Long-term Durability Characterization and Prediction of a Urethane-based Adhesive

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Abstract

Polymeric adhesives play an increasingly critical role in today’s engineering designs. When used, adhesively bonded components reduce or eliminate the need for bolted or welded connections. In many cases, this can reduce stress concentrations and weight. With energy dissipating adhesives, noise and vibration reduction are possible, as is the use of unique or complicated designs that could not otherwise be constructed. Adhesive properties however, can vary greatly with time, temperature, and environmental exposure conditions such as moisture. It is therefore critical to understand the behavior of adhesives over the range of conditions that a bonded component might experience. In this work, the behavior of a urethane-based adhesive was characterized and long-term durability predictions were developed as a result of the data collected.

The popular T-peel sample geometry has been used extensively in this study to explore the mechanics of a bonded system and the resulting impact on adhesive durability. The T-peel specimens used, consist of two aluminum sheets or adherends bonded together, with tabs bent back in the shape of a “T” for gripping in a universal load frame. Unlike some other test geometries, T-peel samples are often made with relatively thin adherends that may experience significant plastic deformation during testing. This extraneous energy dissipation greatly complicates the analysis to extract meaningful fracture properties of the adhesive. During testing, the load required to propagate a crack in the adhesive layer is measured at fixed displacement rates. The total system energy can then be partitioned into the energy dissipated within the adhesive (fracture energy), and the energy dissipated through plastic work in bending of the adherends. By performing these tests at different temperatures and rates, the calculated fracture energies span a wide range of possible material behavior. Using the principles of Time Temperature Superposition (TTS), the collected data can be shifted to different times or temperatures. This behavior is well understood in polymer physics, and is made possible with material specific “shift factors”. By using the principles of TTS, data collected in a relatively short experimental window, can be used to accurately predict the behavior of the adhesive in years or even decades.

In this work, nearly 200 T-peel samples were tested in four different studies. A preliminary set of unaged specimens was used to develop testing and data analysis methodologies. A second set of unaged samples was tested over a wide range of temperatures and rates, in addition to a third group, subjected to constant moisture and cyclically varying temperature. The final set of specimens, was exposed to 20 separate isothermal aging conditions. The experimental data showed that the 400+ cycles, were insufficient to statistically distinguish these samples from their unaged counterparts. Additionally, samples aged for up to 2000 hours in a dry environment, or 500 hours in a wet environment, showed no reduction in fracture energies in comparison with unaged samples. Specimens aged for more than 500 hours however, were observed to have a significant decrease in fracture energy values.
Strong correlations between the thickness of the adhesive layer and estimated fracture energy values were found in this study. As adhesive thickness varied substantially due to manufacturing differences in the specimens tested, new analysis techniques were developed to deal with the variations in adhesive thickness. A MATLAB code based on the ICPeel program was written to provide a spatial variation of parameters such as adhesive thickness, peel load, and fracture energy. This provided additional insights into the behavior of these T-peel coupons, and prompted the investigation of the Universal Peel Diagram concept. While this diagram was not found to be applicable to the adhesive tested in this study, the analysis indicated that T-peel coupons could be multivalued. That is, a single measured load value does not always describe an adhesive’s fracture energy (as is widely believed). Depending on the sample’s geometry and material properties, several measured loads could cause debonding. This has potentially far reaching implications on the selection of appropriate T-peel test geometries, as a single measured load is often assumed to correlate to an adhesive’s true fracture energy.

In this work, both aged and unaged T-peel specimens were tested and the basis of the Universal Peel Diagram investigated. Given sufficient exposure times to moisture, elevated temperatures were found to significantly reduce the amount of energy dissipated in the urethane-based adhesive. Additionally, the Universal Peel Diagram indicated that for some systems, the load required for debond is in fact, multivalued. Therefore, care should be taken when designing a T-peel test configuration to avoid the multivalued regions.
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General Audience Abstract

Polymeric adhesives play an increasingly critical role in today’s engineering designs. When used, adhesively bonded components reduce or eliminate the need for bolted or welded connections, reducing their weight in the process. With adhesives, noise and vibration reduction are possible, as is the use of unique or complicated designs that could not otherwise be constructed. Adhesive properties, however, can vary greatly with time, temperature, and other environmental exposure conditions such as moisture. It is therefore critical to understand the behavior of adhesives over the range of conditions that a bonded component might experience. In this work, the behavior of a urethane-based adhesive was characterized in order to develop long-term durability predictions.

Numerous test methods have been developed to characterize the behavior of adhesively bonded joints. In this work, T-peel specimens were used consisting of two aluminum sheets (the adherends), bonded together with tabs bent back in the shape of a “T” for gripping in a universal load frame. During testing, the load required to propagate a crack in the adhesive layer is measured. An outcome of this measurement and subsequent data analysis is the fracture energy—a measure of the effectiveness of the adhesive in transferring loads. If we perform these tests at different temperatures and loading rates, we can determine fracture energy values which span a wide range of possible material behavior. Using principles from basic polymer physics, the collected data can be shifted to different times or temperatures enabling us to accurately predict the behavior of the adhesive over years or even decades.

In this work, nearly 200 T-peel samples were tested in four different studies. A preliminary set of unaged specimens was used to develop testing and data analysis methodologies. Unaged and cyclically (temperature) aged samples were tested over a wide range of temperatures and rates. The fourth set of specimens was subjected to 20 separate isothermal aging conditions and also tested at different temperatures and rates. The experimental data showed that the 400+ temperature cycles were insufficient to damage these samples significantly. Additionally, samples aged for up to 2000 hours in a dry environment, or 500 hours in a wet environment showed no reduction in performance in comparison with unaged samples. Specimens aged for more than 500 hours in a wet environment however, demonstrated a significant decrease in fracture energy values. Strong correlations between the thickness of the adhesive layer and estimated fracture energy values were found in this study, and new analysis techniques were developed to analyze the effect of these thickness variations on the joint performance.
Dedicated to Robert Stinebaugh. Role model, friend, and fellow engineer.
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Chapter 1 – Introduction and Background

Motivation

Adhesives are seeing an increasing role in today’s complex engineering designs, as they offer several key advantages over bolted or welded connections. Because adhesives allow bolted joints to be eliminated, stress concentrations around the fastener holes no longer exist and structural members can often be made smaller and therefore lighter. Reducing the weight of one member often has a snowballing effect, where the other members now support a smaller load, and can therefore be designed to be smaller and lighter. Eliminating bolted joints not only reduces material costs, but also lowers supporting costs as the entire structure can be lighter and often more aerodynamic. Other key benefits of using adhesives over bolted connections are noise and vibration mitigation as well as energy adsorption. This is particularly common in the automotive industry where adhesives not only help to reduce vibrations and increase ride quality, but also absorb energy in the event of a crash. Adhesives also allow dissimilar materials to be bonded along with unique geometries that would be difficult or impossible to obtain with bolted or welded components. In some cases, the use of mechanical fasteners is not feasible at all due to the geometry or weight restrictions. In addition to structural and energy adsorption considerations, the use of adhesive bonds can significantly reduce part count and simplify assembly efforts.

Adhesives are, however, not without drawbacks. Some adhesives are particularly susceptible to moisture, solvents, ultraviolet light, and extreme temperatures. They behave rubbery when hot and glassy when cold. The viscoelastic behavior of polymers complicates the engineering design, as both the time and temperature behavior of these materials should be considered during the design process. Manufacturing of bonded components can also be more difficult than their welded, riveted, or bolted counterparts. Adhesives typically have a finite shelf life, and often require careful surface preparation to obtain a good bond. Special equipment such as jigs, ovens, and presses are routinely required during the curing process. Additionally, adhesives are often purposely engineered for a particular application and their behavior needs to be carefully studied before they can be used in alternative applications.

In many cases, however, the advantages of using bonded components outweigh the more complicated design and manufacturing required. As with any engineering component, it is critical to understand the material behavior over the environmental conditions likely to be seen by the part. This is particularly true for adhesives, where the material properties change not only with time and temperature, but also with environmental factors such as moisture, solvents, or ultraviolet light. In this work, a urethane-based adhesive was studied. As the long-term behavior of this adhesive was not previously well characterized, the measurement of material properties needed for durability predictions were a major focus of this study. The material behavior after exposure to elevated temperatures and/or moisture was also of great interest, as these conditions can significantly impact the integrity of the bond. Because the part manufactured using this adhesive is expected to remain in service for decades or longer, accurate long-term durability predictions are critical to the design of a robust and reliable product.
Background

Adhesion has been defined as “the state in which two surfaces are held together by interphase forces” [1]. This broad definition can be refined by introducing two classes or categories of adhesion: specific and mechanical adhesion. Specific adhesion is the “adhesion between surfaces which are held together by intermolecular forces of a chemical or physical nature” [1]. The properties of the adhesive, adherends, and primer all contribute to specific adhesion. Here, an adherend is defined as “a body held to another body by an adhesive” [1].

Mechanical adhesion is the “adhesion between surfaces in which the adhesive holds the parts together by interlocking action” [1]. This class of adhesion depends on an adhesive’s ability to conform to, or penetrate into the adherends to create a physical link. Factors such as the adhesive’s viscosity, the roughness of the adherends, and surface prep all play a role in mechanical adhesion. Both specific and mechanical adhesion contribute to the overall adhesion of a bonded joint. As adhesion is dependent on the interaction between an adhesive and adherends, it is not a material property, but rather a system property. The degree of adhesion in a bonded joint, as well as the geometry, adherend stiffness, and environmental conditions, determine the mechanical performance of the joint.

Numerous mechanical tests have been developed in order to characterize the performance of bonded joints. Generally speaking, these tests can be broken down into two categories: strength tests and fracture tests. In strength tests, flaws are minimized and their influence ignored. In these types of tests, the samples are loaded until failure, and the strength (typically load at break/bond area) is reported. Common adhesive strength tests include those conducted on lap shear, butt, and cleavage joints.

In contrast, fracture tests intentionally introduce a dominant flaw into the adhesive material. Often this flaw is a machined notch or a sharp crack tip, but it can also be the start of an adhesive bond, as is the case in peel tests. In fracture tests, the load is measured as a crack is propagated from the dominant flaw. The measured load can then be used (often with required knowledge of the crack size) to calculate fracture energies for the adhesive bond. This fracture energy term describes the energy required to propagate a crack per unit debond area. Beam tests, both single and double cantilever, as well peel tests are common examples of fracture tests.

Fracture tests, specifically the T-peel test, has been the focus of this work. When a mechanical test such as T-peel must be performed, the ASTM standards are often the first choice, especially in industry, for testing procedures, sample geometry, and analysis methods. ASTM D1876-08 [2] defines a “standard test method” for T-peel tests. This standard defines T-peel strength as “the average load per unit width of bond line, required to produce progressive separation of two bonded, flexible adherends.” In part one, this test method is described as “determining the relative peel resistance” for a given adhesive and adherend combination. This peel strength cannot be used to characterize a particular adhesive, but can only be used to compare two adhesives with the same adherends and sample geometry. Often there are cases, however, when the test geometry cannot match that of the intended application.
The peel strength used in the ASTM standard does not take into account the fact that only some of the measured force is used for debonding. The remaining force, acting through a distance, dissipates energy by plastically deforming the adherends. T-peel specimens with sufficiently thin or ductile adherends will often exhibit large plastic deformations after unloading, highlighting the importance of accounting for this dissipated energy. The total system energy \( G_{\text{total}} \) often called practical adhesion, is the sum of the fracture energy \( G_c \) and plastic work in bending \( G_d \) contributions. Fracture or decohesion energy is used to describe the energy dissipated within only the adhesive. These fracture energy values are a direct measure of an adhesive’s integrity and can therefore be compared between different adhesives with a given pair of adherends.

Fracture energy, usually expressed in \([\text{J/m}^2]\), has the same base units as “peel strength.” The important distinction is that peel strength includes contributions from both the adhesive and adherends, while fracture energy is a true measure of the energy dissipated within the adhesive. As Kim and Kim [3] point out, the peel strength can be approximated as the fracture energy only if certain (very limited) conditions are met. For typical T-peel specimens with aluminum 2024-T3 adherends \((E = 72.4 \text{ GPa}, \sigma_y = 331 \text{ MPa})\) using the ASTM D1876 geometry \((t = 0.635\text{mm})\), load values would have to be 8.1 N or less for peel strength to be approximated as fracture energy. Note that this value is twice that of a 90° peel test, as the geometry is symmetric about the centerline for T-peel samples, so crosshead travel is doubled. There are, however, many adhesives that can hold significantly higher loads. Therefore, a method is required to determine what portion of the total system energy went into breaking the adhesive \((G_c)\), and what portion was used to plastically deform the adherends \((G_d)\). This partitioning of the total system energy is critical and has therefore been studied in considerable depth in literature.

One of the early peel tests was devised by Aero Research Limited and used by Spies [4] in 1953. In an effort to improve the quality control methods used in the manufacture of bonded aircraft components, a peel test was designed to replace the shear tests then in use. In this test, one adherend was fixed to a freely spinning wheel, while the other adherend was pulled at an angle perpendicular to the wheel. The bonded portion of the sample was modeled as a beam on elastic foundation, while the debonded portion was thought to act as an elastica. By recording the force required for debonding over the length of the sample, one could determine if the specimen met the minimum standard for average peel force. Spies described the stresses in the bonded portion of the strip, and the deformation of the debonded portion, but was unable to produce meaningful numerical results due to a number of assumptions about the plastic and elastic behavior of the samples. He did, however, note correlations between the average adhesive thickness and modulus as well as between peel strength, elasticity of the adhesive, the adhesive thickness, and adherend properties.

Other contributors to the analysis of peel tests were Chen and Flavin [5]. They examined steady-state peeling of a flexible adherend bonded to a rigid substrate using an elastic-plastic model. As a result, a computer program using an analytical model was developed and the results compared to experimental data. Although plastic bending of the adherends was considered by
Chen and Flavin, reverse bending which occurs as the adherend is straightened was not included in the analysis.

In [3], Kim and Kim examined the adhesion of very thin copper films in order to extract the fracture energy or interfacial “toughness” values and construct a universal peel diagram (UPD). This novel diagram allowed the fracture energy values to be found from the experimentally measured load and sample properties. The energy dissipated through plastically bending of the adherends and breaking of the adhesive was related to the stored elastic strain energy through an energy balance. Using 1-D slender beam theory, the deformation of an elastic-perfectly plastic adherend was described. Force and moment equilibrium equations as well as moment-curvature relationships then allowed the nondimensionalized governing equations to be developed.

Kim and Kim then considered steady-state peeling like Chen and Flavin [5], but accounted for reverse-bending as the adherend is straightened during debonding. Equations describing the maximum curvature and bending moment were then developed and used to find the work dissipated through plastic bending. Using the energy balance equation, the normalized peel force ($\bar{p}$) can be related to the normalized adherend thickness ($\bar{t}$) through the angle at maximum curvature ($\theta_B$). If $\bar{p}$ and $\bar{t}$ are plotted on a log-log plot, one obtains the universal peel diagram (UPD). Here, $\eta$ is the nondimensionalized peel force, $\Upsilon$ is decohesion or fracture energy of the system, and $p$ is the measure peel strength. This diagram allows the interfacial “toughness” or fracture energy to be found from a known peel force and adherend properties. To find an unknown $\Upsilon$, one simply finds the intersection of the universal curve and $\eta$ for a particular specimen, then uses the measured $p$ to calculate $\Upsilon$. From Figure 1, one can see that each red line represents a distinct fracture energy value. Potentially, there are up to three different values of load ($\eta$) that would give a single fracture energy value ($\Upsilon$).

![Figure 1: Universal Peel Diagram from [3] showing multiple possible values of load for a given fracture energy value](image-url)
The peeling of flexible laminates was also studied by Kinloch et al. [6]. In addition to determining the dissipated energy from bending of the adherends, their analysis models the peel test as an elastic beam on elastic foundation. This corrects the assumption that the peel arm is fixed in place at the crack tip by accounting for root rotation. After correcting for plastic bending, a material property (fracture energy), was found that is independent of test geometry (assuming cohesive failure) for a given adhesive thickness, test rate, and test temperature.

The effect of root rotation on peel models and specifically on fracture energy values has been studied by others [7, 8]. Moidu et al. [7] considered the plastic deformation of the adherend at the root by modeling the deflection of an elastic-plastic beam on an elastic foundation in their analysis. They also investigated adherend properties, peel angle, and the effects of mixed mode fracture on peel tests. This work was improved in [9] by taking into account the shear stresses in the adhesive and by modeling the adherend using a bilinear stress-strain response.

The work from Kinloch et al. [6] later developed into the ESIS Protocol for ICPeel [10, 11], which describes the theory behind the ICPeel spreadsheet from Imperial College. This protocol discusses both the fixed arm peel and the T-peel tests, as the geometries are similar. As it is in spreadsheet form, this analysis package can quickly and easily be used to determine the fracture energy value of a particular sample. In the analysis, the total system energy is broken down into the elastic and plastic deformations of the adherends, as well as energy dissipated through breaking the adhesive. As in [3], the stored elastic strain energy (in extension) is neglected in the resulting system energy balance. The energy input to the system can be found by taking the easily measured peel strength, and correcting for the angle of the specimen’s remaining bonded portion or tail.

Using material properties from both the adherend and adhesive, the ICPeel spreadsheet calculates the fracture energy value of the adhesive ($G_c$), as well as the plastic work in bending ($G_d$), input energy after correction for stored strain energy ($G_{tot}$), and the total input energy ($G$). In addition to the elastic-perfectly plastic model used in [3], ICPeel allows power law or bilinear models to be used to describe the stress-strain behavior of the adherends. A second version of ICPeel, allows for the use of digitized stress-strain curves instead of the adherend models mentioned above. For T-peel, the only required modification to the analysis is performing the calculations twice, once for each adherend and adding the results together (or multiplying by two if 90°).

Mechanical tests such as the T-peel test have historically been conducted in the laboratory to characterize a particular adhesive and adherend combination. The collected data is then post processed to obtain strength or fracture data that is typically used in the design of bonded structures. In the case of T-peel tests, this post processing typically involves partitioning the total system energy into the $G_c$ and $G_d$ portions. Analytical partitioning methods such as those developed by Kim and Kim [3] or Kinloch et al. [6], have been discussed above. Analysis packages such as ICPeel [10] have also been developed that iteratively solve the analytical equations to partition the T-peel system energy. For the purposes of this work, ICPeel will be considered an analytical method.
While analytical partitioning methods have been used successfully, there are times where a numerical model of the system is advantageous. The finite element method offers some unique advantages over analytical methods when evaluating fracture tests such as the T-peel test. Once a model has been developed, simulations can often be run faster and more economically than mechanical tests in the laboratory. This allows many different loading or environmental conditions to be investigated without running laboratory tests. In some cases, the actual bond geometry or test conditions cannot be replicated in the laboratory. In other cases, only few validation experiments can be feasibly conducted. In addition to the cost or time savings associated with FEA simulations, the model of an adhesive joint can often be integrated into larger models of entire structures. This is highly beneficial to automotive or aerospace industries where component and sub-assembly simulations can be run during the design process.

Considerable work has been done in developing models to describe crack growth, as this is a critical part of accurately predicting the behavior of T-peel specimens. Dugdale [12] and Barenblatt [13] both contributed to early cohesive zone models (CZM). Improvements include the cohesive surface formulation by Needleman [14], where an initial crack is no longer required, and the cohesive zone size is not a variable in the model. Tvergaard and Hutchinson [15] investigated plasticity of multi-phase materials as it relates to the toughness of materials with an interface and developed an embedded-process-zone (EPZ) model.

The EPZ model was used by Yang et al. [16] to examine the failure of adhesive joints where the adherends have undergone significant plastic deformation. A series of wedge DCB specimens with thin (1-3 mm) adherends was experimentally tested and modeled with Abaqus. As a result an “interfacial traction-separation law” was found by comparing the experimental and numerical results. Additionally, Yang et al. found that the EPZ model could be used without modification for samples with varying adherend thicknesses and geometry. As with the wedge DCB tests, numerical and experimental results were compared for the T-peel geometry. Good agreement was found between the experimentally derived peel force-displacement curve and the Abaqus results. Using the method put forth by Thouless et al [17], the interfacial toughness (also called fracture energy, $G_c$) was found in both cases.

The cohesive zone model has also been used by Alfano et al. in [18] to examine T-peel joints. They examined parameters such as the cohesive strength, fracture energy, shape parameter, and slope indicator. Additionally, they considered how these parameters can influence the measured or predicted load. In doing so, the predicted peel force-displacement curves were found to depend on the fracture energy of the epoxy material.

In [19], Hadavinia et al. investigated the fracture energy of 90° and T-peel tests, comparing the experimental results to numerical simulations and the linear-elastic fracture-mechanics (LEFM) test method. Unlike the popular cohesive zone model, where $G_c$ and an unknown traction stress must be defined, Hadavinia et al. used a critical plastic strain fracture model (CPSFM). In doing so, the traction stress, which is not a material property and is geometry dependent, can be eliminated. By using a node-release technique to model crack propagation, the authors found steady-state peel loads which were found to agree with those
measured experimentally. Additionally, Hadavinia et al. found that the fracture energy values were truly independent of the test specimen geometry.

The analytical and finite element approaches previously discussed, assumed that steady-state debonding has occurred. In this case, the measured load reaches a plateau, and varies little as the crack continues to propagate. The selected sample geometry and bond integrity can both affect the degree of adherend plasticity and self-similar debonding present in a particular T-peel specimen. Dillard et al. [20] detail four categories that can be used to describe the T-peel configurations that may be encountered during testing. These categories include plastically deforming adherends with self-similar debonding (II) and non-self-similar debonding (III). Additionally, elastically deforming adherends can also exhibit self-similar (I) and non-self-similar (IV) behavior. Here, self-similar debonding refers to the crack propagating at the same speed as the test frame crosshead, thereby maintaining a constant distance between the crack front and the applied load.

There are, however, cases when a particular specimen does not fall into to the desired category, or initially belongs to one category but shifts to another if the bond integrity degrades. In this study on a urethane-based adhesive, single specimens has been observed to exhibit behavior from multiple categories when testing under certain parameters. As such, care is required when designing a T-peel specimen so that the analysis methods are valid.

One key advantage of finite element models over analytical approaches is that steady-state debonding need not be assumed and analysis is not restricted to Category II tests where plastic deformation of the adherends and self-similar debonding occur. Li et al. [21] conducted an experimental and numerical study on four different T-peel adherends. A cohesive zone model (CZM) was used in Abaqus to determine the transition from non-steady-state to steady-state debonding. This was done in order to determine if common T-peel configurations would reach steady-state debonding as indicated by a plateau in the load curves. The experimental and numerical results were found to have good agreement for the category II, III or IV specimens encountered in the study. Additionally, the partitioned fracture energy, $G_c$, confirmed that the commonly used “T-peel strength” has only limited correlation to the actual energy required for peeling.

T-peel tests are among the most common for testing of adhesive joints. The often significant plastic deformation of the adherends however, complicates the analysis. For example, Li et al. [21] found that as much as 88% of the total system energy was used to plastically deform the adherends. As the plastic contributions cannot be ignored, considerable work has been done in the literature to produce analytical and numerical methods for accurately partitioning the total system energy. The universal peel diagrams and ICPeel spreadsheet have been developed in addition to numerous finite element models in an effort to effectively partition the total system energy. In many cases, the results obtained through these analysis techniques are quite good, but it is up to the researcher to apply the proper analysis technique to the system they are studying. In this study, ICPeel was used for processing the experimentally collected data, as it provided a quick and accurate method for determining fracture energy values.
Chapter 2 – Test Methods

Sample Geometry and Preparation

In this study, T-peel tests were chosen because they are easy to conduct as a function of rate and temperature, and avoid the need to identify or even see the debond tip. This feature is especially attractive when tests are conducted in environmental chambers and is in contrast to double cantilever beam (DCB) tests where the crack length, along with the measured load is most often recorded as a function of time. T-peel specimens use minimal aluminum and are lightweight and compact, making them easy to condition prior to testing. The key disadvantage of this specimen geometry is the complication of extracting meaningful fracture energies, as extensive adherend plasticity confounds data reduction. The finite element method or analytical means can be used to partition the total input energy into the amount of energy per unit area required for debond - the fracture energy. ICPeel[10, 11], developed by Imperial College, is an Excel spreadsheet that provides a method to partition the total system energy into the fracture and plastic bending energies. This program has been used to analyze the data presented in the following sections.

Sample Manufacture

T-peel specimens are constructed by bonding two panels or adherends together and leaving a small region unbonded at one end. This region forms two tabs when the adherends are bent back at 90°, resulting in the “T” shape shown in Figure 3. All T-peel specimens were manufactured by this project’s industrial sponsor. In total, four sets of samples were manufactured. As each set of samples was prepared at a different time, they were each assigned a batch identifier so that any differences could be tracked. A select number of Trial Batch specimens were manufactured in August of 2018. Batch 1 and Batch 2 specimens were prepared in October 2018, with Batch 3 specimens arriving in May 2019. In manufacturing T-peel specimens, the 203 mm by 228 mm (8 in by 9 in) aluminum panels were first cleaned and a proprietary surface treatment applied. Both a tape and plastic film were used to mask off the tab region on each panel from the trial, Batch 1, and Batch 2 specimens. A schematic showing the sample construction can be found in Figure 2. In Batch 3 specimens, the tape masks did not overlap as in prior batches, and no plastic film mask was used. This resulted in an adhesive layer coating one of the sample tabs.
The two part urethane based resin was mixed using a 356 mm (14 in) static mixing nozzle (254 mm mixing length) and applied to both panels. A notched blade was then used to coat both panels before a scrim cloth was added, and the panels sandwiched together. The scrim, also depicted in Figure 2, was used primarily to control adhesive flow prior to cure, but also served to control adhesive thickness. A heated hydraulic press was used to uniformly compress the panels together at 2,000 lb for 2-minutes at 250°F (121°C). This procedure was used to produce a relatively uniform bond thickness during cure.

**T-peel Geometry**

ASTM D1876-08 [2] defines the standard T-peel test method and geometry. While different adherend thicknesses may be used, the adherends need only be thin enough to allow for the 90° bend. The T-peel test geometry however, facilitates the use of relatively thin adherends, allowing the test geometry to closely match that of the intended application.
In this study, 0.635 mm (0.025 in) thick 2024-T3 aluminum adherends were used. The sponsor used a water jet cutter to section the bonded panels into the 25.4 mm (1 in) wide T-peel coupons. The bonded portion of the sample was shortened from the ASTM’s standard 229 mm (9 in) to 152 mm (6 in) in order to allow the samples to fit in the environmental chamber. The 76 mm (3 in) long tabs, and 25 mm (1 in) width was maintained from the ASTM D1876 standard. Once prepared, these coupons were then shipped to Virginia Tech to be environmentally conditioned and tested.

Sample Adherend and Adhesive Thickness

Adherend and adhesive thickness are two of several parameters that are used in the determination of fracture energy values. It is critical to obtain adherend and adhesive thickness measurements that are representative of the entire specimen. As the adhesive thickness cannot be measured directly, the adherends are measured, and their thicknesses subtracted from the total specimen thickness. This yields the inferred adhesive thickness that was used in the subsequent ICPeel analysis. Additionally, as the two adherends are bonded together, only the specimen tabs can be measured. Out of almost 200 samples tested, the adhesive thickness was found to vary between 0.13 and 0.54 mm depending on the batch. The adherend thickness was also found to vary between 0.63 and 0.72 mm. In an effort to minimize measurement error, the procedure for determining average adhesive thickness values changed several times.

A standard micrometer with 0.01 mm resolution and was used for the majority of the Trial Batch sample measurements. Total thickness measurement were taken in the center of the specimen, with the adherend measurements taken in the center of each tab. While several measurements were taken for verification, only a single adherend and total specimen thickness was recorded. The adherend thickness values can be seen in Figure 4. The majority of these trial
batch specimens had adherends slightly thicker than the specified 0.635 mm, with values spanning the range of those observed across all batches.

Figure 4: Adherend measurements for all batches

The “representative” adhesive thicknesses found in the trial batch specimens can be seen below in Figure 5. These specimens were found to be the thickest of all samples, ranging from approximately 0.43 to 0.54 mm.
The measurement technique described above for the trial batch was continued for early Batch 1 and Batch 2 specimens. Given the sample manufacturing process described above, however, it was thought that the bondline thicknesses between samples should be more consistent than was indicated by the trial batch and early Batch 1 and Batch 2 measurements. A drop micrometer was therefore employed with resolution 0.0001 mm. Five measurements were taken along the length of the specimen, but not in any particular order. These measurements were then averaged and the adherend thicknesses subtracted to obtain a single adhesive thickness value for use in ICPeel.

Despite this change in measurement technique, variation in the adhesive thickness persisted. The average adherend thickness however decreased slightly in most cases, and became much more consistent. This is evident from the drop in adherend thickness around test 25 in Figure 4. A select number of samples measured with the drop micrometer and already tested, were again measured with the standard micrometer to verify the prior measurements. Unfortunately, as these samples were already tested, several measurements along the sample length were not able to be taken as was done previously. In the cases where the micrometer was unable to be used, a digital caliper was used instead. As seen in Figure 6, the spread in measured values are much the same between the standard and drop micrometers. This added confidence that the T-peel samples were being measured accurately, and the bondline thickness varied significantly between samples. Average adhesive thicknesses obtained with the standard micrometer were found to be slightly thicker than those with the drop micrometer.
Suspecting that perhaps the specimens were not lying flat on the drop micrometer base, the standard micrometer was used again for the next nine specimens. Five measurements were taken along the length of the specimen from the bonded to non-bonded ends. Three measurements were also taken in the center of each tab. In each of these cases, the adhesive thickness was found to vary slightly along the length of the sample. The average adhesive thickness value used in ICPeel, however, continued to vary between samples and was not found to be significantly different than prior Batch 1 or Batch 2 samples measured with the drop micrometer.

As the drop micrometer had more resolution than the standard one, and measurements can quickly be exported to a spreadsheet via a cable, this was the preferred measurement instrument. To alleviate concerns about curved samples not lying flat on the micrometer base, a cylinder was attached to the base as shown in Figure 7. This allowed the sample to effectively contact the base at only one point along the length, minimizing possible errors associated with a slightly bowed sample. A second cylinder was also added to keep the specimen level during the measurement process. The addition of these two cylinders likely contributed to the more consistent adherend thicknesses seen in Figure 4 after approximately test 25, but did not appear to have much of an effect on the variation in adhesive thickness shown in Figure 5.
For the remaining Batch 1 and Batch 2 tests, 10-12 total sample measurements were taken along the specimen length. These measurements were averaged to obtain a “representative” total specimen thickness. Three adherend measurements were also taken in the center of each tab, and averaged for both adherends. The two adherend thicknesses were then subtracted from the total specimen thickness, resulting in an adhesive thickness value. While the number of measurements doubled, they were taken at random locations (from the tab to tail ends) and only the average value was recorded. These adhesive thickness values were also found to vary slightly along the length of the sample.

From Figure 5, Batch 1 and Batch 2 samples exhibited much the same variation as the Trial Batch, with none of the different measurement techniques appearing to make a significant difference in the average adhesive thickness. Within the Batch 2 samples, the adhesive thickness was found to vary significantly, ranging from approximately 0.2565-0.4327 mm. It is also apparent from Figure 5, that while manufactured at different times, these samples appear to belong to the same thickness population, as they are similar in both thickness and scatter. While exhibiting similar variation, Batch 1 and Batch 2 adhesive thickness measurements were considerably lower than those from the Trial Batch. This seems to indicate that while there is significant specimen to specimen variation, there is also a difference in adhesive thickness between batches. Keeping this in mind, consistency in sample manufacture was a key focus for the project’s sponsor as they manufactured the remaining T-peel coupons.

Early Batch 3 samples were measured with the drop micrometer and cylinder base, with 10-12 sample measurements taken along the length of the sample. The three measurements in the center of each adherend tab was maintained from the prior measurement techniques. These samples however, continued to exhibit considerable scatter in the average adhesive thickness.
measurements as is evident in Figure 5. Significant variations in the measured load were also observed in these specimens. Based on the variations in adhesive thickness measured along the length of the specimen, it was thought that perhaps this variation could be contributing to the large load variations. To investigate these load variations further, a map of the adhesive thickness along the length of the sample was needed.

Shown in Figure 8, are the eight measurement locations used to measure the majority of Batch 3 specimens. The four measurement locations on each edge, were spaced 24 mm apart to roughly correspond to the regions of the sample tested at each of the three rates. This allowed a thickness measurement to be recorded at the beginning and end of each test rate. Additionally, the first set of measurement points were spaced 8 mm from the end of the mask region. In general, around 8 mm of debond is required for the sample to reach an equilibrium load. The measurement scheme presented in Figure 8 is important, because thickness values are only obtained for the tested portion of the sample, unlike samples from Batch 1 and Batch 2.

![Figure 8: Batch 3 sample measurement locations](image)

Unlike previous batches, Batch 3 specimens did not have a full-length tape mask or a plastic film separating the two tabs of a T-peel specimen. As shown in Figure 2, this results in the two tabs bonded together and requiring them to be separated via a hammer and sharp wedge (razor blade). Four adherend measurements were taken on the adherend that could be measured, while the other adherend (the one with an adhesive film) was assumed to have the same thickness. As Batch 1 and Batch 2 samples showed little variation in the adherend thickness, once a sample had been accurately measured from a given panel, that adherend thickness was used for all other samples from that panel. This was done to simplify the sample preparation and measurement process. Measuring only one adherend on each panel did not appear to significantly impact the recorded value, as Batch 3 measurements exhibited similar variation to those seen in the later Batch 1 and 2 samples (Figure 4).

Having obtained the total thickness measurements at eight locations, these values were averaged, and the average adherend thickness subtracted. Overall, Batch 3 samples had adhesive thickness values that were half or less than those seen in prior batches. As will be discussed in a later section, the dependence of fracture energy on adhesive thickness seen in literature was also observed for this system. Despite this change in adhesive thickness between batches, all specimens were within the manufacturing tolerance indicated by the sponsor.

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1 Some specimens with complete debond were tested outside of the normal 88 mm test section, as the crack propagated faster than the crosshead.
The change in measurement procedure for Batch 3 specimens did not have an effect on the average values reported in Figure 5, but allowed a spatial variation in adhesive thickness to be found. As the adhesive thickness was known for fixed specimen points, adhesive thickness could be correlated with the corresponding measured load along the length of the sample. To do this, load values were assumed to start at the end of the 76.2 mm (3 in) mask region shown in Figure 8. Because the Instron crosshead moves 2 mm for every mm of debond, the sample measurement locations are simply multiplied by two to obtain the equivalent Instron crosshead displacement. Shown in Figure 9, are the load and adhesive thickness variations plotted along the length of two samples. In both cases, the load varies with a change in adhesive thickness. As will be discussed in a later section, a “representative” average load and average adhesive thickness likely do not fully describe this system.

Figure 9: Spatial variation of load and adhesive thickness for specimens 22G (top) and 35D (bottom)
Referring back to Figure 4 and Figure 5, both adherend and adhesive thickness measurements can be found for samples from Batch 1 and Batch 2 using an alternate measurement method. These samples were untested Batch 1 and Batch 2 samples that were measured with the drop micrometer according to the fixed measurement locations presented in Figure 8. While time did not allow for testing of these samples, their adherend and adhesive thicknesses could be compared to samples previously tested, but not measured according to Figure 8. From Figure 4, it appears that while slightly thicker than many of the other Batch 1 and Batch 2 samples, there is little if any difference between the batches in terms of adherend thickness. Looking at the adhesive thickness in Figure 5, Batch 1 and Batch 2 samples measured using the Batch 3 method, were found to be on average, thinner than those previously measured. These samples are on the higher side of Batch 3 samples, and exhibit similar scatter as those tested from Batch 1, Batch 2, and Batch 3.

Adherend and Adhesive Material Properties

In addition to the adhesive and adherend thickness discussed above, ICPeel requires the adhesive and adherend material properties. The adherend modulus and yield stress, can both be found by performing a simple tension test. Three uniaxial tension tests were performed at room temperature on the 0.635 mm (0.025 in) thick aluminum 2024-T3 adherend stock, according to ASTM E8 [22]. One of these tests is shown below in Figure 10. The modulus values (E) were found to be 73.0, 71.2, 72.3 GPa, while the yield stress (σy) was found to be 335.8, 335.8, and 330.5 MPa using the 0.2% offset method. Representative values of E = 72.4 GPa and σy = 331 MPa were chosen and used for all data analysis. From [23], it appears that the material properties do not change significantly with a modest increase in temperature. In fact, at 200°F E = 69 GPa, while σy = 346 MPa. This is only a 4.53% change from the values measured at room temperature. As all T-peel tests were conducted at 180°F or less, the material properties were not changed from the representative values obtained at room temperature (E = 72.4 GPa and σy = 331 MPa).

Figure 10: Al 2024-T3 stress-strain curve
Tensile tests were also performed on the adhesive material by the project’s sponsor at room temperature. A representative adhesive secant modulus was obtained from the values shared by the project’s sponsor. This modulus was found to be \( E_a = 0.027 \text{ GPa} \), and was used for all subsequent analysis. Additionally, DMA data was collected on the base material by Eric Gilmer from the Chemical Engineering Department. The glass transition temperatures (\( T_g \)) were estimated to be in the vicinity of 86°F (30°C) and -130°F (-90°C) [24].

**Aging Conditions**

**Batch 1 and Batch 2 Cyclically Aged Specimens**

The Trial Batch specimens as well as most of the Batch 1 and Batch 2 specimens were tested unaged. These results, in addition to some data collected by the project’s sponsor, indicated that exposure to high temperatures and/or moisture could prove detrimental to the integrity of the bond. To study the effect of both elevated temperatures and moisture on bonded coupons, a total of thirty specimens, fifteen each from Batch 1 and Batch 2, were selected and placed in an ESPEC ESX-4CA environmental chamber. A cyclic aging profile from -65°F (-53.9°C) to Temperature A was chosen under three different humidity conditions. As shown in Figure 11, three plastic tubs were used to individually expose the samples to dry (sealed with Drierite), high humidity (suspended above water), and chamber humidity (atmospheric) conditions. The high humidity (HH) and dry samples, represented the two possible environmental extremes. The chamber humidity (CH) condition would fall somewhere in the middle of these two extremes, and would most closely follow the conditions previously tested by the project’s sponsor. By testing these three groups of samples, a baseline was set (dry samples), the sponsor’s prior results were approximated (chamber humidity), and a possible “worst case scenario” was probed (high humidity). As the environmental chamber used only offered refrigerated cooling, each cycle took approximately four hours to complete. As such, only 402 cycles were completed in the 1608 hours the cyclic aging was conducted.

![Figure 11: Environmental chamber setup: no humidity (left), ~100% RH (middle), variable humidity, (right)](image-url)
Batch 3 Isothermally Aged Specimens

Evolving opinions suggested that elevated temperatures, in combination with moisture, would be the cause of any measured degradation, rather than thermal cycling. The limited number of cycles and short exposure times at Temperature A achieved in the Batch 1 and 2 cyclic aging, prompted most of Batch 3 samples to be isothermally aged. These new samples contained nine unaged specimens to form a baseline, as well as 162 samples to be aged at 20 different conditions. Shown in Table 1 is the aging matrix used for Batch 3 specimens. As a combination of time at temperature was the predicted degradation mechanism, samples exposed to higher temperature were aged for shorter times.

<table>
<thead>
<tr>
<th>Aging Condition</th>
<th>Aging Times (hr)</th>
<th>Drying Times (hr) @ 150°F</th>
<th>Storage after drying</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pristine</td>
<td>0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>180°F (82.2°C) dry</td>
<td>500</td>
<td>1000</td>
<td>2000</td>
</tr>
<tr>
<td>200°F (93.3°C) dry</td>
<td>50</td>
<td>100</td>
<td>200</td>
</tr>
<tr>
<td>Temperature B dry</td>
<td>10</td>
<td>20</td>
<td>40</td>
</tr>
<tr>
<td>180°F (82.2°C) wet</td>
<td>500</td>
<td>1000</td>
<td>2000</td>
</tr>
<tr>
<td>200°F (93.3°C) wet</td>
<td>50</td>
<td>100</td>
<td>200 500</td>
</tr>
<tr>
<td>Temperature B wet</td>
<td>10</td>
<td>20</td>
<td>40 500</td>
</tr>
</tbody>
</table>

Due to some very long aging times (1000+ Hr.), four of the aging conditions (highlighted blue in Table 1) were performed at the sponsor’s facility. This allowed testing to be conducted at a faster pace, by aging some samples simultaneously. This was critical as the project timeline required all specimens to be tested in a little under eight months. Samples aged at the sponsor’s facility were aged at 95% RH, rather than the ~100% RH used at VT.

Those samples highlighted in green were additional aging conditions not initially planned, but conducted as part of a pilot study designed to examine longer aging times at elevated temperatures. The additional aging conditions required the use of samples previously aged in the dry condition: 200°F 10 Hr. and Temperature B 50 Hr. These samples were further aged wet for 500 Hr. at their initial aging temperatures (200°F and Temperature B). Based on the results of prior tests, the possible effects of the previous dry aging were expected to be very minor.

Because of the unknown moisture content in the previously cyclically aged T-peel specimens, this parameter was tightly controlled during the isothermal aging. Three aging condition replicates were prepared, however, only the first two replicates were to be tested due to time constraints. The majority of replicate three samples would serve as a contingency, with a few samples used in exploratory tests.

Once Batch 3 samples were manufactured by the project’s sponsor, they were shipped to Virginia Tech. The samples were then labeled, randomized, and their mass recorded. Samples assigned to the very long aging times were shipped back to the sponsor for environmental aging at their facility. After aging, these samples were again shipped to VT in sealed aluminum bags to
prevent moisture loss. Sample mass was recorded after aging, and again after drying, storage, and testing. This allowed the relative moisture wt. % to be tracked through environmental conditioning, storage, and testing.

Drying Conditions and Storage

**Trial Batch, Batch 1, and Batch 2 Samples**

Trial Batch specimens, in addition to unaged specimens from Batch 1 and Batch 2, were tested as shipped from the project sponsor. These specimens were stored in the lab at room temperature and under normal humidity conditions until tested. As indicated in the previous section, thirty specimens from Batches 1 and Batch 2 were cyclically aged under three different humidity conditions. Although no formal drying process occurred, these samples were stored in a desiccator after aging. As replicate three was tested last, these specimens would have been in the desiccator the longest, and could have a lower moisture content than the other samples.

**Batch 3 Samples**

Starting with Batch 3, the project sponsor requested that all T-peel samples be tested dry, in order to determine the permanent damage associated with time at temperature and with exposure to humidity. This was done primarily to eliminate concerns raised about the moisture content in the cyclically aged samples. While drying Batch 3 samples led to a relatively constant moisture content within each sample, doing so would not necessarily indicate the “worst case scenario”. If the bonded joint was exposed to higher temperatures, more moisture or either condition for longer periods of time, the energy required for debond could be reduced. In this case, any long term durability predictions may not accurately represent service conditions. To explore this possibility, most of the replicate three aged wet specimens were not dried. These specimens are marked in red in Table 1, and were sealed in plastic Ziploc bags to limit moisture loss during storage.

Pristine samples (i.e. those not aged) were dried for 48 hours at 150°F (66°C), while the wet samples were dried for 1000 hours also at 150°F (66°C). This drying was performed in a Thermo Scientific Lindberg Blue M digitally controlled oven. Samples aged in the dry condition were not dried further. After aging (if dry) or drying (if wet or pristine), all samples were stored in a desiccator until testing. Indicating Drierite desiccant was used and changed when required to maintain a dry atmosphere in the desiccator. The drying temperature and times were specified by the sponsor based on their previous experiences with the adhesive. It is possible that the wet samples were not completely dry after 1000 hours at 150°F (66°C); however, these specimens would have had the same relative weight percent of moisture. It was assumed that samples aged dry would be completely dry upon completion of the specified aging time.

If the samples aged wet were not fully dried after 1000 hours, they would likely have continued to dry out during storage in the desiccator. The time each sample spent in the desiccator varied, depending on the aging condition and testing order. Additionally, as a typical test lasted anywhere from 24 to 72 hours, it is also possible that samples continued to dry out during testing. This was particularly relevant at higher temperatures, but could not be prevented as the Instron test frame at was not equipped for testing under controlled humidity conditions.
Testing Conditions

Trial Specimens

The ultimate goal of this study was to collect data and develop methodologies for long-term durability predictions. In order to accurately predict the adhesive behavior at these long times, accelerated testing was employed in which the testing temperature was increased to effectively shift the fracture data to longer times. However, even with increased temperature, the expected service times of this adhesive were not achieved, as reaching these times would have required testing temperatures that would likely have caused degradation of the adhesive. Consequently, material behavior predictions were required in order to estimate the long-term durability of the adhesive in question. The ASTM D1876 standard [2] recommends T-peel tests be conducted at a constant crosshead rate of 254 mm/min (10 in/min) for at least 127 mm (5 in) of debond. This intermediate rate recommended by ASTM is not however, the most useful in terms of long-term durability predictions. Therefore, as shown in Table 2, two additional rates of 2.54 mm/min (0.1 in/min) and 0.0254 mm/min (0.001 in/min) were selected to be tested. This combination of testing rates allowed for durability predictions with Time Temperature Superposition (TTS). The slowest test rates and highest temperatures, when viewed in terms of TTS, provides the data closest to the expected service condition.

One of the goals for the Trial Batch specimens was to determine the feasibility of testing multiple rates on a single specimen. This would reduce the number of specimens required to be manufactured by the sponsor, but would also reduce the amount of data that could be collected at each rate. While the ASTM standard recommends 127 mm (5 in) of debond, little additional information would be gained if the load reached equilibrium well before this point. In fact, coach peel samples, which have significantly shorter bond lengths (75 mm), have been successfully tested [25, 26]. As these initial specimens were part of the pilot study, the elevated test temperatures and slower rates, were chosen in order to ascertain the feasibility of predicting the long-term behavior of this adhesive. Test temperatures of 75, 120, and 180°F (23.9, 48.9, 82.2°C) were selected, to span those the likely to be seen by the adhesive joint. Each specimen then, was tested at one of the three temperatures and all three test rates as indicated in Table 2. All tests were conducted on an Instron 5800R load frame. Additionally, a Thermotron F-2-CHM-LN2 chamber was utilized for both the high and low temperature tests.

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Test Rates</th>
</tr>
</thead>
<tbody>
<tr>
<td>°F</td>
<td>°C</td>
</tr>
<tr>
<td>75</td>
<td>23.9</td>
</tr>
<tr>
<td>120</td>
<td>48.9</td>
</tr>
<tr>
<td>180</td>
<td>82.2</td>
</tr>
</tbody>
</table>
**Batch 1 and Batch 2 Unaged Specimens**

New test rates were selected for unaged specimens from Batch 1 and Batch 2 to better capture both the short, and long-time behavior of the adhesive. The fastest rate was chosen to be 500 mm/min (19.7 in/min), as that is the upper limit of the Instron load frame. Additional rates of 50, 5, 0.5, and 0.05 mm/min (1.97, 0.197, 0.0197, 0.00197 in/min) were added as indicated in Table 3. As only three rates could be tested on a single specimen while still reaching an equilibrium load in most cases, two samples were needed to test all five rates at each temperature condition. While the samples were randomized prior to testing, each pair of samples corresponding to a single test temperature were from the same batch. Additionally, each temperature test condition also included at least one replicate from each batch in order to probe any batch-to-batch variability.

In addition to the 75, 120, and 180°F (23.9, 48.9, 82.2°C) tests conducted on the preliminary specimens, temperatures of 15, -20, and -65°F (-9.44, -28.9, -54.9°C) were added to the test matrix. While not the most important test temperatures in terms of long-term durability predictions, the adhesively bonded part may experience these colder temperature conditions. Due diligence therefore, required that these conditions be tested. Tests at these colder temperatures also assisted in the creation and fitting of master curve plots, which will be describe in detail in a later section.

For a given temperature condition, the first sample was tested at 500, 50, and 0.5 mm/min (19.7, 1.97, 0.0197 in/min). The second sample was tested at 5 and 0.05 mm/min (0.197, 0.00197 in/min). By testing the two slowest rates on different samples, the total time at temperature was not significantly different between the two specimens. This was done to prevent unintentional aging from occurring in one sample, but not the other. Additionally, by testing the slowest rate on each sample last, the tests could be run overnight.

**Table 3: Batch 1 and Batch 2 unaged test matrix**

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Test Rates</th>
</tr>
</thead>
<tbody>
<tr>
<td>°F</td>
<td>°C</td>
</tr>
<tr>
<td>-65</td>
<td>-54.9</td>
</tr>
<tr>
<td>-20</td>
<td>-28.9</td>
</tr>
<tr>
<td>15</td>
<td>-9.44</td>
</tr>
<tr>
<td>75</td>
<td>23.9</td>
</tr>
<tr>
<td>120</td>
<td>48.9</td>
</tr>
<tr>
<td>180</td>
<td>82.2</td>
</tr>
</tbody>
</table>

As can be seen in Table 3, the low temperature tests were only conducted at 500, 50, and 5 mm/min (19.7, 1.97, 0.0197 in/min). As low temperatures can be thought of as equivalent to faster rates, there was little to be gained by testing samples at both low temperature and slow rates. Additionally, the long times associated with these tests (~33 hours) would make them cost prohibitive, as the environmental chamber used in the Instron was cooled by liquid nitrogen.
**Batch 1 and Batch 2 Cyclically Aged Specimens**

Due to the limited number of cyclically aged samples from Batch 1 and Batch 2, these samples were tested at 500, 50, and 5 mm/min (19.7, 1.97, 0.197 in/min) as shown in Table 4. This allowed rates spanning three decades to be tested on a single specimen. Additionally, only tests at 75, 120, and 180°F (23.9, 48.9, 82.2°C) were conducted, again due to the limited number of coupons available. As with the unaged samples, three replicates were tested, and each temperature condition contained specimens from both batches.

*Table 4: Batch 1 and Batch 2 cyclically aged test matrix*

<table>
<thead>
<tr>
<th>Test Temperature °F</th>
<th>°C</th>
<th>Aging Condition</th>
<th>Test Rates mm/min</th>
<th>mm/min</th>
<th>mm/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>75</td>
<td>23.9</td>
<td>High Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>120</td>
<td>48.9</td>
<td>High Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>180</td>
<td>82.2</td>
<td>High Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>75</td>
<td>23.9</td>
<td>Chamber Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>120</td>
<td>48.9</td>
<td>Chamber Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>180</td>
<td>82.2</td>
<td>Chamber Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>75</td>
<td>23.9</td>
<td>No Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>120</td>
<td>48.9</td>
<td>No Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>180</td>
<td>82.2</td>
<td>No Humidity</td>
<td>500</td>
<td>50</td>
<td>0.5</td>
</tr>
</tbody>
</table>

**Batch 3 Specimens**

The majority of specimens from Batch 3 were aged in order to determine the long-term effects of aging. These specimens, then, were tested at 75, 120, and 180°F (23.9, 48.9, 82.2°C) as these conditions are of the most interest when predicting long-term behavior. Two replicates were planned, with a third allowing for contingencies and some exploratory tests. Replicate one tests were conducted at crosshead rates of 500, 5, and 0.05 mm/min (19.7, 0.197, 0.00197 in/min). This plan kept the fastest rate, while still testing an intermediate and very slow rate. The other intermediate rates from Batch 1 and Batch 2 were eliminated due to project time constraints.

As indicated in Figure 9, the measured load can vary quite significantly over the course of a test. As significant rate effects had not previously been observed, replicate two samples were tested at a constant rate. While limiting the data collected, testing at a single rate offered valuable insight into the relationship between load and adhesive thickness variations along the length of the sample. The majority of replicate two samples were tested at a constant 5 mm/min. A few of the specimens aged wet (1000 and 2000 hr.) were tested at the slower 0.05 mm/min (0.00197 in/min). This was done in order to further investigate the reduced sample curvatures observed during some of the replicate one tests. The few replicate three tests that were performed on aged materials, were all conducted at the slowest 0.05 mm/min (0.00197 in/min) rate. As the pristine samples were considered a Batch 3 baseline, all of these samples were tested at all three rates, regardless of the replicate they came from.
Chapter 3 - Data Analysis

Time Temperature Superposition

The mobility of polymers such as the urethane-based adhesive examined in this work, is directly related to temperature and the interactions between polymer chains. At low temperature, molecular motions are relatively slow, but increase with higher temperatures. Although chemical or physical crosslinks (entanglements) restrict mobility, the polymer chains can move past each other given sufficient temperature or time.

This time and temperature dependence, affects material parameters such as the modulus. At low temperatures, polymers have high modulus values and are in the “glassy state” due to the slow motion of the polymer chains. This same behavior is observed with short time scales, because the polymer chains do not have a chance to move significantly. As temperature or time increases, the polymer transitions to its “rubbery state” and the modulus decreases. This is due to increased molecular motions or more time available for the polymer chains to move past each other. The temperature at which this transition occurs is called the glass transition temperature ($T_g$), and corresponds to the onset of cooperative, long-range segmental backbone motion.

Often dynamic mechanical analysis (DMA) is used to probe the changes in the modulus and determine the storage and loss moduli. As the name implies, the storage modulus ($E'$) is a measure of the energy stored elastically during deformation, while the loss modulus ($E''$) measures the energy lost, typically in the form of heat. The ratio of these two parameters ($E''/E'$) is referred to as the tan δ, and is one metric that can be used to find the $T_g$ of a polymer.

While temperature can be experimentally varied relatively easily, the time dependence of polymers is more challenging. It is difficult, for example, to test materials at very short (< 0.01 s) or long (> 1 week) time scales. Because modulus is a function of both time and temperature, a polymer at elevated temperature would behave the same a polymer at an appropriately lower temperature, but tested at a slower rate (long times). This is a well-known concept in polymer physics is called the time-temperature superposition principle (TTSP). Using this principle, the thermomechanical behavior of a material may be examined at the time scales that are typically unable to be tested in the laboratory, due to equipment limitations or time constraints.

By conducting tests at different temperatures and rates, the molecular behavior can be understood over a wide range of conditions. Each dataset can then be plotted together, and shifted horizontally on the frequency or time axis until the data overlaps, forming a master curve referenced to an un-shifted dataset. Shown in Figure 12, is the storage modulus found by conducting DMA tests on a urethane-based adhesive\textsuperscript{2} [24]. Frequency sweeps ranging from 0 to 100 Hz, were performed at 22 different temperatures ranging from -60°C to 110°C. The data from Figure 12, was then horizontally shifted in reference to the data at 30°C to form the master curve shown in Figure 13.

\textsuperscript{2} DMA data collection and analysis was conducted by Eric Gilmer from the Chemical Engineering Department.
As can be seen, the modulus measurements that previously spanned three decades have now been expanded to encompass twenty decades; an impossible range to measure practically in the laboratory.

Figure 12: Un-shifted DMA data from a urethane-based adhesive

Figure 13: Shifted DMA data
The amount each dataset is shifted corresponds physically to the relaxation time of the polymer. Shown in Figure 14, are the shift factors used in constructing Figure 13. These “shift factors” allow data collected experimentally at one temperature and frequency, to be shifted to a different effective temperature and frequency. Thus data collected in hours or days, can be accurately shifted to approximate the behavior of a polymer in milliseconds or years. In many cases, the shift factors are described using Arrhenius or Williams-Landel-Ferry (WLF) like behavior, which relates directly to the amount of free volume in a polymer.

Shift factors are most commonly found using a DMA to measure the storage and loss moduli, while varying the test temperature and frequency. Using TTSP, these shift factors have been widely applied to constitutive properties such as modulus in order to estimate the behavior of polymers at very short or very long time scales. These same principles can also be applied to fracture data, as was done with the urethane-based adhesive tested in this study. T-peel tests were conducted over a wide range of temperatures and rates. As only one data point is collected at each temperature and rate, there is much less data available than would be typical for a DMA temperature/frequency sweep. These fracture energies can then be shifted to provide data at relevant service times. While this is routinely done, the confidence in applying TTSP to fracture data is often lower than with constitutive properties. This is due to inherent scatter in fracture data, as well as the limited number of data points. Therefore, care should be taken when using fracture data to ensure TTSP is applicable to the system.

Figure 14: Experimentally derived and WLF shift factors
ICPeel and System Energy Partitioning

ICPeel Theoretical Basis

ICPeel [10, 11] is an analysis program developed by the Imperial College London and was used in this study to extract approximate fracture energies from the measured T-peel load. Unlike (ideal) DCB tests, where the adherends only deform elastically, the adherends permanently deform in T-peel tests. This results in some of the total system energy going into breaking the adhesive, with the remainder being dissipated through plastic deformation of the adherends. Therefore, a method is needed to partition the energy so that the adhesive fracture energy ($G_c$), which is a fraction of the total input energy per unit debond area, can be determined.

There are two different free versions of ICPeel available for download as Excel spreadsheets from Imperial College’s website [10]. The first is the standard program, and uses an elastic-perfectly plastic, bilinear, or power law material model for the peel arm(s). The second version of ICPeel known as ICPeel (Digitized), uses a numerical analysis from the digitized stress-strain curve of the peel arm material. This stress-strain curve can be easily obtained by performing a uniaxial tension test on the adherend material. ICPeel (Digitized), requires that the standard version be run first, and the outputs then used to complete the analysis. In this study, the standard version of ICPeel using the elastic-perfectly plastic adherend model, was used for the data analysis presented in subsequent sections.

The ESIS Peel Protocol [11], provides some guidance in conducting peel tests, and discusses the calculations used in the ICPeel program to determine the adhesive fracture energy. The ICPeel program was originally developed for fixed arm peel tests, but is applicable to T-peel tests with some minor modifications, as discussed below. The total system energy resulting from the external work input, can be broken down into the elastic and plastic deformation energies, as well as the energy being applied directly to deform and break the adhesive. If debonding were to occur over a small distance, $da$, in a specimen of width $b$, an incremental energy balance can be written in the form of Equation (1).

$$G_A = \frac{dU_{\text{ext}}}{bda} - \frac{dU_s}{bda} - \frac{dU_{\text{dt}}}{bda} - \frac{dU_{\text{db}}}{bda} (1)$$

Equation (1) expresses the energy release rate being applied to the adhesive ($G_A$), in terms of the increments in external input work ($U_{\text{ext}}$), stored elastic strain energy ($U_s$), dissipated tensile energy ($U_{\text{dt}}$), and the dissipated bending energy ($U_{\text{db}}$). If the energy stored elastically is negligible, then: $G_A = G - G_p$. Here, $G$ and $G_p$ are the total input energy and energy plastically dissipated (both per unit debond area) in extension and bending. After neglecting tensile elastic/plastic deformations within the adherends, the input energy per unit area can be expressed as Equation (2). It is important to note here, that this analysis is only valid once steady-state debonding has been reached.

$$G = \frac{P}{b} (1 - \cos \theta) (2)$$
As shown in Figure 15, P is the load as measured during testing, and θ is the peel angle. Unlike a standard fixed arm peel test, which has one rigid and one flexible adherend, T-peel tests include two flexible adherends. Therefore, calculating the total adhesive fracture energy requires using Equation (2) twice; once for each adherend and adding the results together. In practice, the ICPeel spreadsheet will use two rows (for peel angles other than 90°), one for each adherend, to calculate a single test case and provide the adhesive fracture energy $G_c$. The results from these two calculations can then be added, to obtain the net adhesive fracture energy. In the case of a 90° T-peel test, the result can simply be multiplied by two, as the upper and lower adherends have the same angle.

In fixed arm peel tests (the basis for ICPeel), the adhesive thickness is measured in relation to a rigid substrate. Although the peel protocol [11] does not specify to do so, in T-peel tests, half of the measured adhesive thickness should be used for each peel arm, so the resulting total adhesive fracture energy is based on the appropriate thickness. This is consistent with fixed arm peel tests, and assumes that the crack is propagating in the middle of the adhesive layer; resulting in a plane of symmetry about the center of the bond thickness.

ICPeel therefore, estimates one fracture energy value for each measured load, sample geometry, and material property set entered into the spreadsheet. In most peel tests, the average load once steady-state debonding has been reached, and a representative adhesive thickness are entered into ICPeel. If this average peel load and adhesive thickness are representative of the specimen, as they are often assumed to be, then the estimated fracture energy value is thought to be representative of the sample tested. When an average peel load cannot easily be determined, or there are spatial variations in adhesive thickness or other parameters, this single fracture energy value may no longer be representative of the tested specimen. Experimental observations correlating the measured load and adhesive thickness, prompted further study on the impact of these two parameters on the partitioning of system energy in ICPeel.
**Energy Partitioning of T-peel Samples in ICPeel**

As discussed previously, ICPeel partitions the total system energy per unit area at steady-state, into adhesive fracture energy \( (G_c) \), and plastic work in bending of the adherends \( (G_d) \). Thus the work per unit area (practical adhesion) is the sum of \( G_c \) and \( G_d \) as shown in Equation (3).

\[
Practical\ Adhesion = \frac{2 \cdot \text{load}}{\text{bond line width}} = G_c + G_d = \left[ \frac{\text{Energy}}{\text{unit area}} \right]
\]

(3)

T-peel curvature, as shown in Figure 16, is physical basis for the partitioning in ICPeel. Large plasticity of the adherends is required to initiate failure of the adhesive and requires the total system energy to be partitioned. Some of the total system energy is dissipated by plastically deforming the adherends, and some of the energy is dissipated within the adhesive by the deformation and debonding phenomena. Thinner adhesive layers (left hand image) require a larger portion of this total energy to be used in plastic bending of the adherends in order to achieve the same percent strain as a sample with a thicker bond line (right hand image). Thus, to achieve say 500% strain in the adhesive layer, for example, the sample on the left would have sharper curvature than the sample on the right, indicating more plastic deformation. Samples dissipating very little to no energy in the adhesive could debond with little to no plastic deformation in the adherend. This behavior has been observed on numerous occasions, and will be discussed in Chapter 4 – Experimental Results.

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*Figure 16: Observed effects of adhesive thickness on T-Peel curvature during testing.*
In Figure 17, $G_d$ can be seen to increase for thinner adhesive thicknesses, and is consistent with the behavior shown in Figure 16. Here, $G_c$ was assumed to be constant, as a given adhesive material when unaged, is thought to dissipate a fixed amount of energy. To achieve the larger $G_d$ with thinner bondlines, and a constant value for $G_c$, the practical adhesion (load normalized by sample width) is required to go up. This practical adhesion, is essentially summing the area under the $G_c$ and $G_d$ curves.

![Figure 17: Energy partitioning in ICPeel. See Chapter 4 – Experimental Results for input parameters.](image)

From a broader perspective, however, $G_c$ is not typically independent of adhesive thickness. Conceptually, the more of a material there is, the more energy it should dissipate. If $G_c$ is now assumed to be proportional to adhesive thickness, twice the amount of adhesive should double the value of $G_c$. Such trends have been reported, as can be seen in the first part of Figure 18 from [27], before the peak at $t = t_m$. This peak occurs when the adhesive thickness is equal to the size of the plastic zone.

An approximately linear relationship between $G_c$ and adhesive thickness (Figure 19) can be seen in the literature for epoxy adhesives [27, 28]. Although different from the urethane-based adhesive used in this study, these examples demonstrate that a linear relationship, such as the one in Figure 18 between $G_c$ and adhesive thickness is not an unreasonable estimation for the experimental data collected in this study. This is postulated in an attempt to ascertain if adhesive thickness variations contributed to the observed variability in peel loads and the resulting fracture energies.
Simple estimates have indicated that for the urethane-based adhesive in this study, the radius of the plastic zone is between 0.2 and 2 mm for unaged samples. Given the observed scatter in the fracture data, and the relationship between the size of the plastic zone and fracture energies, it is not surprising that the radius of the plastic zone could vary significantly. Average Batch 3 adhesive thicknesses (~0.2 mm) are about the same as the smallest estimated plastic zone radius, but are much smaller than the upper radius. This seems to indicate that for most T-peel tests, the samples would fall in the initial linear region of Figure 18. In fact, these trends were found in the experimental data, and will be discussed in detail in Chapter 4 – Experimental Results.
Sensitivity Study on ICPeel Input Parameters

As indicated in a previous section, ICPeel requires a number of different inputs in order to partition the total system energy, and extract fracture energy values. In addition to the load; the peel angle, adhesive thickness, and adhesive modulus all play a critical role in partitioning the total system energy. Although not always easy to measure, the peel angle was recorded for each test. In Figure 20, the adhesive fracture energies for the peel and supplementary angles were added together to provide $G_c$ as a function of the measured peel angle.

Here, the peel angle ($\theta$) is taken as the angle greater than or equal to 90°, while the supplementary angle, $\phi = 180° - \theta$. Additionally, the load, material properties, and adhesive thickness were held fixed while the peel angle was varied for a range of experimentally observed values. These values including the scale factor used to normalize $G_c$, can be found in Chapter 4 – Experimental Results.

From Figure 20, it is clear that the peel angle has only a small effect on the fraction of total system energy that corresponds to the energy dissipated in the adhesive during debond. In fact, there is only around a 3% change in the partitioned fracture energy over the range of experimentally measured peel angles (90° to 125°). It should also be noted, that Figure 20 only represents the theoretical changes in the estimated fracture energy due to a change in the measured peel angle, not those measured experimentally.

![Figure 20: Dependence of adhesive fracture energy on measured peel angle and its supplement. See Chapter 4 – Experimental Results for input parameters.](image_url)

Shown in Figure 21 is a plot showing the normalized fracture energy ($G_c$) as a function of adhesive thickness, while keeping the load, peel angle, and material properties fixed. Essentially, Figure 21 shows the fraction of the total system energy that was dissipated by the adhesive during debond. The fixed values of load, peel angle, and material properties can be found in Chapter 4 – Experimental Results, in addition to the scale factor used to normalize the $G_c$ values.
As can be seen from Figure 21, there is a significant change in $G_c$ given relatively small changes in the total adhesive thickness. In fact, the fracture energy can be seen to vary by approximately 20% over the range of adhesive thicknesses measured in this study (0.13 to 0.54 mm). Please note that the plot in Figure 21, only shows the predicted variation in $G_c$ due to different adhesive thicknesses, and in no way reflects the actual variation seen in the experimental data, where adhesive thickness could affect the real fracture energy and hence the load required to propagate a crack in the specimen.

While the adherend modulus was not found to vary significantly with temperature, the adhesive modulus can. Shown in Figure 22, are the changes in fracture energy as predicted by IC Peel for various adhesive moduli, both above and below the value used in this study ($E_a = 0.027$ GPa). The experimental window indicates the range of adhesive thicknesses encountered during testing. From this plot, it can be seen that changes in adhesive modulus can have a significant impact on the value of the predicted IC Peel fracture energy. For the ranges of moduli and adhesive thickness considered in Figure 22, the effects of moduli on fracture energy are approximately 3.5 times greater than those of adhesive thickness.
Figure 22: Fracture energy and adhesive thickness for various adhesive moduli. See Chapter 4 – Experimental Results for input parameters.

Figure 22 demonstrated that fracture energy can vary with adhesive modulus, however a single value of $E_a = 0.027$ GPa was used for the analysis in this study. Changes in adhesive modulus are largely due to different testing temperatures, and are considered to vary predictably. As has been shown previously however, adhesive thickness varies from sample-to-sample, and from batch-to-batch in an unpredictable manner. These variations in adhesive thickness contribute heavily to the scatter in the fracture data, and make useful durability predictions highly challenging. Considerable efforts were therefore dedicated to understanding and minimizing the effects of adhesive thickness variation on the fracture energies.

The focus on adhesive thickness rather than modulus variations can be partially justified by considering how these two parameters manifest themselves in the fracture energies. Both adhesive thickness and modulus are used in ICPeel to estimate the fracture energy of the system. Adhesive thickness however, plays a second role in the energy dissipated within the adhesive during debonding. Conceptually, the more adhesive material there is the more energy is dissipated. Additionally, when considering the stress equation for Beam on Elastic Foundation (BoEF) behavior, the relationship between adhesive thickness and modulus is proportional to $(E_a/h)^{1/4}$. This behavior can be seen in Figure 23, where the values have been normalized by the largest values considered in Figure 22 ($E_a = 0.27$, $h = 0.54$ mm). In Figure 23, $(E_a/h)^{1/4}$ varies by approximately 0.76 due to changes in adhesive thickness, but only 0.69 due the change in adhesive modulus. This demonstrates that adhesive thickness will have slightly more influence on BoEF like behavior than adhesive modulus, primarily because a thicker adhesive layer will dissipate more energy than a thinner layer.
As has been shown in this section, the energy partitioning in IC Peel is the result of the observed plastic deformation in the adherends. Additionally, this partitioning could be affected by the thickness of the adhesive layer, as fracture energy and adhesive thickness are thought to be linearly related. As a result, estimates of fracture energy were also found to vary significantly with input parameters such as adhesive thickness, and adhesive modulus. Peel angle on the other hand, was not found to have a significant impact on the calculated fracture energies.

**MATLAB Implementation of IC Peel**

The IC Peel spreadsheet used in partitioning the total T-peel system energy has proven to be extremely useful in this study. There are however some limitations of this analysis package. First, the data analysis process cannot be automated, and requires the user to manually input the specimen geometry and material properties as well as the measured load and peel angle. While possible, this process does not lend itself to calculating fracture energies for many thousands of different load and adhesive thickness points across the length of the specimen. Additionally, as calculations need to be performed on the upper and lower adherends separately, then added together. IC Peel calculations required the two spreadsheet rows for each load/adhesive thickness be connected, further complicating the analysis. These limitations required that IC Peel be adapted for use in MATLAB. While further automating the analysis process, the MATLAB IC Peel had some additional benefits in terms of connecting the variations in adhesive thickness to the measured load.

During testing, a separate CSV file is created for each rate tested that includes the measured load and crosshead displacement data. The purpose of the MATLAB code is to read in the data from each of these files, and connect each measured load value with an adhesive thickness measurement. Because of the fixed measurement locations on the majority of Batch 3
samples, the corresponding load values can be found at each of the four locations shown previously in Figure 8. While some edge to edge adhesive thickness variations were found in Batch 3 samples, these are typically much smaller than the variations along the length. The two edge measurements were therefore averaged in order to obtain adhesive thickness measurements at 16, 64, 112, and 160 mm of crosshead travel. Note that these crosshead travel locations are twice the measurement distance locations shown in Figure 8, as the Instron crosshead travels twice the distance that the crack front does, assuming steady-state propagation and negligible stretching of adherends. This is a direct result of having two peeling arms rather than one.

Having obtained adhesive thickness measurements at four sample locations and corresponding load values, a plot such as the one in Figure 24 can be obtained. In this test, three rates were tested: 500, 5, 0.05 mm/min as indicated by the black, red, and blue curves respectively. The orange open circles indicate the averaged four adhesive thickness measurements that were taken along the specimen length according to Figure 8. In this case, the variations in load correspond quite nicely to variations in the measured adhesive thickness. These variations are, however, quite small, ranging from 0.145 mm to just over 0.277 mm.

As adhesive thickness measurements were only obtained at the four discrete locations shown, a spline fit (MATLAB function *spline*) was used to obtain adhesive thickness measurements across the full range of crosshead displacements. Typically, load values would only be considered valid if the peeling reached a steady-state. Most often, this is indicated by the load values reaching a relatively constant plateau. Given the observed variations in adhesive thickness, many T-peel tests did not reach the classical “load equilibrium” indicative of steady-state debonding. As such, it was determined that all “valid” load values should be used in the analysis. From Figure 24, an initial spike in load can be seen in the 500 mm/min rate data before the load settles around 20 mm of crosshead displacement. This initial spike is almost always present during the first rate tested, and often on subsequent rates as well. This initial spike
however, is not thought to represent steady-state behavior, but rather the transition from the initial specimen curvature (as a result of bending the tabs back at 90°) to the steady-state curvature. The most extreme example of this curvature transition always occurs during the first rate, but can sometimes be observed to a lesser extent when transitioning between rates. This is to be expected anytime there is a change in the steady-state curvatures for two different loading rates.

In general, it takes 10-16 mm of crosshead travel for the crack to move past the point where the initial 90° bend affects the measured load. Therefore, the load values and fitted adhesive thickness values from Figure 24 were trimmed as shown in Figure 25. In this case, the load for the first rate (500 mm/min) was trimmed 16 mm to correspond with the first measured adhesive thickness value. The subsequent 5 and 0.05 mm/min rates were each trimmed 10 mm. Load and corresponding adhesive thickness measurements have thus been obtained for the portions of the test where the load values are considered to be representative of the system.

![Trimmed load and adhesive thickness measurements (27G)](image)

In many cases, using a spline fit to estimate adhesive thickness values in between known points works quite well. This is evident from Figure 24 and Figure 25. In some cases however, the spline fitting process does not work quite so well. This can be seen in Figure 26 and Figure 27. In Figure 26, the load plateaus around 100 mm of crosshead displacement. The adhesive thickness however, decreases rapidly after 160 mm. This is a direct consequence of the spline fit used. Because there are only four actual adhesive thickness measurements, the spline fitting function in MATLAB maintains the curvature of the fit after the fourth point. In this case, an unrealistically thin adhesive thickness was predicted for the end of the measured load.
In the case of Figure 27, the load for the last rate tested (0.05 mm/min) remained fairly constant after approximately 125 mm of crosshead travel. The fitted adhesive thickness however did not. In fact the fitted thickness numerically became negative, although this has no physical meaning. Negative adhesive thicknesses while obviously wrong, are quite rare, occurring in only five of the 85 tests where a fitted adhesive thickness was calculated.

Despite some issues resulting from fitting splines to the known adhesive thickness values, being able to correlate measured load values with the adhesive thickness is insightful. As material properties are considered constant, the ICPeel analysis can be run for each measured load and calculated spline thickness. Doing so yields potentially thousands of fracture energy values along the length of the specimen. These values can then be viewed as functions of the...
adhesive thickness. Shown in Figure 28, are the fracture energy values as a function of fitted spline thickness for the specimen shown in Figure 24 and Figure 25. The details and implications of this plot will be discussed in Chapter 4 – Experimental Results.

Figure 28: Fracture energy vs. fitted adhesive thickness (27G)

Regression Analysis and Equivalent Service Condition

Having conducted numerous T-peel tests on unaged specimens from different batches, a regression analysis can be performed to fit the data. Not only does this help with long-term durability predictions by allowing extrapolation to longer times, but fitting the data may also help separate trends. Additionally, this process can be performed independently of DMA shift factors. The data in this study, as well as other work have suggested that the shape of the master curve is similar to that of the loss tangent (tan δ). While DMA tests can be used to find the appropriate shift factors (a_T) for constitutive data, independent shifting using only T-peel data was performed in this study. To do this, the WLF Equation (4) which is appropriate for T_g and above can be used.

\[
\log(a_T) = \frac{-C_1(T - T_{ref})}{C_2 + (T - T_{ref})}
\]

Here T_g refers to the glass transition temperature of the adhesive. The T_g values for the two phases of the adhesive material were found to be in the vicinity of 86°F (30°C) and -130°F (-90°C) [24]. In Batches 1 and 2, tests were conducted at -65, -20, 15, 75, 120, and 180°F. Using trial values for the C_1 and C_2 constants, the shifted rates for each test can be found for a given reference temperature (180°F in this case). The shape of tan δ and the fracture data suggests that a parabolic fit would be appropriate. Thus, G_c can be found according to Equation (5).

\[
\log(G_c) = A + B \log(a_T \delta) + C[\log(a_T \delta)]^2
\]
Excel’s LINEST function was then used to find the A, B, and C constants in Equation (5) using the $\Delta T$ values from Equation (4). Excel’s SOLVER function was used to minimize the sum of the squares of the residuals between the fitted $G_c$ values in Equation (5) and the experimentally measured $G_c$ values from T-peel tests by finding the appropriate values of $C_1$ and $C_2$ in Equation (4). Because of the combined forms of Equation (4) and Equation (5), direct linear regression was not possible. In the case of the unaged data, $C_1$ and $C_2$ were found to be 77 and 1042 °F respectively. It should be noted that the exceptionally large $C_2$ value is due to the near linear fit of the fracture data temperature shifts.

Despite significant scatter in the unaged data, a reasonable fit was found using the method described above. This fit is shown as the gray curve in Figure 29. In Figure 30, the shift factors obtained with a DMA are compared to those found independently using only T-peel fracture data. The excellent agreement between shift factors obtained using two different measurement methods provides confidence that the relevant processes have been captured in the regression analysis.

Figure 29: T-peel master curve for all unaged data
Figure 30: Shift factor plot comparing DMA and T-peel shift factors. DMA data from [24].

While the master curve in Figure 29 demonstrates long-term behavior at a given reference temperature, the fitted parabolic curve does not account for the effects of aging. To do so, a series of curves can be found for each aging condition. First, the max fracture energy value from each aging condition was scaled by the peak of the unaged data. Next, Excel’s LINEST function was used to individually fit each of the three aging temperatures (180°F, 200°F, Temperature B), and calculate “aging shift factors” by Equation (6).

\[ \phi = \text{fitted slope} \times \text{aging time} + \text{fitted intercept} \]  

Equation (6)

When the log of the appropriate “aging shift factor” Equation (6) is added to Equation (5), the result is Equation (7).

\[ \log(G_c) = A + B \log(a_T \delta) + C \left[ \log(a_T \delta) \right]^2 + \log(\phi) \]  

Equation (7)

Equation (7) then, is a parabolic fit of the unaged data, with an additional term to account for the effects of wet aging. A comparison of the unaged and aged fitted curves resulting from Equations (5) and (7) can be found in Chapter 4 – Experimental Results.

While in service, the sponsor’s part is expected to experience a range of temperatures, each for a different amount of time. A combined equivalent service time can be found for a given reference temperature, using the principles of Time-Temperature Superposition (TTSP). Each service time and temperature is first shifted to a reference temperature of 180°F. These shifted times are then added to give an equivalent time at 180°F. This equivalent time is thought to be representative of both the combined times, and temperatures the part will likely experience while in use. Further details on this equivalent service condition will be given in Chapter 4 – Experimental Results.
Alternative Methods for Extracting Fracture Energy Values

In some cases, T-peel specimens can act more like an elastically deforming DCB rather than a peel specimen with significant plasticity. This occurs when there is little to no plastic deformation of the adherends. As previously mentioned, Dillard et al. [20], describe four common T-peel testing configurations or categories. In Category I tests, the adherends only deform elastically and self-similar debonding occurs. Here, self-similar debonding refers to the crack propagating at the same speed as the crosshead; thereby maintaining a constant distance between the crack front and the applied load. While self-similar debonding also occurs in Category II tests, the adherends are deformed plastically. Categories III and IV describe tests when the adherends deform plastically and elastically, but so not reach self-similar debonding. In these cases, the crack propagates faster than the crosshead is moving.

In those cases where little to no plastic deformation of the T-peel adherends occurs (Categories I and IV), the ICPeel calculations for fracture energy values are meaningless, as a steady-state condition is never achieved. To be able to accurately calculate fracture energy values, a new analysis method is needed. Unlike a DCB test, the crack length is not measured on T-peel specimens, so the standard DCB analysis cannot be used. Using simple beam theory however, an approximation of the fracture energy can be found in terms of only the measured load and the crosshead displacement.

Per simple beam theory, the sample cross section is assumed to remain perpendicular to the neutral axis. Additionally, this analysis assumes that there are only small displacements in each “beam” or adherend, and that the tabs are pinned as shown in Figure 31.

![Figure 31: Pinned and rigidly gripped specimen geometries (not to scale)](image)

The fracture energy, $G$, can be found for these assumptions by Equation (8).

$$G = \frac{P^2 a^2}{b EI}$$  

(8)
In Equation (8), \( P \) is the load, \( a \) is the crack length, and \( b \) is the specimen width. Additionally, \( E \) is the “beam” modulus, and \( I \) is the second moment of area. The displacement, \( \Delta \), can also be found in terms of the measured load, and crack length via Equation (9).

\[
\Delta = \frac{2Pa^3}{3EI}
\]  

Equation (9) may be solved for \( a \) in terms of \( \Delta \), with the result substituted into Equation (8) to obtain the fracture energy in terms of the displacement (Equation (10)).

\[
G(Pinned \ DCB) = \left(\frac{3}{2}\right)^{2/3} \frac{p^{4/3} \Delta^{2/3}}{bEI^{1/3}}
\]  

If instead of pinned, the DCB ends were bent back so as to form a T-peel specimen (Figure 31), and the “tabs” were rigidly gripped, Equation (11) from [20] can be obtained.

\[
G(Rigidly \ Gripped \ DCB) = \frac{p^2a^2}{4bEI}
\]  

Note here, the factor of four difference between Equations (8) and (11). If Equation (9) is again solved for \( a \), and substituted into Equation (11), Equation (12) can be obtained.

\[
G(Rigidly \ Gripped \ DCB) = \left(\frac{3}{2}\right)^{2/3} \frac{p^{4/3} \Delta^{2/3}}{4bE^{1/3}}
\]  

The fracture analysis for a pinned DCB serves as the upper bound for expected dissipated energy values as shown in Figure 32. As T-peel specimens are not pinned, the actual dissipated energy is thought to be significantly lower, closer to that of the rigidly gripped case also shown in Figure 32. Given the factor of four difference between these two approaches, the average fracture energy estimated by ICPeel is likely too high. In most cases, the sudden drop in \( G \), observed at around 115 mm of crosshead travel in Figure 32, is due to the complete debond of the specimen. As the sample has likely debonded at this point, these energy values then are invalid, and has been neglected in this analysis.
In reality, the true energy dissipated by the T-peel specimen would fall somewhere between the pinned and rigidly gripped cases as shown in Figure 32. This is due to the fact that there is an approximately 23 mm gap between the bottom of the grip, and start of the crack tip as shown in Figure 31. While not always the case, the T-peel adherend geometry and material properties used in this study, worked out such that the ICPeel fracture energy estimate were not an unreasonable approximation of the pinned DCB case. In fact, the ICPeel value was found to be between the pinned and rigidly gripped cases of Figure 32. Further work would need to be done using Castigliano’s second theorem to accurately determine what energy was dissipated by T-peel samples that did not exhibit significant plastic deformation in the adherends. As only a limited number of samples exhibited no plastic deformation, and the ICPeel estimates were found to be reasonable, ICPeel was used for all data analysis presented in the following sections.

**Approximation of Peel Strength Values**

For long-term durability predictions, fracture energy values are of the most interest as they are used in the construction of master curves. Additionally, ICPeel takes into consideration factors such as adherend plasticity in the calculation of $G_c$. The project’s sponsor commonly uses peel strength in their testing however, and there are times where a direct comparison between fracture energy values and peel strength would be useful. Peel strength is defined by Equation (13). If the specimen width is assumed to be constant (25.4 mm), then only the load would change between samples (when comparing unaged materials and assuming a constant adhesive thickness).

$$Peel\ Strength = \frac{average\ load}{bond\ width} \quad (13)$$
By varying the load in ICPeel but keeping all other parameters constant, a relationship between load and $G_c$ can be found. In Figure 33, the resulting fracture energy values are presented in terms of the measured load.

These values span all T-peel tests, with the exception of the reduced or zero curvature specimens. In these cases, the $G_c$ values from ICPeel are suspect anyway, so a peel strength estimation would not be very useful. By selecting a linear fit for the data in Figure 33, an approximate relationship between $G_c$ and peel strength can be found and plotted on a second y-axis along with fracture energy values.

While a better fit to the data could be obtained with a power law, such an approximation could not be plotted on a linear scale along with fracture energy values. Even with a linear fit, the agreement over expected loads is quite good, especially considering how much the load can vary for just a single rate and temperature condition. In Figure 34, the measured loads are plotted along with those calculated using the linear fit from Figure 33. For the two samples considered here (28E and 29B), the difference between the measured and calculated loads are well within any variation in the measured load itself. Thus any error associated with a linear fit is much smaller than the test error. While not exact, a peel strength approximation of $G_c$ can be used for the purposes of general discussion and comparison with data previously obtained by the project’s sponsor. This approximate peel strength can be found on many of the subsequent fracture energy plots, where the approximated peel strength is added as a secondary ordinate axis, thus allowing both to appear on the same figure.
Figure 34: Measured and calculated load for samples 28E and 29B
Chapter 4 – Experimental Results

Chapter 4 – Experimental Results has been removed from the public version of this document at the request of the project sponsor.
Chapter 5 - Peel Tests: Analysis of Multi-valued Systems and the Universal Peel Diagram

Development of a Universal Peel Diagram with ICPeel

The universal peel diagram (UPD) such as the one in Kim and Kim [3], is conceptually useful. In principle, it allows fracture energy values to be determined from the material properties, sample geometry, and measured peel load using only the diagram. If truly universal, many different systems can quickly be compared, as the axes are nondimensionalized by quantities such as adherend modulus, yield stress, and thickness. Although not discussed in the literature (to the author’s knowledge), the sigmoidal shape of the UPD suggests that peel test debonding loads could potentially be multi-valued, that is one, two, or three values of steady-state load could exist for a given fracture energy value, with all other parameters being fixed.

In order to explore this possibility in more depth, a hypothetical 90° peel specimen model was constructed in order to perform calculations in ICPeel. As with the UPD, ICPeel uses material properties, sample geometry, and the measure peel load to partition the total system energy and determine the fracture energy value(s). As ICPeel is in spreadsheet form, various combinations of input parameters can quickly be evaluated. Additionally, if the ICPeel results framed in the context of a UPD align with those obtained by Kim and Kim, the concept of multi-valued debonding loads is further supported. Further details on the theoretical basis for ICPeel can be found in Chapter 3 - Data Analysis.

The chosen hypothetical 90° specimen model, used an aluminum 2024-T3 (E = 72.4 GPa, σ_y = 331 MPa) adherend with a width of 25.4 mm. A generic polyurethane adhesive was also selected with an elastic modulus of 0.0423 GPa. As ICPeel does not accept a value of zero for adhesive thickness, a very small value of 1 µm was used. Recall from Chapter 3 - Data Analysis, that the relationship between adhesive thickness and modulus is proportional to (E_a/t)^1/4 for Beam on Elastic Foundation (BoEF) behavior. The effective stiffness (E_a/t) then becomes 42.3 TPa/m when an adhesive thickness of 1 µm is used. By making this adhesive layer very thin, the effective stiffness increases exponentially, and acts like a sample without an adhesive layer. This was done to closely replicate the analysis performed by Kim and Kim in [3] where they did not have an adhesive layer.

Using ICPeel with the geometry and material properties described above, a fracture energy value (G_c) of 1000 J/m^2 was chosen as a target. An elastic-perfectly plastic adherend model was used to maintain consistency with [3]. The load was then iteratively varied for an adherend thickness of 0.1 mm, until a value of approximately 1000 J/m^2 was obtained. In this case, a load of 109N was found to correspond to G_c ≈ 1010 J/m^2. Additional load values were iteratively selected over a wide range of adherend thicknesses (0.1 to 70 mm) to obtain G_c values closely matching the target value of 1000 J/m^2. The load and the adherend thickness values were then normalized according to Equations (14) and (15).
\[ \bar{t} = \frac{\sigma_y^2 t}{6EG_c} \]  

(14)

\[ \bar{p} = \frac{p}{G_c} = \frac{F}{G_c b} \]  

(15)

Here, \( t \) is the adherend thickness, \( p \) is the “peel strength” or force per unit width, and \( b \) is the bond width. In manually varying the load, it quickly became apparent that relatively small changes in the load would sometimes cause random variations in \( G_c \). In one case, a 0.05% change in load would cause between 0.07% and 0.18% changes in \( G_c \). As ICPeel uses an iterative method for converging on a solution, there is no way to pick a load that would yield exactly the same \( G_c \) for a different adherend thickness. The step size used to control the degree of convergence is fixed in ICPeel. Given the less than 2% observed variation, the step size was not modified in this study.

By plotting the normalized load (\( \bar{p} \)) and normalized adherend thickness (\( \bar{t} \)), a plot very similar to the universal peel diagram (UPD) from [3] was obtained as shown in Figure 35. This analysis procedure was repeated for a total of three different fixed adhesive fracture energy values. As can be seen, there is good agreement between the results plotted using \( G_c \approx 1000, 10000, \) and \( 20000 \) \( J/m^2 \). The plot also shows that not all values of \( \bar{t} \) are admissible for every fixed value of \( G_c \). For low \( G_c \) values, the adherend thickness \( t \) must be smaller than the 0.1 mm value used in this analysis in order to obtain very small \( \bar{t} \) values. This is evident by the lack of \( \bar{t} \) values smaller than \(~0.02\) in Figure 35 for \( G_c \approx 1000 \) \( J/m^2 \).
The diagonal \( \eta \) axis in Figure 35 represents a nondimensionalized peel force calculated according to Equation \((16)\).

\[
\eta = \frac{6Ep}{\sigma_y^2t}
\]

The other diagonal axis represents the fracture or decohesion energy \( G_c \). By using these auxiliary axes, the fracture energy value can be found if \( \eta \) is known. This is important, because the sample geometry, material properties, and measured force per unit width \( (p) \) are usually known or directly measured experimentally, but \( G_c \) is not.

In [3], a number of samples were tested with varying thicknesses of Cr and Cu deposited by e-beam evaporation onto a Si substrate. The thickness of Cr and Cu were such that the combined thickness would add up to 2 \( \mu \)m. An additional Cu layer of varying thicknesses (15-77 \( \mu \)m) was then added through electroplating, resulting in five different total film thicknesses. These five film thicknesses, produced by Kim and Kim, were then examined in ICPEel for the 0.05 \( \mu \)m Cr/1.95\( \mu \)m Cu case. As was done for the UPD in Figure 35, the input load was varied in order to achieve a constant \( G_c \) across the entire range of film thicknesses.

Without Kim and Kim’s raw data, there is no way to know for certain if the chosen loads, and resulting fracture energy values are accurate. Based on the 25 lb (111 N) load cell used in [3] however, the loads \( (p \leq 1.64N) \) used in ICPEel should be reasonable. In fact, Kim and Kim report an average peel strength of \(~600\ J/m^2\) which translates to a force of approximately 1.2 N.
The process of choosing a load for each adherend thickness was repeated for five separate fixed $G_c$ values while keeping the load values under 1.64 N. The results from this analysis can be seen in Figure 36. Based on the scale in Figure 36, a significant number of the points on this graph have $\bar{t}$ values that are much smaller that the lower limit of the graph from [3] (0.001).

![Figure 36: Universal Peel Diagram for copper adherends](image)

When examining the copper and aluminum UPD’s together (Figure 37), the copper adherends exhibit much the same slope as the aluminum adherends, however they have $\bar{p}$ values that are almost a factor of three higher than those predicted by the aluminum UPD. In addition, both the copper and aluminum adherends have $\bar{p}$ values that are almost an order of magnitude smaller than those predicted in [3].
The UPD presented in Figure 37, also appears to have a much steeper slope for $\bar{t} < 0.1$ and lower $\bar{p}$ values than those seen in [3] across the entire range of $\bar{t}$. These differences are not well understood, with the assumption being that these differences are the result of different approaches between [3] and ICPeel. Further study of both analysis methods will be required in order to identify the cause of the steeper slope in Figure 37.

**Comparison of Analysis for 90° Peel Tests**

As seen in Figure 38, there is good agreement between the UPD from [3], and the UPD obtained with ICPeel for aluminum and copper adherends for $\bar{t}$ values greater than approximately 0.3. As previously mentioned, there are significant differences both in terms of the slope and magnitude between the ICPeel analysis and the analysis performed by Kim and Kim.
While a $\bar{p} - \bar{t}$ relationship was found analytically by Kim and Kim, these relationships include a parameter $\alpha$ where $\alpha = 1 - \sin \theta_B$ and $\theta_B$ is the angle between the tangent to the adherend, and the substrate at point B. This point indicates the last location where the adherend remains bonded to the substrate as indicated in Figure 39. This angle would presumably depend on the material and thickness of the adherend, as well as the compliance of the substrate (or adhesive layer if present). As indicated by Figure 40, the $\bar{p} - \bar{t}$ plot depends greatly on the value of $\theta_B$. Without knowing the value of $\theta_B$, it would be impossible to construct a UPD using the analytical relationships found by Kim and Kim.
In fact, it appears that Kim and Kim may never have used the analytical equations to produce the universal peel diagram, but instead conducted numerous peel tests and calculated $\bar{p}$ and $\bar{t}$ for arbitrary values of $G_c$ ($\gamma$ in [3]). The data was likely then translated along the diagonal $G_c$ line ($\eta = \text{constant}$) until the UPD was obtained, thereby finding the appropriate $G_c$ value. In doing so, multiple $p$ values for the same adherend thickness were required to all have the same $G_c$. This assumption would only be clear however, if multiple specimens were tested with the same adherend thickness, but different loads were measured. Given the very small loads required by the copper adherends, these variations could have easily been discounted as simple instrumentation or sample to sample variation.

While the analytical equations do not appear to have been used in [3] to construct the UPD, they were used to create Figure 40 below. This graph from [3] is effectively the analytical version of the UPD (plotted on linear axis) for various $\theta_B$ values. If the UPD from ICPeel for the copper and aluminum adherends is also plotted on a linear scale, Figure 41 is obtained. The similarity between Figure 40 from [3] and Figure 41 is quite noticeable for the aluminum adherends, and to a lesser extent the copper adherends. Not only is the shape very similar for the aluminum adherend, but the relative magnitudes only differ by a factor of two. This difference is likely a result of the difference between $\theta_B$ chosen by Kim and Kim, and the angle $\theta_0$ that results from the ICPeel analysis. In fact, $\theta_0$ for the aluminum adherend varied from $0.3^\circ$ to $39.7^\circ$ over the range of $\bar{t}$, while $\theta_B$ only included discrete values of 0, 10, 20, and $30^\circ$. For the copper adherend, $\theta_0$ varied from $0.02^\circ$ to $32.2^\circ$. A potentially useful exercise would be to take the $\theta_0$ from ICPeel, and use the analytical equations from [3] to find $\bar{p}$ and $\bar{t}$.
In [3], Kim and Kim appear to have considered root rotation by using the Winkler foundation model to describe the compliance of the substrate with a normalized relative substrate stiffness value ($\bar{n}$). This normalized stiffness value however, does not seem to be used in constructing the universal peel diagram, or in the equations relating the normalized peel force ($\bar{p}$).
to the normalized adherend thickness ($\bar{t}$). It is possible that Kim and Kim used the normalized substrate stiffness as a correction factor to make the universal peel diagram fit the experimental data. If used as a vertical shift factor, the normalized substrate stiffness could account for the difference between the UPD from [3], and the ICPeel UPD even though Figure 40 and Figure 41 closely resemble each other.

Conclusions

In examining the elastic-plastic behavior of 90° peel tests and the universal peel diagram, a similar diagram was produced using ICPeel. Copper adherends matching the geometry from [3], and aluminum adherends from [2] were analyzed by varying the load and adherend thickness in order to maintain a constant fracture energy value. This demonstrated that peel tests could in fact be multi-valued, indicating that a sample with a single fracture energy value and adherend thickness could have potentially three distinct but valid load values. Significant differences however, were found between the numerical values for copper and aluminum in the ICPeel UPD. Additionally, the numerical values of the ICPeel UPD are quite different from those in [3]. While the results obtained so far are encouraging, further work would be required to determine what causes the differences between the two universal peel diagrams.

Peel tests are a very popular due to a number of factors including their compact geometry, and analysis that is independent of crack length. Traditionally, sample geometry and an average load values is all that is required for ICPeel or the analysis in [3] to yield a fracture energy value that is representative of the system being analyzed. The concept of multi-valued systems introduced by the universal peel diagram (UPD) however, complicates the analysis. If a real system has nondimensionalized peel force ($\eta$) values that fall in the multi-valued portion of the UPD, then multiple valid fracture energy values will be obtained for a given load. In designing a peel test then, $\eta$ should be checked prior to testing for the anticipated test conditions. Designing a peel test outside of the multi-value region of the UPD helps to insure that changes in fracture energy are the result of test parameters (aging, test conditions, etc.) rather than the multi-valued nature of the UPD.
References


