keywords

Adhesive joints, bonded wood, fracture mechanics, double cantilever beam, mode I, beam theory, stiffness variability, layered material, grain orientation, growth ring, earlywood, latewood, composite laminates.

### 4.3 Introduction

Since the early works of Mostovoy, Ripling et al. [20, 121, 122], fracture mechanics has become a very common means for mechanical characterization of adhesive joints. The fracture resistance is characterized by the critical strain energy release rate, $G_c$, which is the amount of energy per unit fracture area that is required to extend a pre-existing crack. $G$ is defined, as shown in Equation 27, as the infinitesimal variation of $U$, the
potential energy, for an infinitesimal growth of $A$, the crack area. $G_c$ is associated to the condition at which the crack grows.

$$G = -\frac{dU}{dA}$$

Assuming that force and displacement are linearly related, and focusing on mode I loading, one can obtain the equivalent compliance formula in Equation 28, where $G_c$ is indicated as $G_{lc}$ and is a function of the geometrical parameters: $b$, the width of the specimen, and $a$, the crack length, as well as the critical value of load at which crack growth occurs, $P_c$, and the compliance of the specimen, $C$. Equation 28 shows that $G_{lc}$ depends only on the variation of compliance associated with crack growth and is independent of the stiffness of other parts of the specimen or testing frame.

$$G_{lc} = \frac{P_c^2}{2b} \frac{dC}{da}$$

One of the most common geometries used for fracture characterization of adhesively bonded specimens is the double cantilever beam (DCB), which consists of two (often equivalent) beams bonded together. During the test, one of the beams is connected to the fixed clevis of a loading machine and the other is pulled by the machine actuator. The connections are obtained by means of loading pins passing through a hole drilled in each beam or in an attached mounting block. This loading condition results in an applied fracture mode that is nominally mode I if the adherends are symmetric. Another geometry that is sometimes considered is the tapered double cantilever beam (TDCB), which offers certain advantages since $dC/da$ is nominally constant during the test, but requires precise construction and calibration [115]. Tests of DCB and TDCB specimens can be performed on a variety of materials without particular restrictions on the material choice, as long as the beams are not damaged or do not yield during the test. The testing methods involving these two specimen geometries have been thoroughly examined by various researchers [22, 90], and also BS 7991 and ASTM D 3433 standards [86, 87] cover the subject. These standards describe the experimental and analytical methods for the determination of the fracture resistance of adhesively bonded DCB and TDCB specimens subjected to mode I loading conditions. In particular, the standards have been applied to bonded wood specimens [89, 116].
Wood is commonly described as an orthotropic material from a mechanical standpoint [26, 27]. Structural heterogeneities, porosity and hygroscopic nature are also factors that influence the mechanical properties of this natural material. With an impressive amount of work, Ebewele et al. [102, 103] showed that fracture properties in bonded wood are affected by a number of factors, such as the wood structure, the adhesive penetration into the wood and the wood surface roughness. Given the influence of a large number of factors and the fact that wood is a natural material, it is commonly found that fracture measurements taken on wood and bonded wood are characterized by data spread that is usually larger than obtained when testing more uniform adherends [88, 89, 102, 103, 116]. In particular, Triboulot [36] indicated with experimental and numerical studies that the high variability in wood properties requires researchers to deal with extensive statistical analysis when critical fracture toughness values are to be evaluated. This intrinsic variability of properties is an issue that forces engineers to design wood applications with large safety factors, thus limiting the efficiency of the constructions.

The present paper evaluates the effects that some of wood’s intrinsic characteristics have on the results obtained for fracture analysis of bonded DCB specimens. The variability of fracture properties that is usually encountered in bonded wood specimens has often been associated with the variations of properties that in a natural material are difficult to detect, but there are also morphological aspects that can possibly be accounted for. The orthotropic nature of the material and the presence of alternating earlywood and latewood layers, for example, can be modeled as intrinsic causes of variability given by the morphology of the material. This paper determines how the grain orientation can alter the values of critical strain energy release rate measured with the approaches described by the already mentioned standards BS 7991 and ASTM D 3433. This is performed by analyzing layered DCB specimens, revealing outcomes that highlight some of the weaknesses of traditional methods for evaluating $G_{IC}$ in bonded systems.

### 4.4 Common analysis methods for mode I fracture

Equation 28 is seldom used directly for evaluating $G_{IC}$. The BS 7991 standard suggests three methods for evaluating $G_{IC}$ in DCB and TDCB specimens. These methods
are the simple beam theory (SBT), the corrected beam theory (CBT) and the experimental compliance method (ECM). The ASTM D 3433 standard only covers the SBT. The methods are thoroughly described in the standards and by Blackman et al. [123]. In a DCB tested in mode I, two equal forces are applied at the ends of the two beams, opening the crack; the crack length, usually indicated as $a$, is measured between the crack tip and the line of action of the applied loads. Tests are usually run in displacement control to avoid unstable crack growth, and values of applied force, displacement and crack length are recorded throughout the test.

With SBT, the term $dC/da$ of Equation 28 is computed assuming that the debonded regions of the two adherends deform as beams perfectly fixed at the section corresponding to the crack tip. SBT, as implemented by the standards [86, 87], includes a correction factor taking into account the thickness of the beam, but does not consider other effects such as the adhesive layer deformation. CBT and ECM approaches overcome these problems by evaluating the actual compliance characteristics of individual specimens and fitting the compliance versus crack length relationship. In the CBT a linear fit is imposed on the data points when the cube root of the compliance is plotted against the crack length, effectively recognizing the dependence of the deflection of a point-loaded cantilever beam on the cube of the free length. In the ECM, a power law relationship is assumed by fitting a straight line through data points of the logarithm of the compliance vs. the logarithm of the crack length. SBT and CBT assume that the elastic characteristics of the adherend and adhesive materials are independent of the crack length. The accuracy of this assumption is deemed to be confirmed by the coefficient of determination, $R^2$, of the CBT and ECM data fit. The $R^2$ value obtained experimentally for CBT and ECM is often very near unity, even in materials with intrinsic variability such as wood [88, 89, 113], masking any possible effects of adherend variability. Nevertheless, $G_{ic}$ data for bonded wood specimens are often characterized by larger scatter [36, 88, 113] than seen for uniform adherends, raising a question about errors introduced by methods recommended in the standards that were developed for uniform properties. Other possible source of data scatter can be recognized in the variability of adhesive layer thickness, as described in [124].
4.5 Wood as an engineering material

All wood is structurally composed of basic units, which are long and narrow cells aligned with the trunk, forming the characteristic wood grain. From a chemical point of view, wood is composed of three basic components: cellulose, hemicelluloses and lignin. Some other components such as gums, resins and waxes may be present in small quantities and usually do not influence the mechanical properties of the wood significantly [3]. Focusing on macroscopic mechanical characteristics, wood within a tree has strong cylindrical symmetry. Thus wood may be described as an orthotropic material, with the principal axes of the stiffness matrix associated with the grain orientation, where the longitudinal (L) axis is parallel to the grain, the radial (R) axis is perpendicular to the growth ring and grain, and the tangential (T) axis is tangent to the growth ring and perpendicular to the grain. The mechanical properties associated with the three directions usually differ considerably from each other [3]. The orthotropic nature of wood is also the result of the two alternating layers that form wood, earlywood and latewood. In most of the temperate climes, the growing season for trees produces annual growth rings. The inner part of the ring is created at the beginning of the growing season and is called earlywood or springwood, while the outer part is called latewood or summerwood. Earlywood is characterized by cells with relatively large pores, usually a lighter color and a relatively low material density. Latewood has cells with thicker walls, greater material density and consequently higher strength. The difference in density between earlywood and latewood is an important factor influencing the mechanical properties of wood along the different directions [125].

Ebewele [102] found that the grain orientation also influences the value of $G_{ic}$ measured in bonded wood specimens. Moreover, due to the usual low fracture toughness along the radial direction, tests on fracture properties of bonded wood require specially prepared specimens to facilitate crack propagation within the bond layer and not along the wood grain. A useful approach for limiting these issues is to maintain the grain orientation slightly tilted in reference with the bondline, as done by Gagliano and Frazier [89]. Experimental practice on yellow-poplar [126] and southern pine [127] has shown that keeping the wood grain orientation between $3^\circ$ and $6^\circ$ with respect to the intended
bond plane, as illustrated in Figure 33, prevents the crack from moving into the adherends in tests conducted in mode I.

![Figure 33: DCB specimen and grain orientation in the beams.]

For a formal investigation of bonded wooden DCB specimen behavior, wood was modeled as a layered material comprised of layers of two materials, earlywood and latewood, with different properties. The two layers themselves are orthotropic [29]. The elastic behavior of the beams, in terms of the force-displacement relation during bending, depends also on the layer orientation, the relative thickness of the layers and their elastic moduli [99]. The grain orientation in a wood beam has a three-dimensional pattern that is illustrated in Figure 34, where the geometrical orientation of the fibers associated with the longitudinal and the transverse planes of the beam is shown and denoted as $\alpha$ and $\beta$, respectively. Wood is thus generally a layered material with layers oriented out of plane.

![Figure 34: Grain orientation in a wood beam used for DCB specimen adherend. The white parts represent the earlywood layer, the black parts the latewood layer. The shaded area indicates the portion of the beam to be bonded.]

4.6 **Effect of grain orientation: longitudinal plane (angle $\alpha$)**

The disposition of alternate layers of earlywood and latewood at an angle $\alpha$, as seen in the face parallel to the plane $xz$ of Figure 34, has an effect on the distribution of the different materials in the sections of the beam that are parallel to the plane $yz$, as shown in Figure 35. In fact, given the elastic modulus difference between earlywood and latewood, the different relative positions of the two materials in the sections leads to a stiffness $EI_{EQ}(x)$ that is also variable. The term $EI_{EQ}(x)$ is “variable” since it is a function
of the position $x$ of the section. It is “equivalent” since it indicates the local $EI$ of a section of a uniform isotropic beam with the same stiffness. The geometry of the problem is illustrated, with exaggerated proportions, in Figure 35, where the letters L and R indicate the longitudinal and radial directions as already indicated in Figure 34. On the bottom left part of Figure 35 the view of the corresponding lateral face of a southern pine beam (plane $xz$) is shown to illustrate a typical adherend.

![Figure 35: Grain orientation along longitudinal dimension of a wood beam, schematic and picture](image)

The variable relative position of the two layers not only affects the distance of the respective layers from the neutral axis but also influences the position of the neutral axis of the section itself, which generally becomes not coincident with the middle plane of the beam. A further complication in the evaluation of the term $EI_{EQ}(x)$ comes from the orthotropic nature of earlywood and latewood. In fact, not only do the layered earlywood and latewood make wood an orthotropic material, but also the individual layers are orthotropic materials; the orthotropic nature of the earlywood and latewood is expected, given the anatomy of the wood cells. Studies investigating directional mechanical properties of latewood and earlywood are much less common than similar studies on solid wood, probably given the fact that performing mechanical tests at the growth ring scale, or less, is not easy. Nevertheless, a useful description was given by Gibson and Ashby [125]. They described how elastic moduli of different cellular materials are influenced by the densities of these materials. The influence is generally non-linear and depends on the direction that is considered. An explicit model of earlywood and latewood was developed for Douglas fir by Bodig and Jayne [128], while some experimental work was performed by Jernkvist and Thuvander [129], who provided some qualitative estimates of the directional properties of earlywood and latewood in Norway.
spruce. Finally, combining assumptions from Gibson et al. [125, 130, 131] and Moden [132], Nairn developed a set of directional properties for earlywood and latewood for Douglas fir [29]. The data indicated by Nairn are utilized herein to describe the apparent engineering elastic modulus along direction $x$, using the formula normally applied to composite materials in Equation 29 [133]. Note that this equation has to be written for both earlywood and latewood.

$$\frac{1}{E_x} = \frac{1}{E_L} (\cos \alpha)^4 + \left( \frac{1}{G_{LR}} - \frac{2\nu_{LR}}{E_L} \right) (\sin \alpha)^2 (\cos \alpha)^2 + \frac{1}{E_R} (\sin \alpha)^4$$  \hspace{1cm} 29$$

Considering the numerical data of elastic modulus and Poisson’s ratio indicated by Nairn [29] and estimating the values of $G_{LR}$ equal to 2490 and 580 MPa for earlywood and latewood, respectively, the normalized moduli for the two layers are shown in Figure 36. An interesting aspect of the two curves is that they do not scale. This means that by changing the angle between the longitudinal axis of the layers and the axis of the beam, the relative stiffness of the two materials along the direction of the beam length varies.

Figure 36: Normalized moduli along x-axes for earlywood (dashed) and latewood (continuous)

The variability of $EI_{EQ}$ along the beam length was investigated by evaluating $EI_{EQ}(x)$ as a function of the ratio between the longitudinal elastic modulus values of earlywood and latewood, the ratio between the thicknesses $t$ of the layers and the grain orientation angle $\alpha$. The model used for the beam is illustrated in Figure 37, which represents a lateral view of the beam, parallel to plane $xz$ in Figure 34. In Figure 37 the thick lines represent the boundaries of the layers, the dashed lines the midplane of the individual layers and the thin line the position of the section neutral axis. Figure 37 represents a unit of beam that, if the grain orientation angle $\alpha$ is constant, is repeated along the entire length of the beam. The length of this unit is equal to $L$ and the thickness of the beam is
the graph plots the geometry as \( z/h \) vs. \( x/L \). Each interface and middle portion of the layers was described with equations that are limited within the interval 0 to 1 both in \( x \) and \( z \) directions.

\[ z/h \]

\[ x/L \]

**Figure 37: Wood beam model along longitudinal direction from Mathematica®**

The procedure for evaluating \( EI_{EQ} \) did not use classical lamination theory (CLT), but was based on plane stress assumptions. This choice was dictated because the orientation of the layers is not parallel to the beam edge. A comparison of the two methods is presented later in this paper to illustrate the effect of plane stress versus plate bending models. The analysis was performed by developing a Mathematica® (Wolfram Research, Champaign, IL) code. For this model and hereafter in this paper, the latewood will be referred to as material 1 and will be the thinner and stiffer layer; the earlywood will be referred to as material 2, and it will be the thicker and softer layer. Once the geometrical properties \( \alpha, t_1, \) and \( t_2 \) and the elastic curves \( E_x/E_L \) for both of the materials are known, the position of the neutral axis can be determined as a weighted average of the neutral axis positions of the sectioned layers. The weight is given by the modulus \( E_x \) of the material of the sectioned layers. After this, the value of \( EI_{EQ} \) in each section was calculated similarly as the second moment of area of stacked rectangular areas multiplied by the corresponding elastic modulus \( E_x \) as shown in Equation 30, where the Sections term indicates the number of layers that are sectioned at the given coordinate \( x \), \( A_i \) the area of each layer section (parallel to axis \( z \) and considering a unit width of the beam), \( I_i \)
the second moment associated with the center of each layer section, \( d \) the distance between the center of each section and the neutral axis and, finally, \( E_{x,i} \) the elastic modulus along \( x \) for the material of the layer.

\[
EI_{EQ}(x) = \sum_{i=1}^{\text{Section}} \left[ (A_i d_i^2 + I_i) E_{x,i} \right]
\]

The model presented in this paper did not consider any deformation effects coming from shear, although these can be important especially in materials where the shear modulus is considerably smaller than the axial modulus. The developments and analysis that follow in this paper are consistent with the choice of neglecting the shear deformation, since shear deformations were not considered in all the models that were developed.

For Douglas fir, the \( EI_{EQ}(x) \) curves for different angles \( \alpha \) are shown in Figure 38. These results refer to a typical beam used in DCBs, with thickness along the \( z \) axis equal to 10 \( mm \) and with thickness of earlywood 2.1 \( mm \) and latewood 1.4 \( mm \) [29], thus resulting in a volume ratio of latewood vs. earlywood equal to 0.75. In these plots the results were normalized by the value of the longitudinal elastic modulus of solid wood, being obtained with the rule of mixtures from the longitudinal elastic moduli of earlywood and latewood. Also, the analysis proceeds on a per unit width basis with respect to the \( y \) axis. Note that in the graph associated with \( \alpha=3^\circ \), the value of \( EI_{EQ} \) can locally be slightly larger than the value of the longitudinal \( EI \) of solid wood, herein indicated with \( EI_{WOOD,L} \). This counter-intuitive result is justified by the variable relative position of the two layers and the neutral axis and by the fact that, given the geometric characteristics of the layers, in some sections the stiffer latewood is present with a volume ratio larger than the average 0.75 used for the determination of solid wood longitudinal stiffness. For higher values of \( \alpha \), the values of \( EI_i \) of the two layers decrease rapidly and so \( EI_{EQ} \) is always lower than \( EI_{WOOD,L} \).
Figure 38: Variation of $EI_{EQ}$ along Douglas fir beam with different angle $\alpha$.

For generalizing the approach, generic plots are presented from Figure 39 to Figure 41. The plots are nondimensionalized and consider values of the elastic moduli calculated with Equation 29, having fixed the parameters $\nu_{RL}=0.05$ and $G_{RL}/E_L=0.065$ for both of the materials and $E_{R1}/E_{L1}=0.1$ and $E_{R2}/E_{L1}=0.08$. The values of $t_1$, $t_2$, $t_1$ and $E_{L2}/E_{L1}$ are variable and not associated with particular wood species, although representative of limiting cases of real wood beams used in DCBs. Although the orthotropic nature of
the individual layers has a role and was considered, the variation of $EI_{EQ}$ primarily results from having two layered materials forming an angle with the longitudinal edge of the beam and consequently affecting the relative positions of the layers in different sections and the position of the neutral axis that changes along the beam.

$\alpha=3^\circ$, $t_1/h=0.055$, $t_2/t_1=4$, $E_{L2}/E_{L1}=0.2$

$\alpha=6^\circ$, $t_1/h=0.055$, $t_2/t_1=4$, $E_{L2}/E_{L1}=0.2$

$\alpha=3^\circ$, $t_1/h=0.065$, $t_2/t_1=4$, $E_{L2}/E_{L1}=0.2$

Figure 39: Variation of EI$_{EQ}$ along beam length for $t_2/t_1=4$ and $E_{L2}/E_{L1}=0.2$. 
\[ \alpha = 3^\circ, \ t_1/h = 0.055, \ t_2/t_1 = 4, \ E_{L2}/E_{L1} = 0.5 \]

\[ \alpha = 6^\circ, \ t_1/h = 0.055, \ t_2/t_1 = 4, \ E_{L2}/E_{L1} = 0.5 \]

\[ \alpha = 3^\circ, \ t_1/h = 0.065, \ t_2/t_1 = 4, \ E_{L2}/E_{L1} = 0.5 \]

Figure 40: Variation of $EI_{EQ}$ along beam length for $t_2/t_1 = 4$ and $E_{L2}/E_{L1} = 0.5$. 
The graphs show that the variability of $EI_{EQ}$ can be quite large, being on the order of 30-40% for the cases with $t_2/t_1 = 4$ and $E_{L2}/E_{L1} = 0.2$. The variation of the parameter $t_1/h$ seems to significantly influence the shape of the $EI_{EQ}$ curve. On the other hand, the variation of angle $\alpha$ between the imposed values $3^\circ$ and $6^\circ$ seems to have little effect on the results.
4.7 Effect of grain orientation: transverse plane (angle $\beta$)

In the previous section the effects of grain orientation along the plane $xz$ of Figure 34, described by angle $\alpha$, were investigated. The effects of grain orientation along plane $yz$, described by angle $\beta$ in Figure 34, were also considered in the study. This orientation is generally not controlled when beams are used for producing DCB specimens, since angle $\beta$ is not a factor limiting the crack from propagating within the beam or with other practical implications on DCB testing as for $\alpha$ [89]. Nevertheless this orientation can affect the bending of the beam. Since the sections are generally non-symmetric in terms of elastic properties, the position of the center of torsion of each section generally differs from the geometrical centroid, which has coordinates $(b/2, h/2)$, given that each section has a base width $b$ and height $h$. For the same reason the principal axes of bending for each section are also not parallel to the Cartesian axes $y$ and $z$. The approach in this case was similar to the one followed in the previous section. The graph of Figure 42 plots the geometry as $y/b$ vs. $z/h$, for a section.

![Graph](image_url)

**Figure 42: Section of wood beam from Mathematica®**

Running the analysis for a beam of section 20 x 10 mm and for Douglas fir, the results showed very small variability of the center of torsion position and principal axes orientations: the center of torsion resulted at a distance of $(0.0005 \, b; \, -0.002 \, h)$ from the geometrical center and the principal axes were rotated $0.5^\circ$ from the horizontal and vertical directions. Additional evaluations were performed with a nondimensional approach, but did not lead to much larger numerical results. The effects of grain
orientation on the \(yz\) plane were therefore deemed as minimal and are not considered further in this paper.

4.8 Comparison of the proposed method with classical lamination theory

The method that was presented and applied in the two previous sections was based on plane stress assumptions and addressed layered materials with layers inclined out of plane. The method proposed above, hereafter referred to as the inclined layer method (ILM), may appear simplistic in a field of layered material mechanics, where well known approaches, such as classical lamination theory (CLT), are usually applied. CLT was not used because the angles \(\alpha\) and \(\beta\) complicate the CLT solution. One aspect that limits the direct applicability of the CLT to the geometry with oriented layers is the fact that CLT assumes that no stresses are transmitted between the layers, in the direction perpendicular to the layers. This assumption is based on the way in which traditional fiber composites are used, such as beams, plates and cylinders with one dimension negligible when compared to the other two. In these configurations three out of six components of stress are negligible [133]. In all the calculations, therefore, the stress components perpendicular to the layers are assumed to be zero. This means that no stresses are transmitted between the different layers. Traditionally, the different layers are also parallel to each other and perpendicular to the out of plane axis. When the layers are oriented at an angle \(\alpha\), as in a normal wood beam, the hypothesis of no stress associated with the direction perpendicular to the layers cannot be totally satisfied if, for example, the beam is subjected to bending.

For comparison purposes with ILM, an analysis with CLT was also developed. Considering that the grain orientation is usually small (3 to 6\(^\circ\)), multiple CLT solutions were developed at different sections of the layered beam along the \(x\) axis (Figure 37). At each section the correct position and thickness of the layers was computed; the grain orientation influences the position of the layers in the different sections, but in each section the CLT does not consider other effects coming from grain orientation. Due to the fact that the number of layers changes along the \(x\) axis, the CLT solution was developed for a particular case for comparison purposes. This comparison was also
based on the data of Douglas fir provided in [29] and assumed the values of $G_{LT}$ equal to 2490 and 580 MPa for earlywood and latewood, respectively. The comparison was performed, considering a laminate of layers of earlywood, with a thickness 2.1 mm, alternated with layers of latewood of thickness 1.4 mm [29], with an angle of grain orientation of 6° and a total thickness of the beam of 10 mm.

Using the common nomenclature of the CLT illustrated, for example, in [133], all the layers have orientation 0°, meaning that the longitudinal orientation of the fibers is parallel to the axis $x$ of the lamina\(^6\). The terms for the comparison are the $EI_{EQ}$ for the ILM and the $D_{11}$ element of the laminate stiffness matrix $\begin{bmatrix} A & B \\ B & D \end{bmatrix}$.\(^7\) With reference to the same nomenclature [133], the bending stiffness that was considered is the one associated with the axis $x$, that in this case is also the longitudinal orientation of the fibers.

The results of the comparison are plotted in Figure 43, where both are normalized to the longitudinal bending stiffness of Douglas fir listed in [29]. The continuous line is $EI_{EQ}$, while the points represents the $D_{11}$ from CLT solutions of the problem repeated at steps of position $0.1x/L$ along the beam. As earlier in the present paper, $L$ is the length of the unit of beam that, if the grain orientation angle $\alpha$ is constant, is repeated along the entire length of the beam.

\(^6\) Note the value 0° is not referred to $\alpha$ and $\beta$. It indicates the angle that, in a lamina of composite material with matrix and oriented fibers, the fibers have with axis $x$, as seen on the plane xy (Figure 34)

\(^7\) The $\begin{bmatrix} A & B \\ B & D \end{bmatrix}$ matrix relates the applied generalized loads vector $F$ to the generalized deformation vector $\varepsilon$. $\begin{bmatrix} A & B \\ B & D \end{bmatrix} (\varepsilon)$
The graph in Figure 43 illustrates that the variations of bending stiffness evaluated with ILM and CLT are similar in shape and amplitude. The fact that the two methods return very similar results can be better understood considering two aspects of wood nature. First, the fiber orientation in earlywood and latewood has to be the same, whereas in composites the different layers usually have different fiber orientations. This means that the direction of maximum stiffness is the same for all the layers in the laminate that was considered for the CLT. Second, the value of $\nu_{TL}$ is equal to 0.041 for both earlywood and latewood [29]. The fact that the Poisson’s ratio is relatively small and has the same value for both of the materials minimizes the difference of results that are obtained with the two methods and would limit the utility of the CLT approach also in the configuration $\alpha=0^\circ$, since the effect that the lateral deformation of one lamina (layer) has on the contiguous ones is minimal.

4.9 Applying standardized fracture tests to bonded layered materials

The variability of local elastic stiffness was illustrated in one of the previous sections and presented with Figure 38 to Figure 41. In the material where this variation is most likely to occur, wood, there are other sources of variability, such as knots and cross grains that complicate the $EI_{EQ}$ distribution, introducing further variability. The plots of Figure 38 to Figure 41 have shown that the curve $EI_{EQ}(x)$ depends on a number of factors and has a variability that can reach amplitudes of ±15-20% around a median value. For evaluating how this variation level affects tests in bonded DCB specimens, an analysis
was performed imposing the $EI_{EQ}$ on the two beams of a DCB specimen. For this part of the study, given the large number of variables affecting characteristics and shape of $EI_{EQ}$ and the other sources of variability that were not considered, it was decided to describe the $EI_{EQ}$ with general sinusoidal equations rather than importing the specific shapes of Figure 38 to Figure 41. The curves $EI_{EQ}$ for the two beams were consequently described as in Equation 31.

$EI_{EQ-1}(x) = EI_{MEAN-1} + EI_{VAR-1} \left[ \sin(\omega_1 x) \right]$  

$EI_{EQ-2}(x) = EI_{MEAN-2} + EI_{VAR-2} \left[ \sin(\omega_2 x) + \delta \right]$  

The parameters of Equation 31 are generally different in two beams. In this study it was chosen to keep the value of $EI_{VAR}$ equal to $0.2EI_{MEAN}$, and $\omega$ equal to either $\pi/L$ or $4\pi/L$, thus resulting in half or two complete sinusoidal cycles within the beam length $L$. The phase angle between the $EI_{EQ}$ of the two bonded beams, $\delta$, was chosen to be either 0 or $\pi/2$. The value $EI_{VAR}/EI_{MEAN}$ = 0.2 was chosen based on the amplitude of $\pm$15-20% seen in some of the plots of Figure 39. The combinations of $EI_{EQ}$ in the two beams that were considered are shown in Figure 44.

![Figure 44: Distribution of $EI_{EQ}$ along two beams of the DCB specimen.](image)

For each case, the analysis of the fracture properties for mode I loading applying the SBT, CBT, ECM, and AM approaches was performed. In these analyses, the value of $G_{IC}$ was imposed to be constant, as one would expect if the fracture energy were only a
material system parameter. The crack growth was imposed to be stable throughout the test, with the crack length \( a \) growing from \( L/5 \), pre-crack, to \( L \), complete debonding of the specimen. Also, crack length readings were assumed to be taken at intervals of \( L/100 \) and no correction factors for large displacements or load blocks were considered, as these effects were not included in the computed compliances. The first step of the analyses was the calculation of the term \( dC/da \) of Equation 28, which was performed by calculating the deformed shape of the two beams at each interval of crack reading, integrating the well-known equation of the deformed beam of Equation 32.

\[
y''(x) = \frac{M(x)}{EI_{EQ}(x)} \quad \text{with} \quad y'(a) = y(a) = 0
\]

where \( a \) is the crack length. In Equation 32, the effects of shear deformation are not considered. This assumption was consistent with the model that was proposed for the analysis of the effects of grain orientation on \( EI_{EQ} \), which also neglected any deformations coming from shear. Equation 32 was in this case applied to beams with the variable sinusoidal stiffness illustrated in Figure 44, while the moment \( M(x) \) was the usual linear function for cantilever beams with an applied force. It was possible to numerically solve Equation 32 for the two beams at the different steps of crack length reading. Thus, the compliance of the specimen was evaluated at each step and the value \( dC/da \) was approximated with \( \Delta C/\Delta a \), where \( \Delta a \) corresponds to the interval for crack reading, \( L/100 \). With this procedure, it was possible to calculate the values of \( P_c \) at which the crack was propagating with Equation 28, as a function of \( a \). The values of \( P_c \) were then used in analyses with SBT, CBT and ECM, while AM does not require this input parameter.

**Simple Beam Theory (SBT) Analysis**

SBT approaches the deformation of the beams in a DCB specimen describing the compliance as a function of \( a^3 \) and correcting the relation for the thickness of the beam, \( h \) [122]. The equation that is used is

\[
G_{I_c} = \frac{4P_c^2}{E_S b} \left( \frac{3a^2}{h^2} + \frac{1}{h} \right)
\]
where $E_s$ is the independently-measured flexural or tensile modulus of the substrate. It was reasonable to consider for the presented analysis $E_s$ equal to $E_{\text{MEAN}}$. Assuming also the thickness $h$ of the beams equal to $L/10$ and the specimen width $b$ equal to 1, the values of $G_{lc}$ calculated with SBT are plotted in Figure 45. Depending on the combination of amplitudes and phase angle of $EI_{EQ}(x)$ of the two beams, the results change and do not match the value of $G_{lc}$ imposed by the definition.

<table>
<thead>
<tr>
<th>$\omega=\pi/L$, $\delta=0$</th>
<th>$\omega=\pi/L$, $\delta=\pi/2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$G_{lc-\text{SBT}}/G_{lc}$</td>
<td>$G_{lc-\text{SBT}}/G_{lc}$</td>
</tr>
<tr>
<td>$\omega=4\pi/L$, $\delta=0$</td>
<td>$\omega=4\pi/L$, $\delta=\pi/2$</td>
</tr>
<tr>
<td>$G_{lc-\text{SBT}}/G_{lc}$</td>
<td>$G_{lc-\text{SBT}}/G_{lc}$</td>
</tr>
</tbody>
</table>

**Figure 45: Values of $G_{lc}$ obtained with simple beam theory (SBT) analysis**

Limitations of the SBT are known, since this method underestimates the compliance of the DCB specimens. SBT, in fact, accounts only for the bending deformation of the beams and neglects the deformations that are coming from the adhesive layer. Shear deformations have traditionally been accounted for with the term $I/h$, but the validity of this correction factor has been questioned by Li et al. [134]. The value of the correction factor $I/h$ is small and usually negligible when compared to the term $3a^2/h^3$. The correction factor was also not of primary importance for the analysis herein presented because, being constant, it could not influence $G_{lc}$ variability but only its average value. Another limit of SBT is that this method traditionally relies on values of elastic modulus for the material of the beam that come from sources that are not the DCB test itself. This can possibly introduce inaccuracies, especially when testing materials with variability of
the elastic properties from specimen to specimen, such as wood. For these reasons, SBT is not widely used in fracture analysis of bonded DCBs.

**Corrected Beam Theory (CBT) Analysis**

The CBT differs from SBT since the data of applied force, displacement and crack length that are collected during a test are plotted in the form $C^{1/3}$ vs. $a$ and linearly fitted to obtain a term, $EI_{EFF}$, that describes the effective stiffness of the tested beams, and a term, $x_t$, that takes account of other issues such as the root rotation and the systematic inaccuracies in crack length reading. This procedure permits one to calculate $G_{IC}$ with one of the following:

$$G_{IC} = \frac{P_c^2 (a + x_t)^2}{EI_{eff}b} = \frac{3P_c \delta}{2b(a + x_t)}$$

If the two beams are identical and of constant stiffness along their length and the shear deformation is negligible, the plot $C^{1/3}$ vs. $a$ is a straight line and the value $EI_{eff}$ is usually close to the stiffness of the beams. In tests performed with specimens of variable $EI_{EQ}$ along the length, the fitting of the data $C^{1/3}$ vs. $a$ was considered statistically acceptable because the coefficient of correlation $R^2$ was higher than 0.9 [113], where the value 1 indicates perfect linear correlation. For example, Figure 46 shows the fitting of the data associated with the case $\omega=4\pi/L$, $\delta=0$, that with the SBT gave high variability of calculated $G_{IC}$. Considering the intrinsic inaccuracies of experimental practice, one would probably not expect that data fittings as in Figure 46 can have any issues, but when these data were used for the computation of CBT, the results did not show the imposed constant $G_{IC}$, but the variability that is illustrated in the plots of Figure 47.

![Figure 46: Nondimensional $C^{1/3}$ vs. $a$.](image-url)
Without much difference from the SBT analysis results, the values of $G_{lc}$ evaluated with CBT were also inconsistent with the assumed $G_{lc}$ value for the material system. Basically the $G_{lc}$ values calculated with the CBT followed the fluctuation of the stiffness of the specimen. In a real test, one cannot easily identify whether the $G_{lc}$ variability that is measured applying the CBT is a real $G_{lc}$ variation, given for example by variable thickness of the adhesive layer, or is a result of the variable elastic modulus in the beams.

**Experimental Compliance Method (ECM) Analysis**

In the ECM, the data of applied force, displacement and crack length that are collected during a test are plotted in the form $\log(C)$ vs. $\log(a)$ and linearly fitted in order to obtain the terms $C_O$ and $n$ that permit calculation of $G_{lc}$ as function of $C_O$, $n$ and the load, or $n$, the load and the displacement, with:

$$G_{lc} = \frac{P_c^2}{2b} nC_O \delta^{(n-1)} = \frac{nP_O \delta}{2ba}$$

Also in this case the data fitting procedure permits one to overcome some issues of the SBT. The technique is considered very stable and, together with CBT, superior to SBT [123]. Moreover, also in this case the data fit did not highlight particular anomalies in terms of a low $R^2$ value; as shown in Figure 48, the data fit is nearly linear.
Nevertheless, when the ECM was performed on the data coming from DCBs with the characteristics of Figure 44, the results did not show the imposed constant $G_{lc}$, but the variability illustrated in Figure 49.

![Figure 48: Nondimensional Log(C) vs. Log(a).](image)

![Figure 49: Values of $G_{lc}$ obtained with experimental compliance method (ECM) analysis.](image)

Also in this case the values of $G_{lc}$ were far from constant and followed the fluctuation of the stiffness of the DCB specimen, in a fashion that did not differ significantly from CBT. For the considered cases, the ECM did not show any particular advantages when compared with SBT or CBT.

**Area Method (AM) Analysis**

Although not suggested by standards, the area method can be applied for determining $G_{lc}$. The method requires one to load the DCB specimen until the crack propagates, then stop the displacement imposed by the testing frame, and, at least partially, unload the
specimen. The procedure is necessary for properly calculating the area of the loading-unloading cycle in the force-displacement graph, since it is rather common that in the unloading phase the point at zero load does not correspond exactly to zero displacement, even when the beam remains elastic. This procedure has to be repeated for some of the steps of crack growth and at some point one can simply draw straight lines back to the zero point. The area between the two curves in the graph of load vs. displacement indicates the energy spent for the crack growth. Basically $G_{ic}$ can be calculated with Equation 27, using finite differences. The periodic unloading and reloading of the specimen is a rather tedious procedure and the evaluation of the area is usually subjected to various sources of error. Moreover, the method is not suitable for analysis of stick-slip crack growth, which can occur in some material systems. A couple of papers [57, 90] have evaluated the AM to be less accurate than CBT and ECM. Nevertheless, the AM was applied in this analytical study where experimental issues were not present. The results follow in Figure 50.

Figure 50: Values of $G_{ic}$ obtained with area method (AM) analysis.

Neglecting the practical issues that limit the applicability of the AM, Figure 50 shows that, for the considered cases, the AM returns results that are much more consistent with the initial hypothesis of $G_{ic}$ constant than SBT, CBT and ECM. This aspect is probably due to the fact that the AM is very close to the original definition of $G_{ic}$ without using
terms such as $E_S$, $E_{EFF}$, $C_0$ and $n$ that average the properties of the different sections of the beams.

**Comparison of Standard Methods**

The results of $G_{fc}$ obtained with SBT, CBT, ECM and AM were presented from Figure 45 to Figure 50 and the accuracy of each method was discussed in the previous sections of the paper. For condensing the results of the analysis, two other graphs are presented in Figure 51 and Figure 52. The graphs show the average values of $G_{fc}$ and the error deviations that were obtained in each of the considered configurations by the four methods. The configurations refer to the stiffness distributions illustrated in Figure 44. Configuration 1 is associated with $\omega=\pi/L$ and $\delta=0$, 2 to $\omega=\pi/L$ and $\delta=\pi/2$, 3 to $\omega=4\pi/L$, $\delta=0$, and 4 to $\omega=4\pi/L$, $\delta=\pi/2$.

![Graph showing the average values of $G_{fc}$ obtained from SBT, CBT, ECM and AM.](image)

*Figure 51: Distribution of average $G_{fc}$ values obtained from SBT, CBT, ECM and AM.*
Figure 52: Distribution of standard error for $G_{ik}$ values obtained from SBT, CBT, ECM and AM.

Although associated with four particular cases of stiffness distribution, both of the graphs give information for understanding findings of the analysis that were performed with SBT, CBT, ECM and AM. The first of the two graphs, Figure 51, indicates that the average $G_{ik}$ obtained with the SBT and CBT are particularly imprecise when applied to configurations 1 and 2, where the wavelength of $EI(x)$ variability is relatively long and also the average stiffness of at least one of the two beams is different from the nominal stiffness. It is interesting to see that ECM and AM yield average values of $G_{ik}$ close to the correct value in all the configurations and that, in configurations 3 and 4, the four methods are, on the average, similarly accurate, with an estimated error in the average value lower than 3%. Nevertheless this information is incomplete and Figure 52 gives better insights of the issue of $G_{ik}$ estimation. This issue is not only associated with the average values, but with the scatter of the data. The graph shows the standard error of the data calculated with the four methods. The higher level of accuracy of the AM when compared to the other three methods is clearly visible, since the standard error for AM is constantly the lowest in the cases that are considered. SBT, CBT and ECM have overall similar levels of error deviation, with slight advantage of the ECM. The graphs of Figure 51 and Figure 52 give an estimate of the inaccuracies of $G_{ik}$ calculated with the four methods: SBT, CBT, ECM and AM. With the representative stiffness distributions that were considered, the AM proves to be the most accurate in the $G_{ik}$ estimate, given the relatively low value of standard error. The SBT and the CBT have some problems of
large standard error in, respectively, the first and the second of the configurations. Finally, the ECM seems to give results that are generally stable, when compared to SBT and CBT, but considerably behind the levels of the AM.

In DCB fracture tests, when one uses beams of a homogeneous material and section, the elastic properties of the adherends are assumed to be uniform and constant. Nevertheless, the results of $G_{lc}$ values measured with SBT, CBT and ECM can be confusing when applied to layered beams with inclined layers. In fact, the three methods indicate high variability for the values of $G_{lc}$, especially if the mechanical properties difference and spacing are relatively large. The three methods SBT, CBT and ECM misidentify EI variations with $G_{lc}$ variations; it is thus challenging to evaluate whether the $G_{lc}$ variations obtained with these methods reflect a true variability of $G_{lc}$ or are, as it was in the illustrated examples, only an artifact generated by the adherends and, in particular, by the bending stiffness variability of the adherends. The AM, on the other hand, seems to offer better possibilities for deconvoluting EI and $G_{lc}$ variations.

4.10 Effects of stiffness variation on mode mixity angle

The symmetry of the geometry and applied loads in standard DCB specimens induces a fracture mode that is nominally pure mode I. In fact, for generating mixed-mode I/II conditions at the crack tip, researchers usually employ asymmetric DCB [135] or impose asymmetric loads on DCB specimens [78]. Other methods for implementing mixed-mode conditions can be found in the literature [82, 112]. In the case investigated in this paper, the specimens were macroscopically symmetric, meaning that the DCBs were obtained by bonding beams of the same section and material; also, the imposed loading was symmetric. This configuration should result in a mode I loading condition at the crack tip, meaning that, other than small local effects due for example to crack kinking, the global mode-mixity angle should always be equal to zero. But because the stiffness along the beam was variable, the configuration is no longer symmetric and a mode II loading component is to be expected at the crack tip. The development of mixed-mode loading conditions in asymmetric beams was described in the work of Hutchinson and Suo [18]. They studied mixed-mode fracture in solid orthotropic layered materials with cracks parallel to the principal axes of stiffness. Although the focus of the present study
was on bonded layered materials where the crack is propagating through the bond and not parallel to principal axes, assuming a minimal layer thickness, as it is in bonded wood, the system of two bonded beams with variable stiffness can be approached as a system of a solid and homogeneous material with beams of different thickness. Consequently, the approach and the findings indicated by Hutchinson and Suo for calculating the angle of mode mixity as function of the ratio of the thicknesses between the two beams was applied. The variability of $EI_{EQ}(x)$ for a single beam was previously evaluated at a level $\pm 20\%$ for some relevant cases. This meant that the ratio between the $EI_{EQ}(x)$ of the two bonded beams of the DCB in one section can vary in the range -34 / +50%. This ratio between the stiffnesses can be transformed into a corresponding ratio between the thicknesses of the beams, which turns out to be -13 / +14%. This transformation is needed in order to apply the formulas of Hutchinson and Suo. In the considered range, the correlation between the ratio of $EI_{EQ}$ and the angle of mode-mixity turned to be almost linear, as shown in Figure 53. It was computed that the angle of mode-mixity can shift from the nominal $0^\circ$ by $5^\circ$, in either direction, when the crack grows along the specimen.

![Graph showing variability of mode mixity as a function of the ratio between the $EI_{EQ}$ of the two bonded beams.]

**Figure 53:** Variability of mode mixity as function of local ratio between the $EI_{EQ}$ of the two bonded beams.

The $5^\circ$ angle shift value can have some effects on the results of the critical strain energy release rate, since the angle indicates that a component of mode II can be present at the crack tip. The mode II component can steer the crack path toward an interface, changing the locus of failure from cohesive to adhesive [74, 81], and may also affect the measured $G_c$ in some material systems. In fact, as observed by Singh et al. [88] in southern yellow pine bonded with polyurethane adhesive, and by Dillard et al. [81], the value of $G_c$ for small mode-mixity angles can be lower than the value at pure mode I, $G_{IC}$. 
This aspect is an additional source of variability in the mode I fracture test results, since the test is always regarded as a pure mode I test and the mode II component is not properly accounted for.

4.11 Conclusions

The variability of elastic stiffness along the beam length is a factor that is usually not considered in bonded DCB specimens where the two beams have the same geometry and material. Layered materials in which the principal directions are not aligned with the beams’ edges are an example of beams where stiffness variation along the length occurs and can be an issue. For example, bonded wood DCBs not only have random variability of elastic properties associated with the nature of the material, but are also usually bonded in a way that enhances the stiffness systematic variation. The fact that the grain is not parallel to the edges of the beam gives some advantages during the tests, but furnishes beams in which the mutual position of earlywood and latwood changes along the length of the specimen. The effects of grain orientation along the longitudinal plane and along the transverse section of the beam on the elastic stiffness were evaluated for a particular wood species, Douglas fir, and for some generic representative cases.

When beams with variable stiffness are bonded to obtain a DCB specimen, the stiffness variability can obstruct the proper measurement of the critical strain energy release rate \( G_{lc} \), including when testing procedures that are widely known and indicated by standards (BS 7991, ASTM D 3433) are applied. Basically, the fracture properties of the bonded system are difficult to measure independently from the elastic properties of the bonded parts, thus variability in the elastic stiffness overlaps and confuses the evaluation of \( G_{lc} \). The methods that were analyzed were the simple beam theory (SBT), the corrected beam theory (CBT), the experimental compliance method (ECM) and the area method (AM). The first three methods generally failed to distinguish variability of elastic stiffness from variability of \( G_{lc} \). Moreover, the graphs used in CBT and ECM did not highlight particularly the fact that the beams did not have constant stiffness, thus in experimental measures one may not notice the existence of a stiffness variation from the experimental data of fracture tests. The AM gave the most accurate results in the considered cases, but is also the only method that is not recommended by the standards.
because of practical problems with its experimental implementation. In addition to these issues, stiffness variability also introduces slight amounts of mode II loading in the DCB specimens. Being only mode I loading expected, the SBT, CBT, ECM and AM incorrectly associate the results to values of $G_{lc}$, neglecting the presence of the $G_{II}$ component that can alter the fracture toughness of the specimen.

The effects presented in this paper show how some of the natural variability that is often encountered when analyzing scattered data in bonded wood joint tests can be analytically explained by expected variations in bending stiffness of the adherends along their length. This suggests accuracy limitations that may result when the traditional methods indicated by the standards ASTM D 3433 and BS 7991, initially designed for uniform materials, are applied to joints containing adherends with varying stiffness which is often unknown. Current work focuses on the measure of the local stiffness values in wood beams, in order to provide experimental validation to the model that was presented [136].

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Chapter 5

Methodology for measuring the local elastic bending stiffness along wood beams\textsuperscript{8}

5.1 Abstract

An experimental method and data analysis procedure capable of determining the local bending stiffness of different sections of beams are presented. The experimental approach is based on three-point bending tests repeated along different portions of an individual beam. The data analysis method computes the local stiffness from experimental data through the Moore-Penrose generalized inverse, a linear algebra technique that estimates the least-square best-fit solution in over-determined and underdetermined systems that do not have a unique solution. The procedure is applied to yellow-poplar wood and, for validation purposes, to a uniform material, aluminum. The results from a limited sample size relevant to bond testing suggest that the local variability of elastic bending stiffness in yellow-poplar beams is likely to be of the order of 30 to 50\% and considerably larger than the variability that the same method obtains for aluminum beams. This analysis consolidates and provides experimental validation for some of the hypotheses that were assumed in [136], where the variability of bending stiffness was shown to influence the measured fracture energies in adhesively bonded wood beam specimens. The quantitative results presented in this paper have particular relevance considering that yellow-poplar is fine grained, and generally considered a wood with relatively uniform spatial morphology [137].

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5.2 Keywords

Stiffness variability, wood variability, local stiffness, 3-point bending, Moore-Penrose, generalized inverse.

5.3 Introduction

Fracture mechanics methods using double cantilever beam (DCB) geometry have been applied to evaluate the fracture toughness of adhesively bonded systems by numerous researchers. DCB specimens normally consist of two equal beams bonded together. For mode I loading conditions, one of the beams is connected to the fixed grip of a load frame and the other pulled by an actuator. From the works of Ripling, Mostovoy et al. [20, 121, 122], where the concept of strain energy release rate was applied, to the studies of Kinloch [138] and the refinements of Blackman, Brunner et al. [91, 123], the use of DCB specimens has become common practice for evaluating the fracture properties of adhesively bonded systems. The DCB specimens and the different test methods that can be applied are described in standards BS 7991 and ASTM D 3433 [86, 87]. The ultimate result of the different methods is the evaluation of the critical strain energy release rate or fracture energy that, for pure mode I tests, is denoted $G_{IC}$. The value of $G_{IC}$ indicates the amount of energy per unit area that is required to extend a pre-existing crack.

As described by Liswell [99] and later by Nicoli and Dillard [136], some issues can arise by applying the methods of BS 7991 and ASTM D 3433 on bonded specimens obtained from layered materials, where the principal axes of the stiffness matrix are not parallel to the axis of the beam. This construction is particularly common in wood DCBs, where a slight tilt in the grain orientation has often proven to be beneficial in preventing the growing crack from moving into the adherends, as described by Gagliano and Frazier [89]. Figure 54 shows a DCB specimen with grain orientation of 3-6° with respect to the bond plane. This grain orientation range limits the crack propagation into the adherends for yellow-poplar [126] and southern pine [127] bonded specimens.
Figure 54: Wood DCB specimen with fiber orientation 3-6° with reference to the bondplane.

The inclined grain orientation is thus advantageous from a practical standpoint, since it allows one to interrogate the adhesive bond by increasing the likelihood that the crack will propagate within the bondline, but introduces variability of both elastic stiffness along the beam length and angle of mode mixity as the crack tip moves forward. The variability of stiffness properties and angle of mode mixity are effects that increase the scatter of the $G_{lc}$ values within a given DCB specimen. This scatter is well documented for bonded wood in various experimental studies [36, 102, 103, 116] and its nature was investigated and analytically described by Nicoli and Dillard [136]. That paper provides a mechanical model addressing a aspect causing the variability of $G_{lc}$ in bonded wood: the variable stiffness $EI(x)$ along the beams of the DCB specimen. This aspect result from the orientation of the grain and the different mechanical properties of earlywood and latewood. Applying the methods indicated by BS 7991 and ASTM D 3433 for calculating $G_{lc}$, the variability in $EI(x)$ is inappropriately reflected as variability of $G_{lc}$. The evaluation methods, in fact, do not discriminate a real variation of $G_{lc}$ from the generally unknown variation of $EI$, thus leading to less consistent results for calculated fracture energies along the length of a specimen.

For strengthening and giving experimental basis to the findings of [136], a technique for evaluating the local stiffness $EI(x)$ along a wood beam was developed. In particular, the procedure was applied to beams previously bonded and tested as DCB specimens. The bases of the assessment of $EI(x)$ were a set of three-point bending tests conducted over small segments of the beams and a mathematical procedure based on the compliance data obtained from these tests. The tests were performed on wood beams and also on aluminum bars, where $EI$ variation was not expected. The tests proved that the variability of $EI(x)$ in wood beams is large.
5.4 Background

The bending stiffness of a homogeneous beam, $EI$, is a parameter that combines a material property, the elastic modulus $E$, with a geometrical property, the second moment of area, $I$. In a beam, an effective value $EI_{\text{eff}}$ can be easily calculated with mechanical testing from the compliance measure in particular loading configurations, such as the cantilever beam or the three-point bending. The value is “effective” because the calculation is easy and direct if one assumes that the $EI$ value is constant along the beam length. If the stiffness $EI$ is variable, one can still obtain a value for $EI_{\text{eff}}$. On the other hand, measuring local values of bending stiffness is not trivial. The common approach for calculating $EI_{\text{eff}}$ gives only an average of the local properties within the gage length [139]. The option of making the gage length short is not feasible, since the principles of beam theory require the specimen to be slender.

The idea that is pursued in this study is to repeat the three-point bending test at different positions along the beam and calculate the elastic stiffness $EI$ using the basic equation

$$C = \frac{\Delta}{F} = \frac{L^3}{48EI}$$

where $C$ is the compliance, which is the ratio between $\Delta$, the displacement of the beam at the midpoint, and $F$, the applied load at the midpoint. $L$ is the span of the three-point test fixture. The self-weight of the beam is neglected. It is fundamental to remember that, since a single three-point bending test would still furnishes an average measure of elastic stiffness, the applied procedure involves repetitive tests performed on a wood beam at different and partially overlapping positions.

5.5 Experimental approach

The experimental analysis of the local stiffness $EI(x)$ values along the beam includes different three-point tests repeated on the same beam. A schematic view of the testing sequence is illustrated in Figure 55. In this example, the beam is divided into 7 equally long sections for illustration purposes, whereas in the real tests the number of sections is larger. In the example, the sections have a length of one unit, the beam is tested with a fixture span of length equal to 4 units, and four three-point bending tests can be performed.
In each of these tests, the measured stiffness is a function of the elastic stiffness of all the sections within the fixture span $L$. Given the geometry, some of the sections influence the measured stiffness of multiple tests, while the sections at the left and right ends each influence the result of one test only.

![Figure 55: Schematic of three-point bending tests for detecting local elastic stiffness. Note that in the real beams the number of sections and tests was higher than what is illustrated](image)

In each test, the values of elastic modulus of the 4 different sections that are within the fixture span combine in giving the equivalent elastic stiffness that can be calculated from the values of load and deflection at the point of load application in Equation 36. This elastic stiffness is equivalent to that of a beam of uniform stiffness that would experience the same midpoint deflection when tested under the same conditions, but could actually be obtained with an infinite number of different distributions of $EI(x)$ within the testing fixture span. The developed method processes a sequence of measurements of the equivalent stiffness from partially overlapping spans of the beam. All the evaluations and analysis are performed not directly on the stiffness $EI$ but on the compliance $C$. $EI$ and $C$ are inversely proportional as shown in Equation 36, but this transformation simplifies the calculations since the different sections act like a series of springs and thus combining compliances is easier than combining stiffnesses. In particular, the equivalent compliance is a weighted function of the compliances of each section within the fixture span. The contribution of a single section in the measure of the equivalent compliance incorporates a weight function, so that one can describe the measured equivalent compliance as a linear combination of the local values with an equation like the following:
\[ C_{\text{equivalent}} = \sum_{i=1}^{n} w_i \cdot C_i \]

The weight function represents the effect that a local change on a section of the beam has on the measured equivalent compliance. One can intuitively understand that in a three-point bending test, the local compliance in proximity of the simple supports (where the moment approaches zero) has a vanishing effect on the equivalent compliance, while the local compliance at the center of the beam, where the bending moment is maximum, has an effect that is probably higher.

### 5.6 Evaluation of weight functions

The evaluation of the weight function was performed analytically using a Mathematica® (Wolfram Research, Champaign, IL) code, obtaining the same results as reported in [140]. This evaluation is based on the elastic theory of a Bernoulli beam, described with the well-known equation

\[
\frac{d^2 y(x)}{dx^2} = \frac{M(x)}{EI(x)}
\]

where \( y \) is the deflection, \( x \) is the coordinate along the beam length, and \( M \) is the local moment. In many engineering problems, the bending stiffness \( EI \) is a constant or, at least, a known function. The analytical evaluation of the weight function was performed with the following procedure consisting of four steps. (1) The deformed equation \( y_{\text{CONST}}(x) \) for a beam of constant stiffness \( EI_{\text{CONST}} \) along the length \( L \) was calculated. (2) The deformed equation \( y_{\text{STEPS}}(x,X) \) for a beam of variable stiffness \( EI_{\text{STEPS}}(x) \) as illustrated in Figure 56 was analytically obtained solving the differential Equation 38; note that \( y_{\text{STEPS}}(x,X) \) is also a function of \( X \), the coordinate of the portion of increased local stiffness, and that the solution was solved numerically, since the shape of \( EI(x) \) did not allow easy analytical computation with Mathematica®.
(3) The difference between the deformed shape described at the points 1 and 2 was calculated with

\[ V(x, X) = y_{\text{CONST}}(x) - y_{\text{STEPS}}(x, X) \]

(4) Since in the real test the displacement was only measured at the midpoint of the length \( L \), the function \( V \) was then evaluated for \( x = 0.5L \); so, the single variable function \( W(X) = V(0.5L, X) \) is the weight function for the particular case of a beam of elastic stiffness \( EI_{\text{STEPS}}(x) \). The generic weight function \( w(X) \) is then obtained by scaling \( W(X) \) in order to have the integral of \( w(X) \) within the testing fixture span equal to 1:

\[
\int_0^L w(X) dX = K \int_0^L W(X) dX = K \int_0^L V(0.5L, X) dX = 1
\]

From now on in this paper, with a slight change in variable notation, the weight function will be denoted \( w(x) \). The choice of \( L/100 \) pointing step (2) for the length of increased stiffness was chosen so that this localized stiffness allowed a fine resolution in the numerical solution of the problem. For the geometry of the three-point bending and fixture span of length \( L \), the weight function is described by the following equation and is plotted in Figure 57:
The terms $w_i$ for Equation 37 come from a discretization of $w(x)$, being the values of the function $w(x)$ at the center of each section in which the beam was subdivided. Looking again at Equation 37, the goal of the procedure is the evaluation of local compliance values, which are unknown, given the measures of effective compliance. The values of effective compliance are associated with each of the three-point bending tests repeated on the beam as illustrated in Figure 55.

5.7 Mathematical solution of the problem

For each test an equation like Equation 37 can be written. Combining the equations coming from the different tests performed on a beam, the system of Equation 42 is formed. The term $w_j$ represents the value of the weight function for section $j$ in the configuration of the test $i$, and the system shown in Equation 42 can be written to summarize the information gathered from multiple three-point bending tests on a single beam.
In this study, the known equivalent compliances and weight function are used to evaluate the local compliances. So in the system of Equation 42 the unknowns are not the terms on the left-hand side of the equation, but those of the vector $C_{\text{section}}$. Thus, the system is underdetermined, since the matrix of the terms $w_{ij}$ has more columns than rows. In fact, the number of three-point bending tests that can be physically run on a finite beam is lower than the number of sections in which a beam is divided. This is due to the fact that sections close to the edges are tested in a limited number of cases, as can be seen in the schematic example of Figure 55, where there are 7 sections (and $C_{\text{section}}$ terms) and only 4 tests (and $C_{\text{equivalent}}$ terms). A solution of the problem can nonetheless be evaluated using the concept of a pseudoinverse matrix, also known as the Moore-Penrose generalized inverse. There are an infinite number of $C_{\text{section}}$ vectors that satisfy the system of Equation 42, but the Moore-Penrose generalized inverse is a procedure that, among the infinite possible solutions, furnishes a unique solution. This solution, in an underdetermined system as in the considered case, is the vector with the smallest Euclidean norm. Mathematical bases and details on the properties, construction and evaluation of the Moore-Penrose generalized inverse are given in [141, 142].

The mathematical method was developed as a code with the software package Mathematica® (Wolfram Research, Campaign, IL) and validated with some simulations. These simulations were performed assuming various $EI(x)$ distributions, numerically computing the equivalent compliances that different three-point bending tests would provide, reconstructing the values of $C_{\text{section}}$ with the Moore-Penrose generalized inverse and finally obtaining $EI_{\text{section}}$. The imposed real distributions $EI(x)$ were simple functions, such as a square step and a sinusoid, where the maximum variation is +/- 20% from a reference value. This variation amplitude is consistent with values that were analytically evaluated [136]. The results of two simulations are shown in Figure 58.
The graphs of Figure 58 show continuous lines representing the real distribution of $EI(x)$, open symbols providing the $EI_{eff}$ of simulated three-point bending tests, and closed symbols conveying the data obtained with the described analysis that obtains $EI_{eff}$ with the Moore-Penrose generalized inverse. The proposed method does not yield correct results for sections that are close to the ends of the beam, where, given the lack of data points, the computation is unstable. Also, for smooth changes of $EI$, such as the graph on the left of Figure 58, the utility of the method is not apparent. But when $EI$ variations occur at higher frequency, such as the graph on the right of Figure 58, the proposed method is able to follow the variations and the correct amplitude, whereas the estimates coming directly from the three-point bending tests are not. In fact, Figure 58 shows that the closed symbols are distributed with an amplitude that matches the amplitude of the real stiffness $EI(x)$, in a continuous line, while the open symbols underestimate the amplitude of the variability. Moreover, with a distribution of $EI$ equal to a square step, as shown in Figure 59, the proposed method traces the sudden variation almost perfectly, whereas the $EI_{eff}$ data coming directly from the three-point bending suggest a limited amplitude of stiffness variations and give a less accurate representation of the real $EI(x)$ distribution.

Figure 59: Examples of Moore-Penrose based solutions for step distribution of elastic stiffness.
The examples presented in Figure 58 and Figure 59 show the strengths and the limitations of the proposed procedure. The main strength of the procedure is that it is able to detect $EI$ variation better than the simple three-point bending test. This particularly means that the amplitude of the variation of the real $EI$ is computed with precision by the proposed method, while the three-point bending tends to underestimate changes. This aspect is important because the procedure was originally developed to provide a good estimate of the variability of the local stiffness $EI$. A limitation of the method is that the results are not reliable close to the beam edges. This problem can be partially overcome in two ways: (1) substituting, in these positions, the results provided by the three-point bending test; (2) focusing just on results associated with the central part of the beam.

### 5.8 Test procedure and validation

For all the three-point bending tests performed in this study, the loading was a ramp at 1 mm/min displacement rate up to a load of 400 N. This maximum load level produced a clear linear load-displacement signal for the evaluation of the compliance without plastically deforming or damaging the beam. Displacement and force signals were recorded. Once a run was completed, the beam was translated by $\Delta x$ and the next loading cycle was performed on the same beam. This process was repeated from one end of the specimen to the other, qualitatively as in Figure 55. The three-point bending test procedure was applied to wood beams that were the main focus of the study. For validation purposes, aluminum beams were considered. In an aluminum beam, one should expect the elastic stiffness not to be a function of the position along the beam. The lengths of the beams were 220-240 mm and the three-point fixture span was 101.6 mm, while the length of the translation step (length of the section) was 10 mm. This means that an average of 13 to 15 loading tests per beam were run and that within the fixture span at least 10 sections of the beam were influencing the results of each three-point bending test.

In the aluminum beam, of section 6.3 x 25.4 mm, one hole of 3.4 mm diameter was drilled through the thickness, resulting in a local reduction in $EI$ of 13.4% based on the net section remaining. The results that were obtained from the measurements and the
analysis are shown in Figure 60, as $EI$ normalized with reference to the tabulated value for a section 6.3 x 25.4 mm and elastic modulus 70 GPa, as for aluminum. The graph shows with squares the values of $EI$ that come directly from Equation 36 applied to the measured $C_{equivalent}$, while the circles indicate values of $EI$ calculated from the $C_{section}$ values obtained with the Moore-Penrose inverse. The graph shows also in this experimental case the strengths and the limitations of the method, which is very sensitive to stiffness changes (the presence of the hole), and is not accurate and becomes unstable close to the ends of the beams. In fact, a relatively small reduction of the values $EI_{section}$ is present in the section corresponding to the hole, the 11th in Figure 60. On the other hand, the data of $EI_{equivalent}$ exhibit a localized minimum at the same section. A key aspect is that the section with the drilled hole tends to lower the compliance data measured in each of the tests, and thus the measured $EI(x)$ is generally lower than the measured $EI(x)$ when the hole is not present. But, in the section of the drilled hole, the measured $EI(x)$ is higher than the analyzed value because it is influenced by the effect of all the sections within the testing span.

One final comment is that the normalized $EI$ values found from both $C_{section}$ and $C_{equivalent}$ were generally smaller than the unit value. This was probably due to the fact that the three-point bending fixture introduced some additional compliance in the experiment. This effect is of limited importance, given that the proposed method was not developed for assessing the absolute value of $EI$, but rather for detecting $EI$ variations. As expected, a limitation of the method is that data points obtained near the specimen edges are very scattered and are not reliable, as can be seen in sections 6, 7, 15 and 16 of Figure 60. Nevertheless, for the sections between the 8th and 14th, the values are quite stable, obviously with the exclusion of the section associated to the hole.
Figure 60: Three-point bending measurements (squares) and analysis (circles) for a drilled aluminum bar.

5.9 Experimental results on wood beams

The material of the tested beams is yellow-poplar (*Liriodendron tulipifera*), a hardwood of the Magnoliaceae family, and the beam dimensions are 20 x10 mm section and 220-240 mm length, with the three-point bending measures repeated at steps of length 10 mm. An example of the data measured and analyzed is shown in Figure 61, where the dotted line shows the estimate of $EI(x)$ from the three-point bending test ($C_{equivalent}$) and the continuous line gives the estimate from the analysis ($C_{section}$). As seen also in the validation of the methods, the analyzed data enhance the variability of the stiffness.
Figure 61: Example of EI(x) measured and analyzed data.

For completeness, the results from all the three-point bending measures are shown in Figure 62 and the results, from Moore-Penrose analysis in Figure 63. These two figures highlight some important aspects of the analysis. First, the EI(x) directly obtained from the three-point bending test, Figure 62, already show a variability that is larger than the corresponding aluminum data illustrated in Figure 60. Secondly, the analysis with the Moore-Penrose generalized inverse still enhances the variability of EI(x). It is noted that the two graphs of Figure 62 and Figure 63 have different scale of the y-axes. Figure 62 shows that the variability of EI(x) within a single specimen can roughly be between 4-5% and 20%, while the analyzed data of Figure 63 estimate that the variability ranges between 30% and 50%, not considering the data points associated with the first and last 3 sections, which may be affected by the data instability seen close to the ends of the beams.
Figure 62: Local EI values for eight tested beams. EI(x) obtained from $C_{\text{equivalent-test}}$

Figure 63: Local EI values for eight tested beams obtained after analysis with Moore-Penrose generalized inverse. The EI(x) values are obtained from $C_{\text{section}}$

5.10 Conclusions

The proposed analysis method computes the local stiffness $EI(x)$ at different sections starting from a series of three-point bending tests on a beam, each of which returns a value of $C_{\text{equivalent}}$. This analysis then extracts values of $C_{\text{section}}$ through a mathematical procedure, the Moore-Penrose generalized inverse. The procedure seems to be effective
and reliable, having been applied to an aluminum bar with a drilled hole and having found stable results of fairly constant $EI$ in all the sections excluding the one with the drilled hole. A limitation of the procedure, which mathematically handles an underdetermined linear system, is that the estimates associated with sections close to the ends of the beams are scattered and thus not reliable. Hence, the applied methodology is accurate only for a portion of the tested beams, but this was adequate for estimating the variability of stiffness $EI$, at least along a portion of beam. In particular, the results obtained from tests performed on the aluminum bar, a material considered to be uniform and isotropic, and from wood bars, a natural material with a morphology that determines $EI$ variability [136], confirmed that the variability in the latter material, in the considered case of yellow-poplar, is considerably larger than the variability in aluminum.

The shape of $EI(x)$ computed in [136] for Douglas-fir, and in a number of representative cases, was not observed in the experimental data. In fact, wood’s natural variability can occur from several mechanisms, such as the presence of defects and the structural differences that go beyond the simple model of earlywood and latewood plane layers, that were not considered in the model provided in [136]. As expected, the variability of $EI$ in yellow-poplar beams is considerably larger than the variability encountered in uniform and isotropic metallic beams made of aluminum. The experimental measures and analysis herein proposed show that the $EI$ variability can be evaluated and, more importantly, that the variability of $EI$ in yellow-poplar beams is likely to be of the order of 30 to 50%, thus qualitatively supporting the hypothesis of variability $\pm 20\%$ (equal to 40% range) chosen in [136].

5.11 Acknowledgments

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Chapter 6

Conclusions

6.1 Research summary and contribution

With an experimental effort performed for measuring the mixed-mode (I/II) fracture properties of yellow-poplar bonded with two adhesives (a moisture-cure polyurethane and a phenol/resorcinol/formaldehyde resin), this thesis first evaluated how the critical values of energy release rate, $G_c$, change for different levels of mode-mixity. The specimen geometry that was considered in the tests is the double cantilever beam (DCB) type, where two beams of the same section are bonded together along their length.

The DCB specimens were quasi-statically tested with a dual-actuator load frame to determine $G_c$ for loading modes ranging from pure mode I to pure mode II. The experimental procedure that was developed for measuring the mixed-mode fracture properties consists of 3 separate testing phases, two of which were mode I tests. This approach led to some insights regarding the corrected beam theory (CBT) and the experimental compliance method (ECM). These two techniques are described in the literature and standards for pure mode loadings, and were adapted here to the mixed-mode tests.

The dual-actuator load frame that was used is a testing machine that offers several advantages during the tests, and introduces some characteristic features that are not seen in mixed-mode tests performed with other experimental set-ups. These features include the variability of the angle of mode-mixity during tests performed in displacement control, and a nonlinear effect that arises with the imposition of loads or displacements at the two unbonded ends of the DCB specimen. An evaluation of the amount of variability of the mixed-mode angle was presented and a closed-form solution for imposing displacements that maintain constant angle of mode-mixity was analytically developed.
For the nonlinear effect, a correction factor $T$ that can be used to normalize the $G_c$ value calculated with traditional methods was computed. This factor can be used along with the factors $F$ and $N$ proposed by Williams.

The results computed with the analysis show an appreciable data scatter of $G_c$ and its components ($G_c)_I$ and ($G_c)_II$. This scatter seems not to be caused by inaccuracies of the test procedures, since it does not occur in a noise-like form where the results are randomly scattered, but has some trends, which are not consistent from specimen to specimen.

Possible causes of these trends were indentified in the variability of thickness of the bondline and the nonconstant bending stiffness of the beams bonded to form the specimens. Microscope measures found that the variability of adhesive layer thickness is considerable even in specimens carefully prepared with power planed wood boards, and comparisons among specimens suggest that a close correlation exist between adhesive layer thickness and measured $G_c$.

The variability of bending stiffness along a wood beam can have several causes. A model for evaluating this variability as a function of earlywood and latewood distribution within the beam was developed, compared to the related classical lamination theory, and applied to Douglas fir data available in the literature. The stiffness difference between earlywood and latewood, as well as the grain angle orientation, cause variability of local bending stiffness along the length of the wood beam. The stiffness variability associated with earlywood and latewood distribution can be considerable, and its effect on the measured $G_c$ was shown to be detrimental in many cases. In fact, some of the traditional analysis methods applied to measure $G_c$ usually fail to distinguish variability of bending stiffness from variability of $G_c$; thus accuracy limitations in the measure of $G_c$ in bonded wood may be expected.

Finally, an experimental and analytical procedure for evaluating values of local bending stiffness was developed and successfully applied to some analytical models of stiffness distribution. Practical implementation of the procedures proved to be sometimes troublesome. Nevertheless, tests performed on the yellow-poplar beams used for the DCB specimens suggest that the bending stiffness variability can be significant.
6.2 Recommendations for future research

The $G_c$ results obtained under mixed-mode (I/II) loading conditions in bonded wood were generated with a dual-actuator load frame. Tests performed with this instrument open a range of possibilities and issues. With the material systems that were investigated, the measured values of $G_c$ proved to be affected by data scatter. Two factors that are likely to influence the data scatter were investigated, and future work should refine the analysis.

**Dual-actuator load frame**

A closed form solution for conducting tests at constant angle of mode-mixity ($\Psi$) was developed. The same equation, implemented in the controller of the load frame, would permit one to carry out tests in which the variation of $\Psi$ is imposed and is, for example, a function of the crack length. The possibility of obtaining this test set-up on double cantilever beam (DCB) specimens is probably unique to the dual-actuator and could be used for testing the effects of $\Psi$ variations on $G_c$ and R-curves.

**Adhesive layer thickness variability**

A limit of the presented analysis is the lack of ability in performing both layer thickness and $G_c$ measures on the same DCB specimens, since both of the measures are destructive. A clear improvement would be the implementation of precise layer thickness measurements with non-destructive techniques. For this goal, magnetic resonance imaging procedures were partially investigated. However, the need to soak the specimen prior to the measurements and the expected low sensitivity (compared to the average adhesive layer thickness) limited the amount of time that was devoted to this particular analysis.

**Beam stiffness variability**

The variability of bending stiffness of the beams bonded as a double cantilever beam (DCB) specimen was demonstrated to be a serious problem affecting the $G_c$ measures with most traditional methods of analysis. The area method proved to be the most accurate in the cases that were considered, but has some practical limitations that reduce its precision in real applications. The experimental implementation of the procedure for
measuring the local bending stiffness would close the loop between stiffness variability and $\mathcal{G}_c$ variability in real specimens. For this analysis, the use of uniform and isotropic materials for the adherends is recommended.
Bibliography


Appendix A

Using standard adhesion tests to characterize performance of material system options for insulated joints

A.1 Abstract

Insulated joints (IJs) are often required every few kilometers along railway tracks for signal blocks and rail break detection; practical experience has shown that their life is often a fraction of the life of other track elements on some rail lines subjected to high tonnage freight [143]. This paper reports findings from a project conducted to study different bond systems consisting of various combinations of adhesive, fibrous insulators, and rail surface treatments that were of potential interest for increasing the service life of insulated joints (IJs) for rail applications. The study was performed in parallel with a finite element analysis [144, 145] and did not focus on testing real IJs but rather on common adhesion test specimens such as the single lap joint (SLJ) and double cantilever beam (DCB) configurations. The aim of using these specimens was to simulate potential load and environmental conditions on standard tests specimens that were less expensive and easier to construct, test, and analyze. The main goal of the project was to compare a number of combinations of potential IJ components through an extensive test program. The results highlighted several possible combinations that may warrant further study as

actual IJ prototypes. In particular, several material combinations involving materials not currently used by IJ vendors had higher overall performances when compared to currently used combinations, although the extension of improved test specimen performance to actual IJ configurations and service conditions may not follow.

A.2 Keywords

Insulated joint, railroad, adhesive, insulator, single lap, double cantilever beam, environmental exposure, surface treatment, crack growth, environmental degradation.

A.3 Introduction

Insulated joints (IJs), illustrated in Figure 64, are vital elements for many railroad lines. Although rail sections may be joined through bolting, welding, or other methods, electrically insulating joints are often required every few kilometers along railway tracks to create localized signal blocks and permit rail break detection. These joints isolate different blocks of a railroad line to form appropriate electric circuits where the passing train acts as a short between adjacent rails, effectively acting like a switch to signal its presence.

Commercial IJs mechanically join abutting rails, typically through the use of a pair of joint bars or doublers. Four or six bolts usually compress the bars and rails together and are electrically insulated by non-conducting thimbles. An insulating layer, consisting of an adhesive and frequently a fiber layer, is placed between the rails and bars, and possible gaps are filled during the fabrication process with epoxy or other polymeric materials. In recent years, freight trains have been required to carry increasingly high axle loads. Davis et al. [143] reported that these freight lines observe a notable reduction in the lifetime of the insulated joints to about 12 to 18 months, significantly shorter than exhibited by most railway components. Furthermore, the failure of the IJ is often reportedly triggered by adhesive degradation that, if not detected, leads to bolt failure [143]. Under normal service conditions, IJs are subjected to a variety of external actions [146, 147], including the almost constant traction of the rail due to the initial tensile bias applied to reduce likelihood of rail buckling in hot weather, the superimposed axial load induced by thermal expansion that cycles on a diurnal and annual basis, and material degradation that
can possibly be caused by weather effects, such as temperature, humidity, and exposure to liquid water. This degradation can especially affect the adhesive and the interface properties. Since the cost of a single IJ and even its installation is only a small fraction of the cost of the service interruption resulting from a failed IJ, an increase in IJ service life would be tremendously beneficial for railroad maintenance.

The evaluation of the economic importance of IJ failures and a survey of the defects that are usually observed in failed IJs was provided by Davis et al. [143], indicating that multiple effects concur to drive IJs to mechanical and electrical failure and that such failures tend to occur in, respectively, cold and hot/humid seasons. Complicating the survey of real IJ in-situ failures is the fact that an unknown, but probably large, number of IJs that have electrically failed remain in use. In fact, although two IJs are mounted at the end of each track signal block, the system cannot detect the failure as long as one of the two IJs is functioning [143]. In addition, reporting of failures is somewhat anecdotal, as each rail joint experiences somewhat different mechanical and environmental challenges. These aspects limit the possibility of drawing accurate conclusions just by analyzing failures of in-situ IJs, necessitating a more controlled study on inexpensive but standardized tests.

A number of variables are involved in the industrial construction of IJs, including the adhesive, the fibrous insulator, and potentially any rail surface pretreatment performed prior to bonding. In evaluating different combinations of these factors, this study focused both on combinations traditionally used by the IJ industry as well as on several other products available in the market, especially for the adhesive and surface treatments. Finding possible combinations of adhesive, insulator layer, and surface pretreatment that could be promising for IJ applications was the goal of the study. Due to the numerous variables and their combinations, the potential test matrix could be quite large, so a sequenced screening and testing program was used. Ideally, one would conduct tests of adhesive systems for IJs on geometries and loading conditions that mimic actual IJ performance. Due to the massive size of IJs, the large and complex loads required to simulate IJ service, and the costs of conducting such tests, simpler test geometries were used. Practical limitations require focusing on standardized tests that, compared to tests on real IJs, can be conducted easily for a large number of specimens with minimal costs.
and time. Before this study, a finite element analysis performed by Plaut et al. [144, 145] was used to guide in the selection of standardized tests that were most appropriate for this application. The specimen geometries considered were (a) single lap joints (SLJs), tested in accordance with ASTM D 1002 [148] for determining the “apparent shear strength”, (b) double cantilever beam (DCB) specimens, tested in accordance with BS 7991 [86] for characterizing the critical energy release rate, and (c) wedge tests conducted on similar DCB specimens to characterize the durability. The specimens are illustrated in Figure 65.

The first part of this work involved development of a screening test matrix to reduce the possible combinations of adhesives, insulators, and surface pretreatments with a simple and quick test that would provide useful indications about the structural capabilities of the final application. At this stage, the choice was to implement tests on as-produced SLJ specimens, without any environmental exposure. After these preliminary tests, the number of material combinations of the test matrix was significantly reduced, and the focus moved towards more insightful tests, such as tests on environmentally-exposed SLJs, on DCB specimens, and on wedge specimens. These tests are believed to highlight some specific features that are involved in the resistance of IJ adhesives to failure in actual field applications, and are standard tests for assessing adhesive performance for many industrial applications.

A.4 Background

For characterizing adhesively bonded joint systems, the SLJ test has traditionally been implemented and is still a widely used method, in spite of limitations associated with the complex state of stress that results when such joints are loaded. The aforementioned standard, ASTM D 1002, and standard ASTM D 4896 [149] specify that dividing the breaking force by the bond area provides an “apparent shear strength” value, which cannot be directly used to predict the strength of joints of different geometries, but is primarily for comparison purposes. Volkersen [150] first indicated the non-uniformity of the shear stress over the bonded area due to what is now referred to as the shear lag phenomenon. Goland and Reissner [151] expanded on this solution by including the bending of the adherends and the resulting peeling stresses. More recent studies [152,
153] involving numerical evaluation of these effects have refined the Goland and Reissner model. Reviews of several other approaches for evaluating the stress state in SLJs can be found in the literature [149, 154-156]. In particular, standard ASTM D 4896 indicates cautions that should be considered when using SLJ test results for predictive purposes.

With the DCB tests, the approach to describe the failure of a bonded joint is not focused on a limit of the applied stresses, but on the energy required to produce a unit area growth of an existing debond. This test characterizes the fracture energy for a specimen in which the adhesive layer is nominally subjected to a mode I (opening mode) fracture condition. In the DCB specimens, the two adherends are pulled apart as illustrated in Figure 65 during the test. The DCB test has been standardized in BS 7991 [86] and ASTM D 3433 [87], and determines the critical strain energy release rate $G_{IC}$. The test gives deeper insights into the fracture properties of a structural adhesive, since both the mechanical and electrical IJ failures are likely to be caused by cracks that develop in the adhesive/insulator layer.

Because the hardness of the steel (details in the next section) prevented drilling and tapping that are often used for attaching endblocks on thin DCB adherends, small steel tubes were welded to the adherends to accommodate pins for loading, as shown in Figure 66. The heat-affected zone did not extend far enough to degrade the adhesive and the tubes worked well for load application.

The wedge test specimens used were identical to the DCB specimens, but were tested differently as indicated in standard ASTM D 3762 [157]. Rather than propagating the crack over a few minutes in a testing machine, the debond is allowed to propagate over an extended period of time while the specimen is exposed to the desired environment. Loading is achieved by a wedge inserted in the unbounded end of the specimen, as illustrated in Figure 65. A description of the test and related aspects is given by Cognard [158]. This test focuses on the durability of the specimen because these specimens can be easily exposed to various environmental conditions; results from the test are the crack growth versus time and, in some conditions, the time to failure. The simultaneous application of mechanical and environmental conditions over time makes these specimens common for many adhesion durability studies for the following reasons. First,
stress and humidity are both factors that influence the crack growth in a material: the two effects synergistically cooperate, since in the areas of stress concentrations the deformation of the material exacerbates the ingress of water molecules. Water often reduces the fracture resistance of an adhesive bond and can significantly accelerate crack growth [18,19]. A second important reason is that real IJs are exposed to extreme weather conditions while loaded, so applying high and low temperature and humidity levels to stressed specimens may be relevant to the final application. Since metal surface oxidation can play a major role in joint durability, the wedge test was used for evaluating the surface treatments that could be beneficial for the durability of the insulated joints. Wedge samples were modified before bonding using a grit blasting operation and via surface modification/derivatization with two silane coupling agents.

A.5 Experimental Variables and Materials

Eight adhesive products were identified and tested, two of which are currently used by IJ vendors; the other six were suggested for use by major adhesive companies. Seven of the adhesives were epoxies, and one was an acrylic. For the insulator layers, two fibrous materials were considered, fiberglass and an aramid fiber, Kevlar®, which is a DuPont trade mark. Both of these materials are currently used by IJ vendors as single layer woven fabrics. In this study, these materials were applied in different forms: as a single layer, as double layers of woven fabric, or as dispersed fibers. One should note that there are a variety of weave patterns, weave densities, and sizing options for insulator fabrics, so results may vary with these features, which were not specifically investigated in this study. Some technical details for the two insulator materials used in this study are shown in Table 3.

As recommended in ASTM D1002 [148], the metal adherends used for the SLJs were obtained as 1.58 mm (1/16”) thick A-109 steel sheets that were sheared into 25.4 x 102 mm (1x4”) pieces The metal adherends were grit blasted using a grit blasting machine (Cyclone manufacturing, Dowagiac, MI) with 70-140 US sieve glass grit. These metal adherends were then wiped and soaked in methyl ethyl ketone (MEK) to remove surface debris and possible traces of oil. Insulating fabric pieces were cut to 12.7 by 12.7 mm
strips from the sheets of Kevlar® and fiberglass woven fabric described in Table 3. The dispersed fibers were obtained by shredding the fabric sheets into fine strands.

With the fabric and adherends prepared, the adhesive was mixed and applied to the ends of each adherend. The insulator was placed on the top of the adhesive layer on one of the adherends and the adherends were paired to form the single lap joint. The specimens were fabricated using a specially designed bonding fixture [159] to ensure correct alignment and a constant bond thickness. The specimens were constrained in the fixture, which was then placed in an oven for adhesive curing as specified by the respective supplier.

For the wedge and DCB tests, rail steel, available as angle shapes (Jersey Shore Steel Company, Jersey Shore, PA), was used as the adherends. Although not designed with reference to any standard, the company produces this steel re-rolling railroad rails as per ASTM specification A499-89 (grade 50). The produced steel bars have mechanical characteristics close to those of ASTM A682 [160] 1070-1080 steel. This material is not specifically requested in ASTM D3433 [87], but it corresponds to commercially available rail steel, according to Jersey Shore Steel. The metal adherends in this case were obtained by cutting some of the angle bars into plates of dimensions 305x25x6.3 mm (12x1x1/4”). The DCB specimens were grit blasted with 18-50 mesh (300-1000 micron) alumina grit at about 550 kPa (80 psi) of compressed air pressure using a Trinco 36/DLX grit blaster machine (Trinity Tool Company, Fraser, MI).

For the surface modification/derivatization, two silane coupling agents were considered. One coupling agent contained aminopropyl functionality (C$_3$H$_5$O)$_3$-Si-CH$_2$CH$_2$CH$_2$NH$_2$, that is indicated as APS in this paper, and the other silane included a secondary amine group [(CH$_3$O)$_3$-Si-CH$_2$CH$_2$CH$_2$]-$_2$NH, referred as TMS in this document. The procedure for the derivatization was performed starting with a solution composed of 5 mL of one of the silanes dissolved in 95 mL of 100% ethanol. This mixture was prepared and combined with a 100 mL solution containing 90 mL of 100% ethanol and 10 mL of 0.1 M acetic acid (CH$_3$COOH). This combined solution was allowed to react for 30 minutes at room temperature to hydrolyze the silane. The steel adherends to be derivatized were immersed in the acidified hydrolyzed silane solution to accomplish the surface modification. The derivatization reaction was carried out for 30
minutes at room temperature. After the silane derivatization, the steel was rinsed with 100% ethanol and dried in an oven at 110°C for 30 minutes to complete the derivatization step. Care was taken not to touch or contaminate the surfaces of the derivatized samples prior to bonding.

A.6 Experimental Approach

For all of the test configurations, SLJ, DCB, and wedge specimens, the curing procedures indicated in the respective technical sheets of the adhesives were followed. The tests were normally conducted within 48 – 72 hours following completion of the bonding process or the desired exposure time, for those specimens that were environmentally exposed. The tests were performed on either an Instron 4468 machine or an Instron 4505 machine (Instron, Norwood, MA), applying a constant crosshead displacement rate of 1 mm/min for both SLJ and DCB tests. In the SLJ and DCB tests, values of load and imposed displacement were automatically recorded by the testing machine at a rate of 10 Hz. For tests on the DCB specimens, values of crack length were also measured during the test and recorded by the operator.

A design of experiments (DOE) [161, 162] approach was utilized in the SLJ screening tests to understand how the different variables influenced the strength of the SLJs, without requiring tests of all the possible combinations of adhesive and insulator. This method is a statistical approach commonly used in studying processes involving a large number of variables. The main idea is that all the results can be analyzed and the effect of the single variable in the final result can be statistically evaluated, thus reducing the need for tests conducted on one factor at a time.

The performed tests can be divided into four groups:

- as-produced SLJs: static tensile tests;
- environmentally exposed SLJs: static tensile tests performed on specimens previously exposed to environmental aging;
- DCBs: fracture tests on as-produced and environmentally exposed specimens;
- wedges: tests under simultaneous mechanical loading and environmental exposure.
The SLJ and DCB tests were all conducted at room temperature. The significant number of combinations of adhesives and fabrics that was evaluated with the as-produced SLJs served as initial screening tests. Based on these results, the systems that performed better were tested with environmentally exposed SLJ specimens and then with DCB specimens, as-produced and environmentally exposed. The initial matrix of different combinations of adhesives and fabrics involved 34 combinations that were statically tested in as-produced SLJs with five replicates. These combinations were selected based on DOE concepts. For comparisons of the different surface treatments, wedge tests were also carried out.

Initially different sets of specimens were created. The sets correspond to groups of specimens with the same characteristics in terms of adhesive and insulator used in the construction. The environmentally exposed SLJs were all exposed to the same environmental cycle, which included two conditions: 1) a hot/humid environment, provided by a water bath operating at 80°C (176°F) and 100% relative humidity (RH); and 2) a cold environment achieved in a laboratory freezer set at -25°C (-13°F). A schematic representation of the environmental exposure procedure is shown in Figure 67 according to the following procedure:

The SLJ specimens were placed in the 80°C, 100% RH environment for two weeks. At that point, five specimens from each set were removed from the water bath and tested on the Instron machine applying the same procedure as used for the previous SLJ specimens. This initial exposure at high temperature and humidity was imposed to allow for some degree of moisture ingestion, although the exposure would be insufficient to saturate the specimens throughout [163, 164].

The remaining specimens were then cycled between the high temperature water bath and the freezer. The specimens remained in the water bath for 22 hours and then were placed in the freezing environment for 2 hours at -25°C. The 22 hour exposure in the bath was chosen to provide more time for diffusion of water into the bondline, while the two hour freeze was sufficient to reach thermal equilibrium and induce cyclic thermal stresses that perhaps could result in water freezing damage within the specimens. After one month of this cyclic environmental exposure, five more specimens were tested from

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each set. The remaining five specimens from each set underwent an additional two months of the same temperature cycle before being tested.

### A.7 Results and Discussion

In the results that follow, the specimens are described with reference to the adhesive used, which is indicated with a letter from A to H; to the insulator which can be either fiberglass (Fg) or Kevlar® (Kv); and to the form of the insulator used: as a single layer (indicated by the number 1), as double layers (2) of woven fabric, or as dispersed fibers (D). Adhesive A and B were provided by IJ vendors and reportedly were in use in the field at the time; adhesives C to H were those suggested by several adhesive companies that were contacted.

**As-produced single lap joints**

This test is, among those performed in this study, the quickest to run; it was used as a screening test to identify promising combinations of adhesive and insulator. No chemical surface treatment variations were considered at this step, but the specimens were all grit blasted with the same procedure with 70-140 US sieve glass grit. These tests highlighted that the apparent shear strength of specimens prepared with different adhesives varied considerably, while the insulator choice did not lead to such a large strength variation. Figure 68 presents the results for the single lap joints constructed with the eight different adhesives. Each column represents the average tensile strength of five specimens, with the error bars representing the amplitude of plus/minus one standard deviation.

The different cross-hatchings indicate the different failure modes for the specimen, as observed by visual inspection as recommended by ASTM D 1002. Adhesive indicates failures which occur at or very near an interface between the two different materials, so that adhesive /metal interface and adhesive/fabric interface refer to different interfaces. Partial adhesive/metal interface indicates that the failure was sometimes on one interface and sometimes on the other. Figure 68 also shows that adhesive D has an apparent tensile strength considerably higher than all the others. Another interesting result is that the adhesives A and B, which are currently used by the IJ vendors, have lower strengths in this test, suggesting the potential benefit of considering several of the other systems in further testing.
Additional comparisons were performed with specimens constructed with different insulator options, such as a different number of insulator layers or dispersed insulator fibers. This comparison helps to understand the effect of insulator type on joint strength, although changes in joint configuration could also affect the results and confound the interpretation, since the addition of insulator layers increases the bondline thickness and introduces new interfaces. Figure 69 shows the effect of several insulator options on the apparent shear strength of SLJ specimens prepared with adhesive B.

Compared to the bonds without any insulator (the Ne column for neat adhesive) the presence of woven fabric insulator layers seems to reduce the overall strength of the specimens; however the standard deviation bars do overlap. An increased bond thickness, as in the case of multiple layers, increases the eccentricity of the bond and the peeling stresses that are detrimental to joint strength; moreover, the presence of one or more insulators leads to new interfaces, which can possibly weaken the joint. On the other hand, the increase of bond thickness increases the compliance of the adhesive layer, which according to the shear lag model possibly lowers the peak shear stresses near the edge of the bonded area. The presence of dispersed fibers increases the apparent strength of the specimen, creating a composite-like material with the fibers completely wetted by the epoxy used for the joint. This approach may not be practical for the IJ applications however, since the absence of an insulating layer cannot guarantee electrical insulation if, for example, the epoxy squeezes out of the joint during the fabrication and the adherends make contact. The presence of one insulating layer is thought to be necessary for robustly achieving the required electrical resistance by maintaining separation of the metal adherends.

Some features of a DOE approach were used for planning and properly evaluating the tests of as-produced SLJs. The aim of DOE is to highlight which input variables are most influential on a measured output. In the case of as-produced specimens, the input variables are the different adhesives and insulator options that can be applied in the specimen fabrication; the output is the apparent shear strength measured with the SLJ tests. These input variables are referred to as factors in the DOE study. The different values that each factor can have constitute the levels of the study. In this study, the adhesive factor has eight levels, which are the eight adhesives considered in the test,
while the insulator option factor has seven levels, which are the different materials (Kevlar® and fiberglass) applied in three different modes (as one layer, two layers or dispersed fibers) and the absence of insulator material. Different combinations of the two factors, as illustrated in Table 4, were tested and considered for the analysis.

Employing a DOE analysis allowed the effects of the factors to be numerically evaluated. The main effect of a factor is the difference between the average output of combinations where the considered factor is applied and the average output of solutions where the considered factor is not applied. A positive value suggests a beneficial influence of the factor toward a high output, a result close to zero suggests an overall independence of the output from the considered factor, and a negative result implies a negative influence. For example, the main effect of adhesive A is the difference between the average of the seven outputs coming from samples constructed with adhesive A and the average of the 27 outputs coming from samples constructed with other adhesives. This difference resulted in a value of -3.9 MPa. Similar calculations repeated for the main effect of adhesive G gave 0.6 MPa. This suggests that adhesive G works better, in terms of apparent shear strength obtained in this limited study, than adhesive A. Similar comparisons can also be made among the different insulator solutions. The results of these comparisons appear in Table 5. The obtained results are important as a qualitative comparison of different effects, and a figure of merit for each of the effects can be seen in the average values of apparent shear strength shown in Table 4.

The analysis shows that adhesive D has the highest apparent shear strength. Also, Kevlar® has a slight advantage over fiberglass, but these two options have very similar outputs when applied in one layer. The dispersed fibers and the two-layer options seem also to be rather beneficial, since their results with different adhesives are overall superior to the results of the one-layer options. The effects of interactions between different factors are also important but are difficult to evaluate because of the large number of levels that each factor can have. In fact, the DOE is better applied in situations where the number of factors can be large, but the number of levels is limited. The fact that the factors are also non-numeric variables complicates the analysis as well [162]. Nonetheless, DOE allowed the identification of combinations giving high levels of
apparent strength and aided in limiting tests for combinations of adhesive and insulator that were not statistically expected to lead to high strength.

**Environmentally exposed single lap joints**

The use of the DOE approach permitted limits on the number of adhesives to be considered in the next step of the experimental work. With the environmental exposure, the focus was on specimens prepared with adhesives A, B, D, F, and G, since they were either the ones then in current use by the IJ manufacturers or that had shown the most promising results from the tests on as-produced SLJ specimens. It is important to note that environmental exposure significantly lowered the strength of the joints, as indicated in Figure 70. The results seem to be directly linked to the state of the surface of the bond after the exposure: the degradation due to oxidation of the steel surface undermines the load capacity of the joint. The systems less affected by the exposure appear to be ones in which the metal surface of the bonded system has been protected from corrosion by the adhesive layer.

Visual inspection of the failed specimens after testing and the values of apparent shear strength highlighted several aspects. For specimens prepared with adhesive D, the failure mode changed from a total adhesive/fabric interface to a partial adhesive/metal interface (especially on the outer ends of the bonded area). Nevertheless, the surface was still clean and without apparent rust, excepts for a few spots of oxidation that appeared in the 6-week exposed specimens prepared with Kevlar®. The joint strength was not drastically reduced by the exposure for specimens prepared with adhesive D. Much different behavior was found in the specimens made with adhesive F: they failed at the adhesive/fabric interface before exposure, but exhibited adhesive/metal interface failures after just 2 weeks of environmental exposure, and showed considerable surface oxidation after 6 weeks. The specimens bonded with adhesive B usually exhibited adhesive failure at the adhesive/metal interface, although some cases could be described as partial adhesive/metal interface failures. The metal surface after rupture was still bright and showed no visible signs of oxidation after 2 weeks of exposure. Rust spots were evident after 6 weeks in the configurations with one insulator layer. Nevertheless, especially for constructions with dispersed fiberglass and one Kevlar® layer, a strength drop was not evident. The failure for specimens prepared with adhesive A, on the other hand, shifted
from a partial adhesive/metal interface in the specimens prepared with dispersed fibers to an adhesive/metal interface failure, which is accompanied by extensive oxidation of the metal surface in the specimens exposed to the thermal cycling. In the specimens prepared with one layer of insulator, although the failure is still partial adhesive/metal interface, the value of the apparent shear strength was reduced by the exposure. Finally, adhesive G also apparently resisted surface oxidation. Moreover, the failure mode remained a partial adhesive/metal interface mode, although sometimes failure occurred at the adhesive/fabric interface.

Generally speaking it seems that, upon environmental exposure, oxidation of the metal surface in the bonded area reduces the strength of the specimens. The systems which performed better after exposure were not necessarily the ones which had better performance in the as-produced SLJ tests, but were the ones which better protected the metal surfaces from oxidation. Specimens bonded with adhesives D, B, and G maintained their apparent shear strength better throughout the exposure process for these SLJ tests. Figure 71 shows two examples of the effect of exposure on different samples.

**Double cantilever beam (DCB) specimens**

DCB tests were conducted for adhesives A, B, and D; the first two were chosen since they were in current use, and the third because it performed better in the as-received SLJ tests and also maintained high strength in the exposed SLJ tests. No insulator material was used for the DCB tests, since previous experience showed that the fibers can bridge the growing crack and significantly affect energy dissipation in the specimen, complicating the fracture analysis. The results refer to sets of 3 replicates for each material system, which was tested in three conditions: as-produced specimens, environmentally exposed specimens, and environmentally exposed specimens prepared with metal adherends that had undergone APS derivatization after grit blasting. The tests are preceded by precracking the bonded specimens by inserting a wedge between the two metal adherends, thereby initiating a crack as required for fracture tests.

The DCB tests revealed that the relatively thin (6.3 mm) adherend thickness choice of this high strength steel was appropriate, as there was no evidence of adherend yielding during testing, which would have complicated the fracture analysis. Also, the welded
tubing mounting solution worked well and should be seen as an alternative mounting method for difficult-to-machine adherends. The results are shown in Table 6.

In as-produced specimens prepared with adhesives A and B, the mode I critical strain energy release rate ($G_{ic}$) values were lower than $1000 \text{ J/m}^2$, while the average for specimens prepared with adhesive D was over $1500 \text{ J/m}^2$. Due to stick-slip behavior encountered for adhesive B during crack propagation, the data points were far fewer than for the other adhesives, reducing the statistical certainty for the fracture energy for this material system. The phenomenon is illustrated in Figure 72, which contrasts the pronounced stick-slip nature of adhesive B with the stable propagation noted for a specimen bonded with adhesive A.

For DCB specimens, the environmental exposure consisted in the first two-week-long part of the hot/humid condition and the first week of cycling illustrated in Figure 67. Rather than reducing the fracture energy, the exposure to humid conditions increased the measured $G_{ic}$ values for all three bond systems. Although the opposite trend is often seen for bonded joints with high-energy adherend surfaces, this modest improvement may have resulted because the exposure was too short to produce significant oxidation, even in the non-bonded portions of the adherends. In fact, and differently from SLJs, the bonded surfaces were still bright after testing. An aspect that may have influenced the result is that the dimensions of the DCB specimens did not allow water to propagate throughout the adhesive and degrade the bond in the total 3 weeks’ exposure time that was allowed. In fact, considering that the minimum width of the bonded area is about $25.4 \text{ mm}$ in the DCB and only $12.7 \text{ mm}$ in the SLJ, one should expect an equilibration time 4 times greater in the DCBs compared to the SLJs, applying Fick’s diffusion law. Other factors that may have had an effect are that the metal used for the DCB specimens was different from the one used for SLJ specimens and that water may reduce the brittleness of the adhesive, since water can act as a plasticizer in many polymers.

The APS derivatization did not have a well-defined effect. For APS-treated adherends bonded with adhesives A and B, the strength after exposure was even slightly reduced, while the strength increased in specimens prepared with adhesive D. Because of the absence of oxidation of the metal surfaces also in non-treated DCB specimens, it was impossible to evaluate the effectiveness of derivatized surfaces. The effect of APS
derivatization can be checked in the wedge tests, where exposure, possible oxidation, and crack growth are contemporaneous aspects.

**Wedge specimen tests**
This test involves simultaneous application of both stress and environment, usually allowing a better interrogation of adhesion with short-term exposure [158]. The wedge tests were conducted with specimens prepared with adhesives A, B, and D. In this test the external load given by the wedge insertion and the environmental exposure are applied simultaneously, and the specimens were tested under two different conditions: at 80°C with either dry or 100% relative humidity conditions. The findings presented in Table 7 are the average crack lengths for the five samples tested under each condition for the three adhesives and the two silane surface modifications. The crack length was measured from the points of contact between the wedge and metallic parts of the specimen. The wedges had a thickness of 2 mm for specimens prepared with adhesives A and D; with specimens prepared with adhesive B, the wedge had to be of 1 mm thickness, since the 2-mm thickness caused complete failure of the specimen during the initial stage of the tests, suggesting inferior performance.

Ultimate crack length data versus time were collected through visual inspection of the specimens at periodic intervals, and the results are shown in Table 7. The length of the wedge specimens was 150 mm. In each of the specimens, the wedge was inserted between the steel plates and then pushed between the steel plates to a distance of about 10 mm using a furniture clamp. Thus, values of ultimate crack length 140 mm indicate that the specimen completely debonded during the test. The value of the strain energy release rate was calculated with the equation

\[ G = \frac{9EIA^2}{4ba^4} \]

where \( G \) is the strain energy release rate, \( E \) the elastic modulus of the adherends, \( I \) the second area moment, \( A \) the wedge thickness, \( b \) the specimen width, and \( a \) the crack length from the crack tip to the points of contact between the wedge and the adherends.

The results of this study demonstrate that both grit blasting and surface derivatization of the steel surfaces enhance adhesive bond durability, especially for the currently used adhesives A and B. The success of derivatization of railroad steel surfaces appears to be
strongly dependent on the nature of the silane reagent used; APS was more effective at modifying the surface than the secondary amine silane (TMS). Limited experiments were carried out with the TMS reagent, and it is possible that optimization of the derivatization procedure could enhance the performance of TMS-modified steel bonded with all three adhesives. In alumina grit blasted railroad steel wedge specimens, the options bonded with adhesive A seemed to be superior to the options bonded with adhesives B or D. Moreover, the durability of specimens bonded with adhesive A was not influenced by the exposure conditions, since the time to failure at 80°C dry and at 80°C 100%RH was the same. The durability results of adhesive A in wedge tests are quite impressive; the already high level of durability without derivatization increased by nearly a decade with the APS derivatization. This seems to indicate that the derivatization, which is a process that can be easily industrialized, could be used with a traditional adhesive to enhance the durability of insulated joints.

Unfortunately, adhesive D, which was the most promising adhesive based on the SLJ and DCB tests, did not perform well in the wedge specimen tests. The surface derivatizations were not as beneficial as with the other adhesives. Adhesive B was also quite weak, being unable to withstand tests with a 2mm thick wedge. Nevertheless the two steel derivatizations also increased the durability of specimens prepared with adhesive B. The durability of real IJs may be considerably increased, especially in the applications that already use adhesive B.

A.8 Conclusions

The standard adhesion tests performed in this project permitted evaluation of numerous combinations of adhesives, insulators, and surface treatments that are of current or potential use for insulated railroad joints (IJs). Single lap joint (SLJ), double cantilever beam (DCB), and wedge tests were performed on specimens that were in some cases also environmentally conditioned before testing in SLJs and DCBs and during testing for wedge specimens. The choice of the tests to perform and the environmental exposures to apply was a compromise influenced by an understanding of typical IJ configurations and service conditions. The necessity of evaluating numerous combinations of adhesives, insulators, and surface treatments was also a consideration.
The IJs are subjected to both static and dynamic loads and to environmental conditions that can be extreme and highly variable, and the different tests permitted a focus on different aspects of the conditions to which the real IJs are subjected. Static load properties were investigated with SLJs, fracture properties with DCBs and wedge specimens, environmental exposure with the wedge tests, and high temperature and humidity plus freeze-thaw cycles with the SLJ and DCB specimens. These tests each provided a different perspective on the potential performance of these bond systems in IJ applications, although none of them captured the complicated loading present in a real IJ. Implementation of a new technique for mounting DCB specimens in loading clevises through the use of small tubes welded to the hard railroad steel adherends proved very successful, and avoided the expensive and time consuming procedure of drilling and threading the beams for mounting traditional end blocks. Reducing time and costs for specimen preparation, and testing was also an important aspect, since the analysis involved several combinations of adhesive, fibrous insulators and surface treatments. This reduction was obtained by applying the DOE approach and substituting the machining phase for the endblocks usually performed on DCBs with the welding of the tubes for accommodating pins for loading.

The different tests were not intended to give a unique indication in terms of the combination of adhesive, insulator, and surface treatment that guarantee the best overall performance for a real IJ. Nevertheless, it was observed that adhesive D had the best static strength, although it did not exhibit the best performance in other tests. Another result of this study is that the surface treatments have capabilities of increasing the strength of the joints, in SLJ, DCB, and wedge tests, especially limiting the negative effects that environmental exposure usually has on materials and surfaces. That chemical derivatization can dramatically enhance the durability of specimens prepared with traditional adhesives is quite evident and can be considered a positive outcome of the study. Further testing may be useful in clarifying and optimizing the gains in durability that seem to be possible with steel derivatization, as these tests could be implemented in current manufacturing facilities.
A.9 Acknowledgments

This research was sponsored by the Association of American Railroads (AAR). The authors thank Portec Rail Products, Allegheny Rail Products, 3M, Henkel North America, and Lord Corporation for providing the adhesives and the insulators that were tested. The authors thank Matthew A. Turner of the Civil and Environmental Engineering Department at Virginia Tech for following the environmental exposure of the specimens. Finally, we acknowledge the facilities provided by the Engineering Science and Mechanics Department and the interdisciplinary research environment fostered by the Macromolecules and Interfaces Institute.

A.10 Figures

Figure 64: Insulated joint (IJ) for rail applications

Figure 65: Schematic view of specimens: (a) single lap joint, (b) double cantilever beam, and (c) wedge
Figure 66: Detail of tubes welded on DCB specimens and paper ruler attached to facilitate crack length measurement.

Figure 67: Temperature profile of environmental exposure and tests for each set of environmentally exposed SLJs and, partially, DCBs (portions at 80°C have 100% relative humidity, portions at -25°C have no imposed humidity level).
Figure 68: Single lap joints tensile test results; adhesives A and B were in use by insulated joint manufacturers

Figure 69: Comparison of strength of single lap joints constructed with adhesive B
Figure 70: Apparent shear strength of SLJs after environmental exposure

Figure 71: Failure surfaces of SLJ specimens tested after three months of environmental exposure. Note the different level of oxidation of the two specimens. The one on the left (adhesive D with fiberglass insulator) maintained its initial strength; the one on the right (adhesive A with dispersed Kevlar®) lost more than 50% of the initial strength.
Figure 72: Load-displacement curves for DCB prepared with, respectively, adhesive A and adhesive B

A.11 Tables

Table 3: Insulator material characteristics

<table>
<thead>
<tr>
<th></th>
<th>Kevlar®</th>
<th>Fiberglass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>0.17 Kg/m² (5 oz/sq. yds)</td>
<td>0.617 Kg/m² (18.2 oz./sq. yds)</td>
</tr>
<tr>
<td>Weave</td>
<td>Plain</td>
<td>Plain</td>
</tr>
<tr>
<td>Thread count</td>
<td>7 x 7 per cm²</td>
<td>5.5 x 5.5 per cm²</td>
</tr>
<tr>
<td></td>
<td>(17 X17 per sq. inch)</td>
<td>(14x14 per sq. inch (*) )</td>
</tr>
<tr>
<td>Thickness</td>
<td>0.254 mm (0.010&quot;)</td>
<td>0.457 mm (0.018&quot; (*) )</td>
</tr>
</tbody>
</table>

(*) = measured quantity
### Table 4: Test matrix and results for tests on as-produced SLJ specimens

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Fabric Type / Number of Layers</th>
<th>Apparent Shear Strength, MPa</th>
<th>neat adh.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Kevlar</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>disp.</td>
</tr>
<tr>
<td>A</td>
<td>5.0 a</td>
<td>5.4 a</td>
<td>10.5 a</td>
</tr>
<tr>
<td>B</td>
<td>5.1 a</td>
<td>11.5 d</td>
<td>6.5 a</td>
</tr>
<tr>
<td>C</td>
<td>8.4 a</td>
<td></td>
<td>10.6 a</td>
</tr>
<tr>
<td>D</td>
<td>17.2 b</td>
<td>14.5 b</td>
<td>19.9 a</td>
</tr>
<tr>
<td>E</td>
<td>5.2 b</td>
<td></td>
<td>9.1 b</td>
</tr>
<tr>
<td>F</td>
<td>11.4 b</td>
<td>13.1 d</td>
<td>11.6 b</td>
</tr>
<tr>
<td>G</td>
<td>12.8 d</td>
<td></td>
<td>11.0 d</td>
</tr>
<tr>
<td>H</td>
<td>11.3 b</td>
<td></td>
<td>6.4 b</td>
</tr>
</tbody>
</table>

a = Adhesive/ Metal Interface failure  
b = Adhesive/ Fabric Interface failure  
c = Adhesive/ Adhesive failure  
d = Partial Adhesive/ Metal Interface failure

### Table 5: Results of DOE analysis for as-produced SLJ, showing results for apparent shear strength, in MPa, for specimens obtained with different insulators (on left) and with different adhesives, as overall (various insulator combinations) or with only one layer of insulator (on right)

<table>
<thead>
<tr>
<th>Insulator</th>
<th>Adhesive</th>
<th>overall</th>
<th>1 layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kevlar 0.3</td>
<td>A</td>
<td>-3.9</td>
<td>-5.2</td>
</tr>
<tr>
<td>Fiberglass -0.5</td>
<td>B</td>
<td>-1.6</td>
<td>-4.4</td>
</tr>
<tr>
<td>Neat adhesive 0.4</td>
<td>C</td>
<td>-1.5</td>
<td>-0.2</td>
</tr>
<tr>
<td>1 layer -1.9</td>
<td>D</td>
<td>7.7</td>
<td>8.9</td>
</tr>
<tr>
<td>2 layers -0.1</td>
<td>E</td>
<td>-4.5</td>
<td>-2.9</td>
</tr>
<tr>
<td>dispersed fibers 3.3</td>
<td>F</td>
<td>4.0</td>
<td>2.1</td>
</tr>
<tr>
<td>Kevlar 1ly -1.4</td>
<td>G</td>
<td>0.6</td>
<td>2.6</td>
</tr>
<tr>
<td>Fiberglass 1ly -1.2</td>
<td>H</td>
<td>-1.9</td>
<td>-0.9</td>
</tr>
</tbody>
</table>
Table 6: Critical strain energy release rate in DCB tests

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Exposure</th>
<th>Surface treatment</th>
<th>$G_{lc}$ (J/m²)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>as-produced grit blasting</td>
<td>410</td>
<td>107</td>
<td></td>
</tr>
<tr>
<td></td>
<td>env. exposed grit blasting</td>
<td>549</td>
<td>239</td>
<td></td>
</tr>
<tr>
<td></td>
<td>env. exposed grit blasting + APS</td>
<td>967</td>
<td>349</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>as-produced grit blasting</td>
<td>754</td>
<td>59</td>
<td></td>
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<td>env. exposed grit blasting</td>
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<td>128</td>
<td></td>
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<tr>
<td></td>
<td>env. exposed grit blasting + APS</td>
<td>898</td>
<td>110</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>as-produced grit blasting</td>
<td>1454</td>
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<td></td>
<td>env. exposed grit blasting</td>
<td>2249</td>
<td>770</td>
<td></td>
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<tr>
<td></td>
<td>env. exposed grit blasting + APS</td>
<td>3224</td>
<td>484</td>
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</table>

Table 7: Results of wedge specimen tests

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Test condition</th>
<th>Surface preparation</th>
<th>Wedge thickness (mm)</th>
<th>Ultimate crack length (mm)</th>
<th>Time at ultimate length (h)</th>
<th>$G_{li}$ at ultimate length (J/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>80°C dry</td>
<td>AGB</td>
<td>2</td>
<td>85</td>
<td>160</td>
<td>772</td>
</tr>
<tr>
<td></td>
<td>80°C – 100%</td>
<td>AGB</td>
<td>2</td>
<td>130</td>
<td>160</td>
<td>141</td>
</tr>
<tr>
<td></td>
<td>80°C dry</td>
<td>AGB+APS</td>
<td>2</td>
<td>85</td>
<td>1200</td>
<td>772</td>
</tr>
<tr>
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<td>80°C – 100%</td>
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<td>2</td>
<td>110</td>
<td>1200</td>
<td>275</td>
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<tr>
<td></td>
<td>80°C dry</td>
<td>AGB+TMS</td>
<td>2</td>
<td>90</td>
<td>480</td>
<td>615</td>
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<tr>
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<td>AGB+TMS</td>
<td>2</td>
<td>110</td>
<td>480</td>
<td>275</td>
</tr>
<tr>
<td>B</td>
<td>80°C dry</td>
<td>AGB</td>
<td>1</td>
<td>140*</td>
<td>22</td>
<td>&lt;26</td>
</tr>
<tr>
<td></td>
<td>80°C – 100%</td>
<td>AGB</td>
<td>1</td>
<td>140*</td>
<td>22</td>
<td>&lt;26</td>
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<tr>
<td></td>
<td>80°C dry</td>
<td>AGB+APS</td>
<td>1</td>
<td>75</td>
<td>1200</td>
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<tr>
<td></td>
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<td>1</td>
<td>130</td>
<td>1100</td>
<td>35</td>
</tr>
<tr>
<td></td>
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<td>AGB+TMS</td>
<td>1</td>
<td>75</td>
<td>500</td>
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</tr>
<tr>
<td></td>
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<td>AGB+TMS</td>
<td>1</td>
<td>110</td>
<td>500</td>
<td>69</td>
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<tr>
<td>D</td>
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<td>140*</td>
<td>160</td>
<td>&lt;105</td>
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<tr>
<td></td>
<td>80°C – 100%</td>
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<td>2</td>
<td>140*</td>
<td>20</td>
<td>&lt;105</td>
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<tr>
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<td>160</td>
<td>&lt;105</td>
</tr>
<tr>
<td></td>
<td>80°C dry</td>
<td>AGB+TMS</td>
<td>2</td>
<td>140*</td>
<td>300</td>
<td>&lt;105</td>
</tr>
<tr>
<td></td>
<td>80°C – 100%</td>
<td>AGB+TMS</td>
<td>2</td>
<td>140*</td>
<td>300</td>
<td>&lt;105</td>
</tr>
</tbody>
</table>

* denotes complete debonding
AGB = alumina grit blasting, APS = APS derivatized, TMS = TMS derivatized