**Characterization of mixed-mode I/II fracture properties of adhesively bonded yellow-poplar by a dual actuator test frame instrument**

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**Abstract**

Experimental results for the fracture behavior under mixed-mode in-plane loading conditions of adhesively bonded wood specimens are reported. The material systems considered involved yellow-poplar (*Liriodendron tulipifera*), a hardwood of the Magnoliaceae family, as adherends bonded with two different adhesives, a moisture-cure polyurethane (PU) and a phenol/resorcinol/formaldehyde (PRF) resin. A dual actuator test frame permitted fine scanning of fracture behavior over a full range of mixed-mode I/II levels for double cantilever beam (DCB) geometry specimens. These tests showed that, in the considered material systems, the critical strain energy release rate, $G_c$, tends to increase as the mode-mixity of the loading increases. In particular, the increase is steeper in proximity to pure mode II loading for the PRF bonded specimens. The experimental values of $G_c$ obtained were fairly scattered, as is common when testing wood systems. This variability is due in part to the natural variability of wood but also to other factors such as the orientation of the grain in the bonded beams and variations of bondline thickness. In particular, measurements of adhesive layer thickness were performed. This analysis was implemented with microscopic examination of samples cut from untested DCB specimens, where the bondline had not been disrupted by the test. Although the wood parts were power planed prior to bonding, rather large variations of the adhesive layer thickness were observed: on the order of 1–100 µm for specimens bonded with the PU resin and 10–50 µm for specimens bonded with the PRF resin, which showed somewhat more consistent fracture behavior.

**Keywords:** adhesive joints; bonded wood; double cantilever beam (DCB) geometry; dual actuator; fracture, fracture I and fracture II, mixed-mode, bondline thickness; PRF resin; PU resin, yellow poplar.

**Introduction**

Wood has unique characteristics among natural materials in terms of its strongly anisotropic nature, providing good ratios in terms of strength to weight and stiffness to weight in the grain directions and relative ease in splitting parts perpendicularly to the grain direction. In addition, it is relatively easy to saw. This is the reason why wood is also a perfect construction material for homes (Wood Handbook 1999; Bowe 2001). There is a large body of literature concerning fracture properties of wood and bonded wood, focusing on mode I fracture (opening or tensile mode) (Ebewele et al. 1979, 1980; Triboulot et al. 1984; Gagliano and Frazier 2001; Conrad et al. 2003; Zheng et al. 2004; de Moura et al. 2008) or on mixed and mode II fracture (in-plane shear mode) (Cramer and Pugel 1987; Yoshihara and Ohta 2000; de Moura et al. 2006, 2011; Oliveira et al. 2007, 2009; Singh et al. 2010). The fracture behavior of wood and wood composites was also reviewed by Stanzl-Tschegg and Navi (2009) and the modeling crack propagation was reviewed by Landis and Navi (2009).

Fracture properties of bonded wood are affected by a large number of factors, such as wood structure, adhesive penetration into wood, and initial roughness of bonded surfaces as recognized early by Ebewele et al. (1979, 1980). Since that time, this topic has remained a matter of investigation, as demonstrated by a few recent quotations concerning the adhesive penetration and the characterization of the bonding performance by various methods: scanning thermal microscopy (Konnerth et al. 2008), image analysis, nanoindentation (Stöckel et al. 2010), air-coupled ultrasound inspection (Sanabria et al. 2011), or by macro- and micromechanical characterization (Follrich et al. 2010; Sahaf et al. 2011), just to mention a few.

A large scatter of values of critical strain energy release rate, $G_c$, of bonded wood has been often seen and regarded as resulting from the intrinsic variability of properties difficult to monitor in natural materials (Triboulot et al. 1984; Zheng et al. 2004; Kamke and Lee 2007). By contrast, a recent analysis has identified grain orientation effects as a structural, and possibly accountable, aspect as one of the factors that increase the scatter of $G_c$ data (Nicoli et al. 2012a,b).

The possibility of performing mixed-mode tests employing a dual actuator load frame (Singh et al. 2007, 2010; Chaves...
et al. 2011) has facilitated mixed-mode testing of bonded systems. This testing frame permits one to impose asymmetric loading conditions with two independently controlled actuators. Depending on the specimen type and its mounting onto the machine, loading conditions ranging from pure mode I to pure mode II can be easily achieved.

In the analysis of double cantilever beam (DCB) and other standard bonded beam 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 et al. (1991, 2006) and Brunner et al. (2001). These techniques address several limitations of simple beam theory by including 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 (Williams 1989; Blackman et al. 1991; Davies et al. 1992; Cotterell et al. 2006). The CBT method evaluates the effective compliance of the DCB beams and corrects the crack length reading using a linear fit of the cube root of compliance vs. crack length data. The ECM similarly evaluates elastic characteristics of the tested specimens with a power law 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 (Wang and Williams 1992; Blackman et al. 2003, 2005, 2006). One goal of this study was to test mixed-mode conditions, but each specimen was partially tested at the same level of mode-mixity, namely pure mode I. This allowed for specimen calibration using the ECM and CBT methods, and allowed mixed mode testing to begin with a longer crack length, minimizing instability issues (Davies et al. 2001). With this condition, the tests proceeded through three separate phases; the first and the third were conducted in mode I and the second phase involved mixed-mode conditions obtained by imposing different displacement rates to the two beams. As ECM and CBT fit data of compliance with crack length, having portions of mode I tests performed with both short and long crack lengths (corresponding to the first and third loading phases) was deemed to be important for an accurate evaluation of the parameters defined by the two fitting procedures. Note that both CBT and ECM assume the elastic characteristic of the bonded beams to be uniform and constant along the beam length, as expected for a homogeneous material. For wood this assumption is not always correct, as explained by Liswell (2004). Nevertheless, ECM and CBT have been traditionally applied for measuring the fracture properties of bonded wood (Gagliano and Frazier 2001; Yoshiiha and Nobusue 2007). The effects of this assumption have been reviewed in the literature (Nobili et al. 2012a,b).

This paper describes a procedure that was applied for evaluating $G_c$ values in mixed-mode I/II loading conditions using DCB geometry specimens in a dual actuator load frame and the experimental results that were obtained. Yellow-poplar (Liriodendron tulipifera) was selected for the adherends and two different commercial adhesives were applied: a moisture-cure polyurethane (PU) and a phenol/resorcinol/formaldehyde (PRF) resin.

### Materials and methods

#### Experimental equipment

The dual actuator load frame (DALF) (Singh et al. 2006, 2010) 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 those widely used for DCB specimens. In principle, the investigation 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 DALF, each actuator is independently controlled and is equipped with a load cell and a displacement sensor (LVDT) that provides information for quantitative evaluation of specimen behavior.

Features of DALF include the ability to test a common bonded joint specimen configuration over the full range of mode I/II mixity by independently controlling the actuator motion, the ability to quickly change mode mixity on a single specimen without use of an additional fixture or modifying the test jig, and the ability to test specimens under general loading conditions spanning a wide range of displacement speeds and in fatigue. The DALF, illustrated in Figure 1a, was built by McGaw Technologies Inc. (Fairview Park, OH, USA). 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 or forces for the two actuators are recorded as the test progresses by a computer equipped with a data acquisition card (DAQ PCI 6229, National Instruments Inc., Austin, TX, USA). The crack length values of the specimen are read periodically by the operator using a magnification lens and facilitated by white correction fluid and a paper ruler applied on one side of the specimen.

The values of crack length are entered into the data acquisition system. The simplest control algorithm imposes different combinations of displacement rates to the two actuators as described in Nicoli (2010). The combination of different displacement rates results in different levels of in-plane mode mixity with a standard DCB specimen. Microscope analysis was performed with a Zeiss Axioskop (Carl Zeiss, Oberkochen, Germany) microscope, the camera for image acquisition is a DS-Fi1 (Nikon Inc., Melville, NY, USA). For preparing microscope samples, some of the DCB specimens were not tested on the DALF, but cut into microscope samples, which are 40-μm thick slices of area approximately 5×5 mm. The slices were also stained with two different solutions, 0.5% safranin O or 0.5% toluidine blue O, 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.

#### Materials and specimens

The study focuses on yellow-poplar (L. tulipifera), a hardwood of the Magnoliaceae family, and two adhesives, a moisture-cure PU and a two-component PRF resin. Specimens consisted of 10-mm thick wooden adherends bonded together with one of the two adhesives. Preparation of the specimens started with two power-planed 10-mm thick boards with dimensions of approximately 250 mm×140 mm. 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 (Gagliano and Frazier 2001). This configuration is beneficial in preventing the crack from moving from the bonded
layer into the adherend substrate and because the 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 excluded from experimentation.

A 50-mm wide region of the board near one of the edges was colored with a wax crayon to limit adhesion and provide a precracked area in the final bonded specimens. One board is illustrated in Figure 1b. To bond the 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 were as specified 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 PF resin and 0.68 MPa (100 psi) for the PRF resin, was applied for at least 24 h.

After curing, the bonded pieces were cut into 20-mm wide specimens and conditioned for at least 2 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 for loading clevis pins (see experimental equipment section). Next, one side of the bonded specimens was painted with white typewriter correction fluid to enhance crack tip detection 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 1c. The definition of axis and section directions in the wood for our specimen is illustrated in Figure 1d. Note that the longitudinal axis is not parallel to the specimen cut because of the 3–6° orientation just described.

**Test method**

With the dual actuator load frame, the standard 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. Given the utility of performing part of the test in pure mode I, the displacements imposed during the tests were as shown in Figure 2. A total of 33 specimens prepared with the PRF adhesive and 28 specimens prepared with the PU adhesive were tested. Some tests were run in pure mode I or pure mode II, but typically mixed-mode loading for the second loading phase was performed for most specimens, following profiles such as illustrated in Figure 2. The resulting critical fracture energy, \( G_c \), values can each be represented as a sum of mode I and mode II components, with the relative contributions depending on the mode-mixity angle, \( \psi \). The relations between \( G_c \), its mode I and mode II components and the angle \( \psi \) are shown in Eqs. (1) and (2).

\[
G_c = (G_{ci})_I + (G_{ci})_II
\]
These mode I and mode II components of a given $G_c$ are not to be confused with the critical fracture energies for mode I and mode II, $G_{cI}$ and $G_{cII}$, respectively. Various methods for partitioning the mode I and II components of the loads have been proposed and can lead to different results. As suggested by Williams (1988), a method based on the global analysis was chosen for this study. In particular, the partitioning of loads was based on the applied moments (Hutchinson and Suo 1991; Kinloch et al. 1993). In the case of the dual actuator load frame, the mode I and mode II components of the loads applied to each beam can be easily calculated as a function of the loads applied by the two actuators (Nicoli et al. 2009).

The two components $F_I$ and $F_{II}$ are indicated in Eqs. (3) and (4), where $F_R$ and $F_L$ are, respectively, the force readings at the right and left actuator and are positive when the actuator is pulling the beam.

$$F_I = \frac{1}{2}|F_R + F_L|$$

$$F_{II} = \frac{1}{2}|F_R - F_L|$$

**Results and discussion**

The curves, in Figures 3 and 4, show the results for the two material systems. The trends of the mode I and mode II components of $G_c$ as well as the total $G_c$ are visible. With the tests performed at constant displacement rates, the data of the single specimens are presented as a function of $\psi$, as the mode-mixity angle increased during the test, as analytically shown (Nicoli 2010).

The significant data scatter in Figures 3 and 4 is often common for wood and bonded wood fracture tests (Ebewele et al. 1979, 1980; Triboulot et al. 1984). In particular, the fracture envelopes in Figure 4 illustrate that the individual specimens are relatively consistent, but that specimen-to-specimen variability is considerable. In particular, within an individual specimen the scatter seems, in most cases, to have some trends, though not necessarily consistent with
the trends of other specimens tested at the same levels of mode-mixity. This aspect can be interpreted that the scatter is probably not related to random variability or noise-like disturbances during the test, but may depend on specimen or bond variability.

Moreover, the variability also seems not to be related to R-curve effects that are seen in some material systems (Irwin et al. 1954), as the observed trends of $G_c$ are not, for example, always monotonic, i.e., are not always rigorously dependent of $\psi$. Figure 3b 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, whereas for higher $\psi$ the $G_c$ increases rapidly. $G_c$ for mode II loading is approximately eight times larger than that in mode I loading.

The fracture envelope of Figure 4a shows that data scatter is more pronounced for tests performed close to mode I loading. Note that the horizontal and vertical axes in Figure 4a have different scales. Figures 3a, b, and 4b show the results of PU-bonded specimens. Also in this case, most of the observations are similar to the results of the PRF-bonded specimens including the appreciable data scatter. The latter is due to the systematic differing trends of $G_c$ depending on the specimen tested rather than some random variability. Also in this case, the value of $G_c$ is almost constant for $\psi$ below 30° and then increases, as can be seen in Figure 3b. The values of $G_c$ associated with pure mode I loading are fairly similar for the two material systems, whereas for mode II loading the $G_c$ of the PRF-bonded DCBs is approximately twice that of the PU-bonded specimens. For mixed-mode I/II (Figure 5) the two material systems have very similar behavior for $\psi<70–75^\circ$. One positive outcome of the results was that the grain orientation has successfully prevented the crack from moving into the adherends even for tests performed with mode II loading and all failures were observed to occur within the bond region.

**Visual analysis of tested specimens**

The common visual inspection of failure surfaces often permits the recognition of regions associated with adhesive or cohesive failure. This simple procedure is not particularly useful in bonded wood samples, however, 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, the failure surfaces have non-uniform characteristics, which sometimes alternate between smooth and rough areas (for the PU-bonded systems) or lighter and darker areas (for the PRF-bonded systems), suggesting different failure mechanisms. Regions in which the adhesive plastically deforms before failing require more energy than regions in which the failure is brittle and, particularly for specimens prepared with PU adhesive, a connection was observed between the roughness of the bonded surface after failure and the recorded $G_c$. Figure 6 is an example on how $G_c$ varies in a test performed with pure mode I loading on one of the specimens prepared with the PU adhesive. $G_c$ varies between 300 and 700 J m$^{-2}$, although 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 $G_c$ is a property of the tested material system.

A monotonic trend for the failure of $G_c$ can, by contrast, be an indication of the dependence of $G_c$ on the crack growth rate, in view of the fact 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, whereas reduced $G_c$ values correspond to smooth portions of the surface. In the example illustrated in Figure 6, particularly rough failure surface areas were obtained at different portions of the failed surface. The different trends in the graph are interpretable that $G_c$ is not influenced by other factors such as the crack length or speed, which change monotonically during the test.

**Effect of adhesive layer thickness**

Further analysis was performed on the state of the adhesive layer to detect possible causes of the scatter of experimental results. The adhesion mechanisms in wood samples are generally very different from what occurs in metal or composite samples. Adhesive penetrates considerably deeper into the wooden beams and the bondline thickness is very thin (Wood Handbook 1999). Moreover, during the production of bonded wood specimens, pressure applied to the pieces to be bonded is controlled rather than the bondline thickness. However, the latter is a factor that influences the extension of the plastic zone and the value of $G_c$ (Kinloch and Blackman 2004). Thus, the variability of the bondline thickness can lead to data scatter, as found in the experimental results previously illustrated.

The procedure for measuring the bondline thickness was based on methods reported in the literature (Johnson and Kamke 1992; Zheng et al. 2004; Kamke and Lee 2007). The analysis was performed with a fluorescence microscope working with transmitted light (see Material and methods section). Some of the samples for microscopic analysis are shown in Figure 7a. The transmitted light microscope for checking bondline penetration needs thin samples that permit light to be transmitted through. This aspect complicates the measurements. 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 because the initial thin layer has been disrupted during the test. In the second case, the cutting of the sample destroys the DCB specimen. Consequently, 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 presented in Figure 7b,c. 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, as with the safranin stain both adhesive and wood exhibit a reddish coloration. Adhesive in the wood vessels is visible because of its slightly pink color. The results show that the values of adhesive layer thickness and penetration in the substrate vary considerably, even within a single specimen. Although all the wood parts for DCB tests were power planed before bonding the specimens, the thickness of the PRF-adhesive layer varies between 10 and 50 μm and the penetration between 0.1 and 0.75 mm. The thickness of the PU-adhesive layer varies between 1 and 100 μm and the penetration between 0.3 and 1.5 mm. The reason for greater variation in PU-adhesive thickness is not clear, but could possibly be associated with the different viscosity of the two adhesives. 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 type 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 (Figure 1b). 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

![Figure 6](image-url) Failure surfaces and local $G_c$ for PU-bonded specimen tested in mode I.
relation between adhesive layer thickness and measured local value of $G_{IC}$ was apparent, as already seen in different material systems (Kawashita et al. 2008; Qian and Sun 2008).

An example is given in Figure 8. Here, the value of adhesive layer thickness is lower than 20 μm for sections where the value of $G_{IC}$ is around 200 J m$^{-2}$, whereas the thickness is around

Figure 7  Light microscopic analyses of the bonds. (a) Slices for microscope analysis ready to be tested (from left to right: toluidine stained, unstained, safranin stained). (b) Images for PRF-bonded untested DCB (unstained sample, radial-tangential section). (c) Images for the PU-bonded untested DCB (stained sample, radial-tangential section).

Figure 8  Local adhesive layer thicknesses and $G_{IC}$ values as a function of crack length for a PU-bonded DCB specimen tested in mode I.
50 μm in sections where the value of $G_{IC}$ peaks to almost 500 J m$^{-2}$. The dependence of the $G_{IC}$ on the adhesive layer thickness can be explained in the following way. 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 $G_{IC}$ value is expected to grow, as more adhesive is allowed to plastically deform ahead of the crack tip. The increase of $G_{IC}$ is not indefinite as the effect of the interfaces fades and $G_{IC}$ reduces to the value for the bulk polymer (Kinloch and Shaw 1981; Kinloch and Blackman 2004), if the layer thickness is large enough compared to the radius of the plasticized zone. In bonded wood, the thickness of the adhesive layer is particularly small, as demonstrated by the measurements. 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 $G_{IC}$ in static tests, as shown in Figure 8.

**Conclusions**

This study investigates the mixed-mode I/II fracture properties of yellow-poplar adherends bonded with a moisture-cure PU or a PRF resin. DCB specimen configurations were tested in a dual actuator load frame. The experimental procedure consists of three separate phases, two of which were mode I tests. This approach considers some aspects of the CBT and ECM in the context of the mixed-mode test results. This 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 $G_{IC}$ versus $\psi$ and fracture envelopes show 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 PU-adhesive layer thickness could vary between 1 and 100 μm and between 10 and 50 μm for specimens prepared with PRF resin. Comparison of adhesive layer thickness and mode I critical fracture energy is interpreted that the thickness variation has a strong influence on $G_{IC}$ that is measured with the DCB samples. In particular, sections of reduced adhesive layer thickness were associated with lower fracture energies and vice versa.

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