In Situ Tomography of Microcracking in Cross Ply Carbon Fiber Composites with Pre-existing Debonding Damage

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ABSTRACT

In Situ Tomography of Microcracking in Cross Ply Carbon Fiber Composites with Pre-existing Debonding Damage

Daniel J. Traudes

Carbon fiber based composites are an essential material in weight-critical applications such as in the aerospace industry. However, these materials are susceptible to damage such as matrix microcracking and fiber/matrix debonding (diffuse damage), which occurs at stresses much lower than the failure stress.

A T700/M21 $[0/90]_s$ laminate was tensile loaded to introduce diffuse damage and prepared for a study on the initiation of transverse microcracks. The material was tensile loaded in a $[+45/−45]_s$ orientation to induce diffuse damage. A diffuse damage indicator was developed by measuring the decrease in shear stiffness. Samples with diffuse damage levels of 0, 0.05, 0.10, 0.15, 0.20, and 0.25 were prepared to be tensile tested in a $[0/90]_s$ orientation to induce microcracks.

A successful development of the microcracking test procedure was performed. The edge of the material was studied with optical microscopy and x-ray to establish the structure of the fiber bundle geometry when undamaged. A sample containing microcracks was treated with diiodomethane dye penetrant, which successfully highlighted microcracks during x-ray imaging. The application time was not sufficient to pro-
duce consistent x-ray images over time, so a 45 minute soak time was recommended instead. The same damaged sample was subjected to a tomographic scan without a dye penetrant and while unloaded. Transverse microcracks were successfully identified from the data, although the results were not clean enough and likely omitted some smaller microcracks. Results are expected to be cleaner if performed during tensile testing.

Future tensile testing will quantify the induced crack density of samples containing various degrees of initial diffuse damage, either using x-rays with a dye penetrant or using x-ray microtomography.
ACKNOWLEDGMENTS

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### LIST OF SYMBOLS

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Chapter 1

Introduction

Fiber based composites are fast becoming a dominant material in many industries for their notable strength versus weight properties, especially in weight-critical areas such as aerospace applications. Their unique properties stem from the fact that composites are a combination of two or more materials of contrasting properties. This, however, complicates the types of damage that are experienced in the loading of any composite material.

In fiber based composites, damage occurs at much lower loads than the ultimate failure load. Because of this, it is crucial that these initial damage mechanisms are understood, in order to make an informed assessment about the structural health of a composite material. Matrix microcracking and fiber/matrix debonding, or diffuse damage, are typically the first types of damage to be introduced. Thus, it is essential that their means of initiation, as well as their effect on each other, are understood. Any insight into the damage mechanisms behind microcracking and debonding contributes a wealth of knowledge towards the damage mechanisms that will be experienced later in the load life: delamination, fiber breaks, and overall material failure.

The goal of the present research was to determine what effect diffuse damage has on matrix microcracking. Since transverse cracks are a measure of toughness in the
longitudinal direction, this work sought to explain the effect of diffuse damage on longitudinal toughness. The scope of the thesis encompassed the following experimental work:

- Preparation of a T700/M21 [0/90]s material system, including manufacturing of material samples for shear tensile testing, and later microcrack-inducing tensile testing.

- Shear tensile testing to induce diffuse damage. A diffuse damage parameter was defined, whose value was dependent on the reduction of shear stress.

- Preparation for testing to induce transverse microcracks. Tensile samples were designed for use in a small testing machine to be used in situ with an x-ray tomograph. Preliminary tests included examination of the laminate edge’s geometry as well as a feasibility study for x-ray dye penetrants.

The material preparation and shear testing was performed in conjunction with the work of Dr. Hedi Nouri. Due to procurement issues, the x-ray computed microtomography (micro CT) microcracking campaign of predamaged samples was not completed. Instead, a successful development of the test procedure was performed. This portion of testing is set to be completed by the end of July 2012, and will be followed by the publishing of the complete study in a journal paper.
Chapter 2

Damage in Carbon Fiber Composites

Due to their high strength and low weight, carbon fiber reinforced polymers (CFRPs) have become a prevalent material, especially in the aerospace and sports industries. Their properties are a product of combining two materials with contrasting features: carbon fibers and a matrix material (typically epoxy). The carbon fibers provide high strength and stiffness. The polymer matrix, while not of high strength, transfers loads between fibers and prevents bucking and bending. This combination of elements results in a composite material with high strength and low weight.

With carbon fibers aligned in one direction, a plate of fiber-reinforced polymer is inherently anisotropic. To adapt the material for loading in various directions, a laminate is created by stacking individual plates, or plies, at various defined angles to add stiffness in those directions. The axis notation for a single ply can be seen in Fig. ??, where axis 1 is aligned with the longitudinal fiber direction, axis 2 is in the transverse direction, and axis 3 is in the thickness direction. When stacking multiple plies, axis 1 of a reference layer is designated as $0^\circ$ and subsequent plies are designated by the angle from their fiber direction to this axis. When dealing with a
complicated material composition such as this, it becomes imperative to understand the types of damage which occur, in order to ensure safe operation of the structure [?].

![Figure 2.1: Axis orientation of a single ply of a fiber-reinforced polymer](image1)

### 2.1 Damage Types

Damage in laminates is a multi stage phenomenon. Typically the first damage that is seen comes in the form of matrix microcracks and debonding of the fiber/matrix interface [?]. As the number of cracks and debonded surfaces increase, the material experiences a linear stiffness reduction. Microcracks nucleate other forms of damage: delamination at the ply interface, fiber breakages, and entry by outside liquids. The addition of delaminations and fiber breakages further reduce the macroscopic stiffness. These various forms of damage can be seen in Fig. ??.

![Figure 2.2: Damage modes in cross-ply laminates. From [?]](image2)
Laminar damage can be sorted into different classification families:

- **Interlaminar and Intralaminar:** Interlaminar damage refers to phenomena occurring between the plies, or laminae. Delamination falls into this category. Intralaminar damage thus refers to damage occurring within an individual ply. Fiber breaks, microcracks, and debonding fit this description.

- **Discrete and Diffuse:** Delaminations, fiber breaks, and microcracks are all forms of discrete damage. They are both observable and quantifiable, either by counting or measuring physical dimensions. Debonding, on the other hand, is classified as diffuse damage. There is no way to directly observe debonded fibers in a CFRP, and thus no method to quantify the damage. Instead, debonding damage is indirectly defined based on the material’s loss of stiffness.

- **Longitudinal, Transverse and Shear Loading States:** By focusing on simplified loading states, the intralaminar damage modes can be separated further. In the case of pure longitudinal loading (in the fiber direction), fiber breaks would be the dominant form of damage, as seen in Fig. ??.

  This loading state is referred to Mode I in the present paper. Similarly, the case of pure transverse loading (perpendicular to the fiber direction) is associated with transverse microcracking of the matrix between fibers (Mode II loading), as seen in Fig. ??.

  Lastly, a state of pure shear loading will result in debonding at the fiber/matrix interface (Mode III loading), as seen in Fig. ??.

  These are simplified views of laminate loading cases. In real world applications, a laminate will experience a combination of these loads, thus resulting in a similar combination of damage modes.

The present research is focused on investigating the relationship between diffuse damage and transverse cracking. Due to the high tensile strength of carbon fibers,
fiber breakage is typically experienced at very high loads, closely followed by the overall failure of the material. In real world diagnostic cases, this information is not useful, since its occurrence gives little advance warning for material end of life. In contrast, debonding and microcracking, as early forms of damage, are more useful indicators of material lifetime. These phenomena have been extensively studied individually. Instead, the goal of the present work is to determine the relationship between these two phenomena.

### 2.1.1 Transverse Cracking

Research on microcracking is generally performed on a cross ply \([0_m/90_n]_s\) laminate, ensuring that fibers are loaded only longitudinally or transversely. This is a pure transverse loading state for the 90° plies (except near the edges), promoting the appearance of transverse cracks. Transverse microcracking also occurs in \([90_m/0_n]_s\) laminates, but the strain to initiate microcracking is lower for surface 90° plies, since those plies are constrained only on one side. Besides 0/90 laminates, transverse microcracks can form in the 90° plies of any laminate [?].

With a cross-ply laminate, damage in the 0° plies is determined by fiber properties, while damage in the 90° plies is controlled by the matrix and/or fiber/matrix interface properties. Transverse cracks in the 90° plies develop in the fibre direction and extend from the free edges of the laminate. They extend over the full transverse ply thickness.
and, for quasi-static tests, most cracks extend over the full test specimen width [?]. When crack density is low, new cracks tend to form midway between existing cracks, where stress is at a maximum. At high crack densities, new cracks tend to form close to existing cracks instead [?].

In tensile tests cracks are introduced with increasing levels of strain. Transverse cracks first start to appear in off-axis plies at strains in the range of 0.2 to 0.5% [?]. The number of microcracks increases rapidly after this until a saturation value for crack density is reached. In general, when a sample is held at constant strain no new cracks are introduced. However, when held at constant stress a significant amount of new cracks may be seen [?].

Certain physical characteristics of a fiber composite have an effect on the introduction of crack damage. For instance, the presence of defects on the edge of a sample can result in a higher crack density, as well as a higher number of non-width spanning cracks [?]. Removing edge defects by polishing can delay the onset of transverse cracking and reduce crack density [?].

**Transverse Ply Thickness**

Transverse ply thickness is one of the main physical characteristics affecting the formation of microcrack damage. The strain required for the onset of cracking increases as thickness of the 90° ply decreases. This onset strain can exceed the failure strain of the composite for sufficiently thin plies, resulting in the suppression of crack damage [?].

The total number of cracks that develop is similarly dependent on transverse ply thickness. For a glass fiber reinforced polymer (GFRP) with longitudinal ply thickness of 0.5 mm, fewer transverse cracks form for transverse ply thicknesses less than 0.4 mm, and complete crack suppression is seen at a transverse ply thickness of 0.1 mm. And while transverse cracks instantly spread across the width of the
ply for transverse thicknesses greater than 0.4 mm, smaller non-spanning cracks were observed for thicknesses between 0.1 and 0.4 mm, which propagated as load increased [?].

In the case of CFRPs, it is likely that crack suppression would also occur, if transverse plies were thin enough. However, the thinnest 90° plies that have been tested were only half the thickness of the 0° ply [?]. In CFRPs, microcracks always form across the full width of the ply [?].

Subsequently, crack density/spacing is also affected by the thickness of the 90° ply. For a given strain, the spacing between cracks increases as the thickness of the transverse ply increases. As the applied load increases, the crack density approaches a saturation value dependent on the transverse ply thickness, as can be seen in Figure ?? [?].

\[ \rho = \frac{h}{L} n_c \]  \hspace{1cm} (2.1)

where \( \rho \) is crack density, \( n_c \) is the number of microcracks and \( L \) is the gauge length, a chosen observation window that must be large enough to provide a stable and accurate
crack density value for the sample. This value of crack density is then valid for all ply thicknesses of a certain material.

### 2.1.2 Debonding

Research on fiber/matrix debonding focuses on the use of a \([\pm 45]_s\) laminate layup. This orientation approximates a pure shear loading state, promoting the occurrence of debonding. In this orientation, microcracking plays only a minor role in the overall failure of the material. Instead, stiffness reductions precede the introduction of cracks. For a \([\pm 45]_2s\) CFRP loaded in tension, cracks only start to appear at about 40% of ultimate stress, while shear stiffness has already dropped 50% at the same point [?]. Microcracks only develop at this point after a substantial amount of interface debonding, which decreases the load transfer between matrix and fibers. The agglomeration of debonded surfaces leads to the formation of larger-scale matrix cracks (as seen in Fig. ??) [?].

![Image](image.png)

Figure 2.5: The joining of diffuse damage to form a microcrack [?]

### 2.2 Mesoscale Damage Modeling

In order to model the behavior of a laminated composite with damage, the damage model of Ladeveze and Le Dantec is used, derived in the following [?]. This model is phenomenonically based and derived from continuum damage mechanics theory.
The model begins by focusing on an individual ply. The model is formulated under the condition that composite damage mechanisms are inherently anisotropic. Anisotropy is created by introducing scalar damage variables $d_1$, $d_2$, and $d_6$, which are constant through the ply thickness. Each is an indicator of a type of composite damage: $d_1$ for fiber breaking, $d_2$ for transverse cracking, and $d_6$ for fiber/matrix debonding. The damaged material strain energy is subsequently written in the form:

$$
E_d = \frac{1}{2} \left[ \frac{\langle \sigma_{11} \rangle^2}{E_1 (1 - d_1)} + \frac{\langle -\sigma_{11} \rangle^2}{E_1^0 (1 - d_1)} + \frac{\langle \sigma_{22} \rangle^2}{E_2^0 (1 - d_2)} + \frac{\langle -\sigma_{22} \rangle^2}{E_2^0} + \frac{\sigma_{12}^2}{2G_{12}^0 (1 - d_6)} - \frac{2\nu_0^1 \sigma_{11} \sigma_{22}}{E_1^0} \right]
$$

(2.2)

where

$$\langle a \rangle_+ = a \quad \text{if} \ a \geq 0; \quad \text{otherwise} \ \langle a \rangle_+ = 0$$

The stress considered is the effective stress in the ply:

$$\bar{\sigma} = K_0 : \varepsilon(U) \quad (2.3)$$

When fiber breaks and microcracks are loaded in compression, they close up and have no effect on the loading behavior. Thus, the strain energy equation is formulated such that when a negative value for $\sigma_{11}$ or $\sigma_{22}$ is entered, there is no contribution to the strain energy from $d_1$ or $d_2$.

The damage evolution forces are given by

$$\begin{align*}
Y_1 &= \frac{\partial E_d}{\partial d_1} = \frac{\langle \sigma_{11} \rangle^2}{2E_1^0 (1 - d_1)^2} \\
Y_2 &= \frac{\partial E_d}{\partial d_2} = \frac{\langle \sigma_{22} \rangle^2}{2E_2^0 (1 - d_2)^2} \\
Y_6 &= \frac{\partial E_d}{\partial d_6} = \frac{\sigma_{12}^2}{2G_{12}^0 (1 - d_6)^2}
\end{align*}
$$

(2.4)

The damage mechanism described by $Y_1$ is brittle, while those of $Y_2$ and $Y_6$ are progressive, then brittle mechanisms. The main idea is that for quasistatic loads, the
damage state depends on the maximum value of the damage force over the history of the structure.

The dissipation due to damage mechanisms is given by

\[ Y_1 \dot{d}_1 + Y_2 \dot{d}_2 + Y_6 \dot{d}_6 \geq 0 \]  \hspace{1cm} (2.5)

When using energy-based methods, the formation of a microcrack is predicted when the energy released by its formation \((G_m)\) is greater than the microcracking fracture toughness \((G_{mc}, \text{measured in } J/m^2)\). \(G_{mc}\) is a material property that characterizes the sensitivity of a material to microcracking, and can be applied to various lamination profiles of the same material [?].

![Figure 2.6: Crack density measurements taken at quarter marks along the sample width](image)

To begin, a clear protocol for defining the crack density must be established. The main problem is encountered with the definition of \(n_c\). In the presence of non-spanning cracks, \(n_c\) is dependent upon the choice of intersection line, along which microcracks are counted. A simple solution involves drawing intersection lines down the center and quarter marks of the ply width (see Fig. ??). The number of microcracks that intersect each line is counted, crack densities \(\rho_{1/4}, \rho_{1/2}, \text{and } \rho_{3/4}\) are calculated, and the three values are averaged to determine \(\rho\):
$$\rho = \frac{\rho_{1/4} + \rho_{1/2} + \rho_{3/4}}{3} \quad (2.6)$$

The energy release of cracking is dependent on both the potential energy of the material, $E$, and the total area of the microcracks, $A$. When microcracks are introduced, $A$ increases and $E$ decreases. Because only the transverse ply is damaged by transverse cracks, the change in the overall laminate potential energy is equal to the change in the transverse ply potential energy, $E_P$. Thus, for a finite fracture mechanics microcracking model, the total energy released per unit crack area due to the formation of a new microcrack ($G_m$) can be calculated:

$$G_m = -\frac{\partial E_P}{\partial A} \simeq -\frac{\Delta E_P}{\Delta A} \quad (2.7)$$

When in displacement control, this microcracking energy release rate can be written as [?]:

$$G_m(\rho) = \frac{h}{2} \bar{\sigma}_{22} \frac{1}{\tilde{E}_2} \frac{d'(2\rho) - d'(\rho)}{\rho} \quad (2.8)$$

Where $\bar{\sigma}_{22}$ is the effective stress within the ply, i.e. the stress that would exist under the current load if no microcracks were present. $h$ is the thickness of the 90° ply. $d'$ is the damage indicator for transverse cracking. $\tilde{E}_2$ is the Young’s Modulus for the ply in which the microcrack is created. This is obtained by homogenizing the crack damage with:

$$E_2 = \tilde{E}_2(1 - d'(\rho)) \quad (2.9)$$
2.3 Damage Observation Methods

2.3.1 X-ray Inspection

2D x-ray techniques are useful in detecting the presence of transverse cracking, fiber breaks, and delamination. Both the position and length of a microcrack can be observed from a 2D x-ray image. If the contrast between damage and solid materials is not sufficient, an x-ray sensitive penetrant may be utilized. When swabbed along the edges of a laminate, the penetrant is absorbed by adjacent cracks and delamination areas, increasing their contrast in the x-ray image. However, this method can be time consuming and is limited to damage networks which intersect sample edges.

2.3.2 Edge Examination

During loading, transverse cracks are opened and can be observed from the outer edge of the laminate. The number and position of cracks can thus be recorded either visually or with the aid of optical microscopy. This method has the advantage of simplicity and the ability to perform it in situ. However, it can be difficult to distinguish cracks from edge flaws, and the lengths of cracks cannot be determined. To determine crack spanning with a reasonable amount of precision would necessitate the use of microscopy performed in parallel on both sides of the material. The method is also restricted to the laboratory setting, because CFRP edges are rarely exposed in real world applications.

2.3.3 Acoustic Emission

It is also possible for damage to be quantified through acoustic emission (AE) techniques. Using acoustic microphones, the vibrations produced by cracking can be recorded, thus providing a time-relative number of damage events in the material. The location of damage can be determined by comparing time of flight of the signal...
at separate sensors. It is possible to differentiate types of damage by correlating low amplitudes with matrix cracking, medium amplitudes with delaminations, and high amplitudes with fiber breakages [?]. But even with this method, AE can’t differentiate between crack initiation and crack propagation damages (however, initiations are more numerous in CFRPs). Slowly propagating cracks may not be detected at all, and comparison of amplitude values can be difficult over different tests and samples.

### 2.3.4 Electrical Detection

As carbon fibers are conductors of electricity, it is possible to estimate damage from variations in electrical resistance. Epoxy matrix is insulating, so an electrical path through the material is dependent on contact between neighboring fibers. Conductance is related to the fiber volume fraction of the composite. As a result, damage from a fiber break will cause fiber volume fraction to decrease and trigger an increase in the overall composite’s resistance. Similarly, microcracking and delamination result in a jump in resistance, due to a decrease in the number of fiber-to-fiber conduction paths through the composite [?]. Since the method uses reinforcement carbon fibers as sensors, no modifications to the material are necessary. Furthermore, the size and location of delamination can be determined by applying an electric current in the fiber direction and observing the resistance of electrodes on the top and bottom surfaces [?].

### 2.3.5 C-Scan

C-scan techniques have been shown to successfully image the 2D network of transverse cracks in a [0/90/0] laminate, including cracks which partially spanned the coupon width and cracks which were completely internal [?]. The drawback to this approach is that only a 2D image of the front face can be obtained, meaning it is not possible to determine which ply contains crack damage. It is also not possible to use c-scan
for in situ load testing.

2.3.6 FBG Sensors

It has also been shown that transverse crack density may be determined through the use of small-diameter fiber Bragg grating (FBG) sensors. When embedded in the $0^\circ$ ply, the change in the sensors’ reflection spectra is indicative of transverse crack initiation. These results come with the limitation that only the crack density can be computed, and therefore precise positions and lengths cannot be determined through this method [?]. Chirped FBG sensors, in which the grating period increases at a constant rate, are able to detect the longitudinal position of microcracks [?], but crack lengths cannot be measured in the case of non-spanning cracks. A more problematic observation is that as crack density approaches saturation, it becomes too difficult to pinpoint dip points in the spectrum.

2.3.7 Micro CT

X-ray computed tomography (CT) provides several advantages over traditional methods in the quantification and imaging of transverse cracks, because it allows for the non-intrusive, non-destructive internal imaging of an object. Using a high resolution Synchrotron Radiation Computerized Tomography (SRCT) system, it was shown that transverse cracking networks could be visualized. SRCT visualization was performed in situ with tensile testing, allowing for the complete internal damage sequence to be observed, including transverse cracks at 20% of failure load, $0^\circ$ splits at 40%, and large delaminations at 60% [?].
Chapter 3

X-ray Computed Tomography

3.1 Basic Concepts

X-ray computed tomography is performed by reconstructing a 3D model of an object’s external and internal structure, based on a set of 2D x-ray projections of the object from different viewing directions. Grey scale values in the 2D x-ray image and 3D reconstruction (tomogram) correspond to x-ray absorption values of the material, which in turn is based on material density and atomic number. This enables the use of helpful techniques in post-processing, such as segmenting materials based on density, or rendering material in a specific density range transparent.

Tomography systems consist of an x-ray source and a detector plate located on opposite sides of a chamber, with the detector plate oriented normal to the direction of x-ray emission. The object of interest is placed between the source and the detector. After each 2D projection is recorded, the object is either translated or rotated (depending on the type of system) by a known amount, and the next projection is recorded.

Industrial CT systems are particularly well suited for the job of composite damage characterization, since their voxels (the 3D equivalent of pixels) can be as small as 1
cubic micrometer. At this scale, these systems are able to capture micro-scale damage in composites, such as fiber breaks and matrix microcracks. Such setups are referred to as micro CT systems for this reason.

3.2 Process

The first step in the CT of an object is to take a reference measurement. During this step, the object of interest is removed from the path of x-ray emission. In place of the object, only a reference material (typically air) remains in the reconstruction region (the area of interest between the x-ray source and detector which encompasses the object at all rotation angles). The goal of the reference measurement is to determine how many photons are counted by the detector, out of the fixed number of photons which leave the source. The resulting detector measurement thus corresponds to the reference material of known density. The density of subsequent materials can therefore be determined by correlating the number of photons counted to the number of photons counted in the reference measurement [?].

After reference measurements, the object of interest is moved back into the reconstruction region and a tomography scan commences. First a collection of 2D x-ray images is recorded, either as stack of parallel slices, or a set of radial slices. From these 2D projections a 3D tomogram is reconstructed using traditional filtered back-projection. This involves a Fourier transform of the projection data, noise filtering, an inverse transform, and finally a back projection to place the data in 3D space. The 3D tomograms that are generated with micro CT range between $500^3$ voxels and $2000^3$ voxels. Each voxel has at least 816 bits of grayscale information [?].
3.2.1 Cone Beam Micro CT

Cone beam CT systems employ a divergent-beam x-ray source. Photon paths from the source to detector form a cone shape. The systems are well-suited for volumetric scans and make up the majority of micro CT systems used in laboratories. Because of the non-parallel photon paths of the beam, cone beam systems provide a noisier image with a lower spatial resolution than SRCT systems. Data is subject to blurring and distortion, which increases as angles from the normal increase. Inaccuracies are also inherent in the reconstruction process. Problems arise because the beam source emits frequencies over the entire x-ray band, but the reconstruction process assumes that all photons are at the median frequency. Despite these shortcomings, these laboratory systems provide adequate volumetric results, at a relatively low cost and in a compact package.

3.2.2 Synchrotron Micro CT

SRCT offers higher spatial resolution and contrast sensitivity than typical commercial micro CT systems. The x-rays produced by SRCT are essentially parallel, monochromatic and coherent. However, object size is limited by the width of the x-ray beam. Voxel sizes for SRCT systems are typically in the range of 1 to 10 \( \mu \text{m} \). Intensity, as compared to usual x-ray generators, is \( 10^6 \) times larger, with photons
that span from infrared to hard x-rays (up to 300–400 keV). Because of the high intensity of SRCT systems, it is possible to filter out all but a narrow band of x-ray frequencies. This creates a monochromatic band, still with enough energy for a quality x-ray image. The result of this is that reconstruction results are more accurate because the x-rays are of a single known frequency. An example of the quality and resolution improvements over tube-based systems is shown in Figure 3.3. The downside to the technology is that access is more limited than cabinet cone beam systems, since SRCT requires large, expensive facilities to operate.

Figure 3.3: Synchrotron (left) and tube-based (right) micro CT slices of the same sample. The voxel sizes are 5 µm and 12 µm, respectively.
3.3 Advantages

CT systems offer several distinct advantages when compared to traditional methods for detecting internal composite damage.

First, micro CT provides precise information about the presence of internal damage. Previous studies on internal damage propagation relied on techniques such as Raman spectroscopy [?], scanning electron microscopy [?], acoustic emission [?], and ultrasound detection [?]. While these techniques are focused on providing information about the presence of damage, they are limited by poor resolution, long test times, surface-based observation, and mechanistic ambiguity [?]. They provide evidence of damage, but no clear picture of location and extent.

Second, micro CT is nondestructive. With other techniques, destructive techniques were required to directly observe internal damage mechanics. This included acid digestion of resin [?] and the combination of an optical or scanning electron microscope and the progressive removal of edge materials [?]. While successful in observing internal geometry and mechanics, these techniques were both time intensive and destructive. With such destructive techniques, additional damage may be introduced by the technique itself. Additionally, damage observation may only be performed once over the course of testing.

Furthermore, micro CT is relatively simple and fast. By combining traditional techniques, it is possible to combine techniques to provide an alternate to micro CT (with sufficient effort). For instance, to characterize damage modes in textile composites, it was shown that a multi-step method, including tensile tests with acoustic emission and strain measurement, C-scan and x-ray inspection, and optical/SEM examination of cross sections could produce accurate results [?]. However, this solution was both time consuming and complicated. The use of micro CT would have replaced C-scan information about damage extent and microscopy information about the micro-characterization of damage. Additionally, a tomography technique would
have avoided the need for cross sectioning the samples, allowing for the in situ testing of a single sample.

Lastly, because of the non-intrusive and non-destructive modeling CT systems offer, a single sample may be scanned in situ at various loading states, providing a comprehensive view of damage propagation in the material. Micro CT, coupled with in situ tensile test is one of the most novel opportunities that the technology affords. A single sample can be loaded to failure, with 3D observation at various load levels. This makes it possible to observe internal damage while under loading, and study how the same damage propagates at higher loads. The ability to do this in 3D, at micro-scale resolution is unmatched by previous methods.

3.4 Disadvantages

While certainly quicker than alternative methods, micro CT scans can still be relatively time intensive. Depending on the reconstruction quality desired, scan times can vary from a couple of minutes to several hours. Additionally, while the cost for industrial cabinet CT systems are cheaper than the use of a synchrotron facility, the price can still be a deterrent compared to cheaper alternatives, such as x-radiography \[?\].

The nature of reconstruction also results in many impediments to quality. Artifacts are an inherent feature in the reconstruction process, including the occurrence of ring and streaking artifacts, and beam hardening. Reconstruction software utilizes algorithms to remedy these features, but loss of quality is unavoidable.

Ring artifacts are caused by imperfect detector elements. They appear in CT images as a set of concentric rings. Their addition to the image gray scale values hinders the post-processing work that can be done. However, image processing techniques have been shown to greatly reduce the presence of ring artifacts in images in a matter of seconds \[?\], as seen in Figure ??.
measured steps for each viewing angle. The result of this is that the impact of image blemishes and defective detector pixels are blurred out in the final reconstruction [?].

Figure 3.4: Left: high resolution micro-CT images showing ring artifacts. Right: corresponding images after reduction of ring artifacts. [?]

Beam hardening is another artifact that impedes post processing. It is characterized by streaking around dense areas of material, as seen in Figure ?. Minimization of these artifacts has been accomplished by a dual-energy method in which two scans were combined. However, this technique also resulted in a higher presence of other types of artifacts [?].

Figure 3.5: An example of beam hardening between 2 aluminum rods, which are suspended in lower density Plexiglas. [?]

It can often be difficult to discriminate between resin and infused carbon fibers when inspecting a 3D CT reconstruction, due to the materials’ similar densities [?, ?]. This is especially the case with neighboring co-aligned bundles of fibers (tows). In the effort to better differentiate these materials, success has been found by coating woven
composites in gold, copper, and iodine contrasting agent. However, these methods come with the drawbacks that interface visualization is reduced, tow geometries deviate from uncoated tows, and void fractions increase [?]. Better results were seen when individual tows were coated in a contrasting agent and then woven. Gold and iodine agents were again successful, but mechanical properties are still affected by the coating of the tows in a foreign material [?].

3.5 Micro CT Studies of Composites

Schilling et al. provided a proof of concept for the use of micro CT in the damage characterization of composites, successfully identifying and measuring the presence of voids, delamination, and microcracks in a variety of composite materials [?]. This laid a groundwork for the abilities and limitations of micro CT in the field of composites. Previously, the majority of CT work had been focused on other materials and in the medial field.

Aroush et al. tensile loaded a 125 fiber quartz/epoxy ”model composite” in situ to determine a prediction for the critical fiber break cluster necessary for failure (N*). This was the first study to assess fiber fracture for a 3D polymer matrix composite using in situ SRCT [?]. Scott et al. expanded on this research with another in situ SRCT study of a larger carbon/epoxy volume of approximately 15,000 fibers. Their work was the first direct in situ measurement of fiber fracture accumulation ”for a high performance material under structurally relevant load conditions.” Because of the direct observation at relevant length scales afforded by SRCT, strong confidence was placed in the results [?]. As seen in Figure ?? the study provided a detailed view of the cascading damage mechanisms at play in the failure of composites, with fiber breaks, ply cracks, splits, and delaminations seen at several stages from unloaded to failure.
For microscale composite damage, micro CT provides a voxel size below the scale of damage. Due to its non-intrusive and non-destructive nature, the process is relatively uncomplicated and time manageable. This makes it a non-issue to add in situ tensile testing to the experimental setup, allowing for the full observation of damage to failure.

### 3.6 Dye Penetrants

It is often difficult to identify sections of damage in a CFRP when using x-ray or CT. Because of the small scales that laminate damage acts on, the gray levels of these areas are not significantly different than those of the surrounding material. A common solution that is used to enhance the contrast difference between material and damage is the application of dye penetrants. Dye penetrants are characterized by their high densities and show up much darker on x-ray image than carbon fibers or resin.
Successful damage identification has been performed with several types of dye penetrants. With a mixture of isopropyl alcohol, zinc iodide, photo solution and distilled water, it was shown microcracks of 0.5-1 μm could be seen in pixels as large as 20 μm, compared to a maximum pixel size of 4 μm otherwise [?]. Similar enhancements were found with the use of a diiodomethane x-ray opaque penetrant [?].

A dye penetrant is applied either by applying it to the outer surface of the laminate or by soaking the laminate in the dye. The dye is then pulled into areas of damage by capillary action. Because the dye travels to interior damage from the outside, it is dependent on inter-connected damage networks. Isolated voids or delaminations cannot thus be penetrated by an applied dye. Longer soak times ensure that the dye penetrates even the smallest areas of damage.

One major consideration that must be taken into account is the potential for a dye penetrant to drive or accelerate the creation of microcracks. Certain dyes can be chemically reactant with resin, weakening the material over time. Furrow et al. studied the effects of a zinc iodine dye penetrant on IM7/LaRC-IAX, IM7/LaRC-IAX2, and IM7/LaRC-8515 material systems. The samples were immersed in the dye for a soak time of 18 hours. The study found that even without applying a load to the material, thermal residual stresses were sufficient to drive microcracking in the presence of dye penetrant [?].

Similarly, Aboissiere studied a [0/90]s HTA/EH25 carbon fiber/resin laminate soaked in zinc iodine for 3.5 hours. This soak time did not drive the introduction of new microcracks, but upon tensile loading microcracks were introduced at lower strains and increased to a higher crack density saturation value, as compared to the untreated samples [?]. These results can be seen in Fig. ??.

Aboissiere found that better alternatives could be found in diiodomethane (DIM), diiodobutane (DIB), and silver nitrate. With a 45 min soak time, samples soaked in diiodomethane showed microcrack densities consistent with the untreated samples,
as seen in Fig. ???. The exception to this was for multiple soak times of 45 min each, in which higher crack densities were seen above 1.1% strain [?].

Figure 3.8: Crack density as a function of strain, with and without impregnation of DIM [?]

3.7 Examples of CT Results

This section provides examples of various materials which were successfully imaged through x-ray tomography. The results demonstrate that tomography had a great amount of versatility over a range of materials and densities.

A [0/90/0] carbon/epoxy composite section of dimensions 22 × 2 × 19 mm is
illustrated in Fig. ?? . The tomographic scan was performed with a 55 kV voltage and 32 W current with a voxel size of 13.6 µm. The scan involved 2889 projections over 360°.

Figure 3.9: CT image of a [0/90/0] CFRP

A sand composite is shown in Fig. ?? . The material is composed of a thick layer of sand/resin mixture surrounded by two thin layers of randomly oriented carbon fibers. The sample measures 13 × 13 × 6 mm, for a voxel size of 8.5 µm. The scan was performed at 91 kV and 40 W with 3016 projections over 360°.

A section of carbon nanotube (CNT)-infused resin is seen in Fig. ?? . A cutting plane has been inserted to reveal bubbles that were trapped in the resin during the curing process. This shows that micro CT is an asset in quickly investigating internal features, in this case to evaluate if the curing process produced a void-free final product. The x-ray settings were 65 kV and 29 W with 3016 projections over 360°. The sample measures 19 × 16 × 4 mm, for a voxel size of 12.4 µm.

An aluminum mesh embedded in a layer of resin is shown in Fig. ?? . The sample measures 5 × 5 × 1 mm, with a voxel size of 6.0 µm. The x-ray was set to 55 kV 27 W and 2877 projections over 360° were recorded. The complete sample is seen in Fig. ?? . In Fig. ?? the resin has been removed, by filtering out densities lower than that of the aluminum mesh. This allows for an unobstructed 3D view of the mesh, a
Figure 3.10: CT image of a carbon fiber/sand composite

Figure 3.11: CT image of a CNT infused resin
nondestructive feat which is not possible with other methods.

Fig. ?? shows a tomogram of a piece of corrugated cardboard. A 3D view is shown in Fig. ??, while a vertical slice has been taken in Fig. ??. The image was taken at 40 kV and 13 W, with 720 projections and 4 frames per projection. The sample measures $15 \times 10 \times 2\, \text{mm}$, for a voxel size of $21\, \mu\text{m}$. The image shows that micro CT is successful on materials of lower density as well, at lower voltages. The process also showed that it is a simple affair to inspect internal geometry by taking slices of a CT image file in the principal directions.
Chapter 4

Process

The main goals of this experimental work were:

- Determine a diffuse damage parameter as a function of applied shear stress
- Obtain crack density as a function of applied strain for cross-ply samples with various levels of diffuse predamage. This data would be used to calculate the strain energy release rate as a function of crack density.

To achieve these goals, a \([0/90]_s\) laminate was configured, cut into \([\pm 45]_s\) samples, tensile loaded in shear orientation to various levels of diffuse damage. This is referred to as Mode III testing, in reference to the diffuse damage mode. After this, the samples were cut again into smaller \([0/90]_s\) samples, and prepared for tensile testing in order to record crack density by x-ray. This testing phase is referred to as Mode II testing, in reference to the transverse microcracking mode of the 90° plies.

The laminate edge was inspected by optical microscopy and x-ray, a dye penetrant was tested for use in crack identification, and a micro CT scan was performed on a sample containing microcracks. The material preparation and shear testing were performed in conjunction with the research of Dr. Hedi Nouri. The microcrack tensile testing of the small \([0/90]_s\) samples will be carried out in future stages of research.
Mode III testing was performed on an Instron 5882 100 kN load frame (Fig. ??), x-rays and tomograms were performed with an X-Tek XT H 225 cone-beam tomograph (Fig. ??), and Mode II samples were designed for a Deben 5 kN tensile stage (Fig. ??).

![Testing equipment](image)

Figure 4.1: Testing equipment

### 4.1 Materials

Laminates plates of T700/M21 carbon fiber pre-preg were laid up in a \([0/90]_s\) configuration with dimensions 300 × 300 × 1 mm and cured in compression molding at a manufacturer recommended single dwell temperature of 180° C. This plate can be seen as the outer square in Fig. ??.

### 4.2 Shear Tensile Testing (Mode III)

The first stage of testing was to subject the material to an approximate shear loading, and to study the evolution of diffuse damage. Each laminate was cut into four 50 × 220 mm rectangles (as seen in Fig. ??) using a numerical control machine. These specimens were rotated at 45° with respect to the original plate, giving them a \([±45]_s\) configuration. Subsequently, these specimens were subjected to an approximate shear loading state when loaded in tension. Aluminum tabs of dimension 50 × 50 mm were adhered to both faces on the specimen ends, shown in blue on the \([±45]_s\) specimens.
Static and quasi-static tensile tests were performed on the \([\pm 45]_s\) specimens. This testing configuration is shown in Fig. ??.

Digital image correlation was used to determine the evolution of strain field at the surface during mechanical testing. This was used to obtain levels of shear damage and microcracking versus the applied measured stress or strain.

First one panel was tested in tensile loading to failure, to establish the ultimate load that the laminate could sustain. Next, six samples were tested in cyclic loading with 250 N increments until failure. The initial Young’s modulus \(E_0\) was computed from the stress-strain slope on the first 250 N loading. Subsequent values for \(E_n\) were
computed from the slope of the unloading portion of the stress-strain plot.

Using the change in stiffness, a diffuse damage indicator was calculated for each load level using the equation:

\[ d = 1 - \frac{E_n}{E_0} \]  \hspace{1cm} (4.1)

Thus, the value of \( d \) is an indication of the reduction of shear stiffness. The value for \( d \) was averaged at each load level from 250 to 5250 N over the five panels, omitting one panel with outlying data. Using this resulting shear damage/stress curve, six panels were damaged in shear loading to levels of 0, 0.05, 0.10, 0.15, 0.20, and 0.25. Digital Image Correlation (DIC) was performed on these panels during the tensile loading, using a camera and Vic-2D software. This allowed for the observation of the deformation field of the loaded sample. From the deformation field it is possible to determine at which diffuse damage level cracks start to be introduced in the material. These observations can be validated by x-ray in future work, in the Mode II testing phase.
4.3 Microcracking Tensile Testing (Mode II)

Two pre-damaged panels of each damage level were cut by water jet to produce three tensile testing samples each, resulting in six tensile samples for each shear damage level (see Fig. ??). These samples had an orientation of [0/90], in order to emphasize the transverse cracking damage mode in tensile loading. Their geometry is shown in Fig. ?? . This sample geometry is based on required dimension of the tensile stage. The length of the middle section was extended to the maximum allowable length in which the failure strain could be reached before the tensile stage attained its hardware strain limit.

Pin holes were included to ensure that the sample did not slip during loading. Tabs made of T700/M21, of dimension $12 \times 20 \times 1$ mm, were bonded to both ends of the sample to enable distributed load transfer from the tensile stage to the sample. The tabs distributed the application of stress along the whole width of the sample to avoid stress concentration around the two holding pins. The sample width was reduced from an initial width of 20 mm on the outside, to an intermediate width of 12 mm, and finally to 4 mm in the middle. This middle section of 4 mm width and 12 mm length was designed to be the area where microcracks nucleate.
4.3.1 Edge Examination

A microscopy inspection of the edge of a $10 \times 10 \times 1$ mm $[0/90]_s$ T700/M21 sample was conducted in order to check for sources of damage from the cutting process, to obtain a surface image of the undamaged material, and to validate the results of an x-ray image from the same viewpoint.

First an x-ray inspection was performed, allowing the observation of features through the entire width. Settings of 45 kV and 32 W were used for the x-ray images. Then optical microscopy was performed on the same sample edge. The material was polished in order to eliminate deformities and make the fibers and matrix materials visually distinct. This was accomplished on a Struers TegraPol-35 with an incremental abrasion sequence of 320, 500, and 1000 grit sandpaper, followed by the polishing application of $\text{Al}_2\text{O}_3$ 15, 5, 0.3, and 0.04 µm abrasive solutions, as seen in Fig. ?? . Magnification of $5 \times$ was used to obtain individual images which were then stitched together to create a high magnification image of the entire edge.

![Grinding and Polishing Sequence](image)

**Grinding**
1. 320 grit sandpaper
2. 500 grit sandpaper
3. 100 grit sandpaper

**Polishing**
4. $\text{Al}_2\text{O}_3$ 15 µm
5. $\text{Al}_2\text{O}_3$ 5 µm
6. $\text{Al}_2\text{O}_3$ 0.3 µm
7. $\text{Al}_2\text{O}_3$ 0.04 µm

Figure 4.5: Grinding and polishing sequence for laminate edge examination
4.3.2 Dye Penetrants

One method for identifying microcracks in CFRPs is to use a dye penetrant in conjunction with x-ray studies. To test the feasibility of using dye penetrant, diiodomethane (DIM) was applied to a tensile sample containing multiple microcracks from Mode II loading, as well as larger cracks and areas of delamination. The DIM was applied with a cotton swab to the edges of the sample in the areas containing transverse microcracks. This was continued for 5 minutes, when it appeared that no more dye was being absorbed by the sample. X-ray images were then taken from the front and side, at settings of 40 kV and 18 W. These same images were taken with a sample that had not been treated with dye penetrant, for comparison. The images were inspected for the appearance of transverse microcracks.

The same DIM application procedure was then repeated. Following this, x-ray images were recorded at each minute after application, in order to test time dependence of the dye effectiveness. If there was too much variation in the image gray levels, a longer soak would be required in order for the dye to penetrate all crack sizes and remain absorbed. Constant gray levels are a requirement when working with micro CT, because the tomographic process acts over the range of tens of minutes.

4.3.3 Computerized Tomography

Another method for microcrack identification is the use of micro CT. Tomography is useful in the imaging of micro-level damage, even without the use of dye penetrants. The same damaged sample from Section ?? was used for a tomographic inspection in an unloaded state. Tomography was performed with x-ray settings of 40 kV and 18 W. 360 projections were captured over 360°, with 4 frames averaged for each projection. The voxel size for the resulting tomogram was $12.1 \times 12.1 \times 12.1 \, \mu\text{m}$. After reconstruction, post-processing was performed on the tomogram with Avizo Fire 7.0.1. This involved smoothing the image to remove small-scale noise with a
non-local means filter, segmenting the data with a tophat quantification in order to isolate microcracks, and then the removal of small damage volumes, which were not indicative of microcracking. This post-processing workflow can be seen in Fig. ??.

![Figure 4.6: Avizo Fire workflow for tomogram post-processing](image)

4.3.4 Future Work

In future stages, the evolution of microcracking in these samples will be observed using Mode II tensile testing coupled with x-ray tomography (see Fig. ??). This will be performed with a Deben 5 kN tensile stage, which is specifically designed to be mounted inside the CT cabinet. The stage will be mounted on the CT manipulator, allowing for 360° movement about its vertical center of rotation. The tensile stage has a maximum stress of 5 kN and a maximum displacement of 10 mm. Tomography will be performed using an X-Tek XT H 225 cabinet system with a 225 kV X-ray tube and a 1900 x 1500 pixel 14-bit detector. The goal of this section is to obtain a plot of crack density versus strain for samples of each diffuse damage level. From this, the microcracking fracture toughness, $G_{mc}$, could be calculated for each.
Chapter 5

Results and Discussion

5.1 Shear Tensile Testing (Mode III)

The stress/strain curves from Mode III testing obtained for five samples cut from five different plates are presented in Fig. ???. The figure provides evidence that the results are repeatable and reproducible. Fig. ?? shows the damage evolution as a function of stress for the five tests. In Fig. ??, damage evolution is averaged over the five tests and shown as a function of stress. The averaged curve shows that the mean level of damage to rupture is $d_{rup} = 0.2754$, at a stress of $\sigma_{rup} = 105 MPa$.

The observed deformation fields in the vertical direction, as obtained from DIC, is seen in Fig. ???. The deformation fields are presented for different damage levels. These maps show, that starting at $d = 0.0445$, the deformation field becomes heterogeneous and from $d = 0.0875$ we see that cracks are introduced between fibers in the upper layer, or $0^\circ$ ply. This is an expected result. Since the $0^\circ$ plies are bounded only on one side, they are expected to host the first signs of intralaminar damage. On the map corresponding to $d = 0.01225$ we see the appearance of cracks perpendicular to the upper layer fibers, indicating microcracking in the central $90^\circ$ layers. The last map shows the state of the deformation field just before the failure of the sample,
Figure 5.1: Tensile cyclic tests for T700/M21 $[\pm 45]_s$ samples cut from different plates.

Figure 5.2: Damage evolution versus stress for tensile cyclic tests of T700/M21 $[\pm 45]_s$ specimens.

corresponding to a damage level of $d = 0.2727$.

The DIC results provide several results that can be verified with x-ray imaging in the Mode II experimental campaign. From these image maps, we would expect to initially see longitudinal cracks in the 0° plies for the samples of damage levels 0.10, 0.15, 0.20, and 0.25. Transverse cracks are expected to be present in the 90° plies for
Figure 5.3: Strain field evolution during tensile static test for different damage levels.

damage levels of 0.15, 0.20, and 0.25.

5.2 Microscopy Edge Examination

An edge inspection study was performed on the tomography samples to determine the state of the material when undamaged. Results from the x-ray study can be seen in Fig. ??, with a detailed view in Fig. ???. Corresponding results from the microscopy study can be seen in Fig. ??, with a detailed view in Fig. ???.

These results show a strong correlation between the micro CT and microscopy images. Because microscopy shows surface features and tomography shows internal features, the similarity of the images suggests that geometric patterns are largely constant throughout the width. The line patterns are observed within the 90° plies in both images are presumed to be the boundaries of fiber bundles, since they are
also features with constant geometry along their length and are not visible in the $0^\circ$ plies from this view. These results are also consistent with previous inspections of T700/M21 laminates [?].

![Micro CT and Optical microscopy images](image)

Figure 5.4: Edge inspection of a $[0/90]_s$ CFRP laminate

![Detailed view of Micro CT and Optical microscopy images](image)

Figure 5.5: Detailed view of the edge inspection of a $[0/90]_s$ CFRP laminate

With the introduction of microcracks, we would expect to see additional horizontal lines spanning the thickness of the transverse plies as fibers in this direction are pulled
apart. However, it was not possible to observe this on our microcracking samples. Acceptable microscopy results require polishing the sample’s outer edge, and this was not possible with the experimental samples because their edges were not straight.
5.3 Dye Penetrant Contrast Enhancement

The results of the dye penetrant application are shown in Figs. ?? and ??.

The front and side of the plain sample are shown in Figs. ?? and ??, while the front and side view of the contrast enhanced sample are illustrated in Figs. ?? and ??.

The improvement offered by the DIM application is evident, even from such a short application time. In the front view, multiple black transverse microcracks are visible in the DIM sample, whereas only a few faint white transverse microcracks are visible in the plain sample after a rigorous inspection. The same is true for the four longitudinal cracks readily visible in the DIM sample. From both samples it can be clearly seen that the macro-scale damage in the top section, where the two middle plies became separated from the outer plies and were pulled out.

From the side view, microcracking is more visible in the plain sample. Microcracks are identified as the white area in between fiber bundles which runs horizontally and spans the thickness of the 90° plies. However, visibility is again greatly increased in the DIM-enhanced samples. Microcracks are easily identified when highlighted by DIM, including some microcracks that were not visible in the plain sample. Even with a short application time, it is clear that DIM is a proficient tool for identifying damage in the T700/M21 material system.

Changes in the dye-enhanced image over time are shown in Fig. ??.

The first image is taken immediately after application of DIM, and each following image is taken at one minute increments afterwards. Cracking damage is evident at all stages, but the overall gray levels change significantly over time. This can be attributed to evaporation of the dye penetrant, both from the material surface and out of the interior damage regions.

Short term time dependence such as this is not desirable, especially when running tomographic scans. For micro CT studies this gives two options: wait until gray levels have stabilized before initiating a tomographic scan, or soak the sample in the
Figure 5.6: Front view of [0/90]₀ tensile sample with and without DIM application

Figure 5.7: Side view of [0/90]₀ tensile sample with and without DIM application

dye penetrant for a longer period before imaging. A longer soak time gives sufficient
time for even the smallest microcracks to be penetrated by the dye, ensuring that
the dye will likewise remain in those cracks for a similar amount of time. In practice, a combination of these options is likely the best strategy. Consistent results are more likely by following the 45 minute soak time recommended by Aboissiere [?], and ensuring consistent gray levels before beginning a tomographic scan.

Figure 5.8: X-ray images of the [0/90]_s tensile sample at specified times after application
5.4 Computerized Tomography

The obtained tomogram from the microcrack sample is shown in Fig. ???. The data contains all the largest areas of damage found during post processing. From the results, it is evident that damage is primarily found in the form of transverse microcracks in the central 90° plies. A large number of microcracks span the width, but there are also a number of smaller cracks that are localized on the right side of the sample. This can be attributed to the lack of proper alignment of the sample in the loading clamps. In this regard, we would expect to see more symmetric results from Mode II testing, because the samples will then be aligned by holding pins in the tensile stage.

The advantage of tomographic data over 2D x-ray imaging is apparent. From the tomogram, it can be easily seen that microcracks span the thickness of an entire ply. Through 3D manipulation, it is also possible to see which ply a specific microcrack is located in. With 2D x-rays, this is possible, but only through a careful comparison of the front and side images of a sample. Damage connectivity can also be seen in a tomogram. In this case, there is mostly connectivity from microcracks to the outer surface, but there are also small areas of delamination that have been nucleated at the crack interface.

Although results were successfully obtained, the data was difficult to segment, resulting either in noisy data or in data in which smaller microcracks are omitted. A key point to note here is that tomography was performed while the sample was unloaded. It is very likely that if tomography were performed in situ with tensile loading, damage in the resulting tomogram would be clearer. The reason for this is that when the sample is under loading, the microcracks are pulled apart, creating a larger area to appear on x-rays.
Figure 5.9: Tomogram of laminate sample containing multiple transverse microcracks
Chapter 6

Conclusion

6.1 Summary

Mode III testing of a $[\pm 45]_s$ T700/M21 laminate plate was performed. By cyclic tensile loading at 250 N increments, reduction of shear stiffness was calculated for these specified loads. The reduction of stiffness was used to determine a damage parameter that was indicative of diffuse damage and some microcracks in the $+45^\circ$ and $-45^\circ$ plies. This damage parameter ranged in size from 0 to 0.2754. By interpolating between points, it was thus possible to obtain the necessary load required to reach any specified diffuse damage level.

Six were subjected to six different damage levels: 0, 0.05, 0.10, 0.15, 0.20, and 0.25. Each of these plates was then cut into six $[0/90]_s$ samples to be used in Mode II testing.

An edge study of a $[0/90]_s$ T700/M21 coupon was performed with optical microscopy and x-ray. After polishing the material edge and examining under microscope a line pattern within the 90° plies was confirmed to be the boundaries of fiber bundles. This was verified by studying the sample under x-ray and observing that the pattern was consistent along the length of the 90° ply. This agreement of results
showed that x-ray is an appropriate tool to observe the internal configuration of fiber bundles. The study also provided an example of T700/M21 x-rays in an undamaged state.

A feasibility study was performed on a spare Mode III testing sample. After introducing microcracks in a Mode III tensile test, the sample was observed by x-ray from the front and edge. DIM was applied to the material edge manually for 5 minutes, and the same observations were repeated. The results showed that multiple microcracks were clearly visible in the DIM-enhanced images, while only a few microcracks could be found after a rigorous search in the untreated images. This confirmed the utility of DIM as an x-ray contrast agent. However, front x-ray images of a DIM-enhanced sample showed that the gray levels produced in these images were not constant in the immediate time after application. This showed that a soak time of 45 minutes is a better option in order to saturate the smallest microcracks and ensure a slower diffusion for the penetrant.

A tomographic scan was performed on the same damaged sample, without a dye penetrant and while unloaded. This resulted in a 3D view of the damage network present in the sample after a mode II test. The results showed a number of transverse microcracks in the 90° plies, which spanned the thickness of the ply. Cracking did not always span the width of the sample, a result that can be attributed to improper alignment of the sample in the UTM. The data proved to be difficult to segment in post-processing, which is speculated to be a result of the sample’s unloaded state. It is presumed that post-processing would be easier, and would yield clearer results, if tomography were performed in situ with Mode II tensile testing.
6.2 Future Research Work

Mode II tensile testing is the main focus for future studies. The present thesis provides a solid foundation for the techniques and tools that would be used for this study. In the next round of testing, six tensile samples will be used from each level of diffuse damage: 0, 0.05, 0.10, 0.15, 0.20, and 0.25. These samples will be tensile loaded to failure, with the crack density computed at specified load levels.

It has been shown that microcracks will likely be difficult to identify in a plain sample from 2D x-rays. And once a tensile test has started, it is not possible to apply a dye penetrant to the sample inside the tensile stage. This leaves 2 main strategies:

- The six tensile samples can be loaded to a range of stresses below the failure stress of the material, each to a different level. After this, the sample would be unloaded, soaked in dye penetrant, and x-rayed. Microcrack density would be readily available from this image, giving a data point for crack density and its associated stress/strain. If this process was repeated for several load levels, a crack density/strain curve could be obtained from this set of discrete data points.

- Tomography can be performed on the samples in situ with tensile testing. This allows for the calculation of crack density at several points during the load profile of each tensile sample. This is possible because microcracks are more readily identifiable when using micro CT versus 2D x-ray images, as proven with the successful micro CT results of this work. This was accomplished on an unloaded sample without the use of a dye penetrant. A tomography study during tensile testing would likely provide clearer results still.

The result of this testing campaign will be a set of crack density/strain curves, one for each level of diffuse stress. From this, the strain energy to initiate microcracks, $G_c$ could be computed as a function of crack density for each curve. With these results,
a clear correlation can be drawn from the amount of fiber/matrix debonding in a material to the resistance of that material to microcracking damage.
APPENDICES
Appendix A

XT H 225 MicroCT Guide

A.1 General Notes

- Open and close door carefully, slamming it may cause damage to the turbofan.

- X-rays will not be generated until the door is closed. When the door is opened, the x-rays will switch off and disperse immediately. Even so, be sure to turn the x-rays off before opening, just to be safe.

- Use supervisor mode for operation, to give more controls than in operator mode. The password is "supervisor"

A.2 X-ray Controller

- Turn the x-rays on and off with (3). Their status is shown in the x-ray status window (5). Make sure x-rays are off before opening the cabinet door.

- Penetration - kV (1)
  - With higher kV, more x-rays penetrate the object of interest. Denser objects require higher penetration.
Figure A.1: X-ray controller

- Intensity - Power (W) or Current (uA) (2)
  - With higher intensity, the image becomes brighter and more saturated.

- The advantage of power controls over current controls is that the relative image brightness remains the same when kV is changed.

- Optimal image contrast is obtained by using the lowest kV that produce the desired level of image quality. Thus two conditions must be met
  - Penetration (kV) must be high enough to see details
  - Image must not be dark, saturated, or washed out

- In general, start at low kV and increase until details are acceptable. Adjust uA to obtain proper contrast.

- Focus adjustment (6) is used for fine focus control. Once you have acquired the right penetration and intensity, adjust the focus until details are at optimal sharpness. If this is not enough to obtain a focused image, x- and y-shift may need to be adjusted.

- The information window (7) shows useful information about the filament and vacuum. The actual filament value should be around 30-40 when x-rays are on. If the value is close to zero and there is no image produced, the filament
probably needs to be replaced. Vacuum value should be as close to 0 as possible, values under 10 are acceptable. Hours on gives the number of hours that the present filament has been used for. Normal lifetime is around 120 hours.

![Information window](image)

Figure A.2: Information window

- The properties window (8) is used mainly for adjusting filament demand and x- and y-shifts (for focus calibration).

### A.2.1 Auto-Conditioning

- Tomograph must be auto-conditioned at least once every couple of days, but preferably before each extended and/or high kV CT scans.
  - If not conditioned, the x-rays will likely shut off during a test with high kV demand
- Set the voltage to 225kV and press the auto condition button (4). Press again to stop once the system stabilizes at 225kV.
A.2.2 Filament Change and Calibration

Filament Change

- When optimised, the expected filament life is 200 hours.
- An indication of filament failure is when switching on x-rays, the kV ramps up but no mA/W registers and no image is produced.

1. Switch off the SR3 power button to shut down the pumps and turn the interlock key to the off position (Fig. ??).

![SR3 power button](image)

Figure A.3: SR3 power button

2. When the pumps have run down, vent the gun vacuum chamber by opening the vent valve (Fig. ??). Once the pressure has equalised, release clip A and carefully open the x-ray gun (Fig. ??).

3. Remove the focus cup with an A/F Key. Using a 1.5mm A/F key, loosen the two Grub Screws holding the focus cup insert (Fig. ??). Remove the focus cup insert to expose the filament.

4. To remove the old filament, grip both filament pins with curved pliers and pull.

5. Inspect the focus cup insert and clean with lint free wipes and acetone. If absolutely necessary use a small amount of metal polish to clean the focus cup and then thoroughly remove all polish residues using acetone.
6. Using the curved pliers, insert the new filament. Do not touch any part of the tungsten wire loop. Ensure the filament is pressed fully into its locating holes and is square.

7. Replace the focus cup insert and tighten the insert screws.

8. Check alignment. Alignment of the filament is critical to system performance.

**Alignment**

- Using Fig. ??, check both the vertical and central location of the filament.
• The filament hairpin loop tip should be centrally located in the focus insert aperture (View A) and just be visible when looking down the plane of the insert recess (View B).

![Figure A.6: Filament alignment](image)

1. Centralisation of the filament: Loosen the filament clamp screws $\frac{1}{4}$ turn. Insert a small screwdriver into the gap between the focus cup module and the filament holder (see Fig. ??) and lever the filament holder to centralise the filament (Fig. ??, View A). Re-tighten the clamp screws.

2. Filament height adjustment: Set filament height (Fig. ??, View B) by removing the focus cup insert and adjusting the 3 height screws (see Fig. ??) by equal amounts until filament tip is just visible. Re-tighten the insert screws.

3. Clean the focus cup using clean lint free wipes to remove all traces of grease/poolish.

4. Replace the focus cup insert and replace in the gun. Close the gun and fasten the clip. Close the main access door (remember to close the vacuum vent valve on the gun first). Switch on interlock key and turn on SR Controller. The vacuum will have to be re-established before X-rays can be produced.

5. Once the vacuum is established and the interlocks are closed then X-rays can be produced. However, the filament must first be conditioned.
Calibration

- x- and y- shifts need to be calibrated each time a new filament is installed.

- Place an object with small details into view of the X-ray source using the manipulator. Set a very high magnification so that the fine detail in the object can be seen.

- Calibration settings can be found in the properties menu (8). Click on X-Ray Controller → Focus

- After installing a new filament, reset the filament lifetime (2).

Switch x-rays on to a low level in which the image is not whited out. Slide the filament demand slider (1) to the left and the image will black out. Slide it slowly to the right and the image will brighten. Move the slider to the point where the image is the brightest and doesn’t become any brighter. Move the slider one click more to the right and click Save.

1. Set the x-ray to 50kV and a suitable brightness for A/W

2. Press the modulation button (looks like a sine wave). The image will move up/down and side to side.
3. Adjust the x and y sliders until the image no longer moves back and forth. It should just go in and out of focus instead, and be at its brightest.

4. Switch off modulation.

5. Press the X-Shift and Y-Shift Save button. This will save the values for 50kV.

6. Repeat the process for 100kV and 225kV

7. Press the Interpolate button. The software will interpolate the calibration values whenever a voltage is used that falls in between these 3 calibrated voltages.

### A.3 Manipulator Controls

- The Stop button (1) halts any manipulator movements in progress. Always look inside the cabinet to keep track of where the manipulator is moving the stage and sample. If your sample is going to collide with the x-ray gun, you need to
be ready to press either this button or the big red physical button on the x-ray cabinet.

- The home button (2): Whenever the software is restarted, a homing operation must be performed. This is because the software has no information about the physical location of the sample platform. The homing operation moves the manipulator to its limits in order to determine position. After the homing operation, position commands may be given again.
  
  - Important: Remove any samples from the manipulator stage before homing. If a sample is not removed, it will slam into the x-ray gun and/or fall off the stage.

- The load button (4) moves the stage to an easily accessible loading position. Again, make sure to remove the sample before pressing this button, to prevent collisions.

- The speed controls (5) dictate how fast the manipulator moves. When moving
close to the x-ray source, a slow speed is recommended.

A.3.1 Controls Window

The Controls menu is the most used operations window:

- The absolute column (9) is the current position.
- The relative column (12) is the current position, relative to the point where the zero buttons (13) were pressed.
- The lock buttons (14) prevent movement on the specified axis, until it is unlocked again. Usually, you’ll want to lock the tilt axis, as well as the X, Y, and Mag axes once your sample is in the desired position.
- The demand column (11) shows where the manipulator will move when you press the Move button (15). Always double check what values are displayed here before you press Move, otherwise catastrophic collisions might once again occur. If you want the position on a certain axis to remain the same after
the move, press the arrow (10) next to that value so that the desired position matches the current position. The demand controls are extremely useful for rotating a sample in increments such as $45^\circ$, $90^\circ$, $180^\circ$. Transfer the absolute value to the demand column, and then add or subtract the change in angle. Type this new value into the demand rotate box. Remember, if you want purely rotation, make sure that all the other desired values match the absolute values.

- To save a manipulator position for later, press the add button (5). To return to that position in the future, just double click the position name in the list menu (4).

![Saved positions](image.png)

Figure A.11: Saved positions

- Besides the controls window, the physical joystick controls are used for general, non-precise movements.

### A.4 Image Processing

- The image processing toolbar can be seen in Fig. ???. Press the green button (1) for the live x-ray image. The arrow next to it controls the number of frames that
are averaged, used to create a rolling average. When the sample is moved, live image is automatically enabled. When the sample is stationary, rolling average is automatically restored.

- Press the red button (2) to capture the live image and display it. You may then save this image with the save button (5).

- To capture a higher quality image, use the average image button (3). The number of frames used is specified in the arrow menu. More frames results in higher quality but longer capture time. You may then save this image with the save button (5).

- Open a previously saved image (4)

Figure A.12: Image toolbar
A.4.1 Image Information

When the mouse tracks across the X-ray image in the main image window, the panel seen in Fig. ?? displays the X and Y coordinates to the image pixel under the mouse (relative to the top left hand corner of the image) and the grey level of the pixel. Grey levels should be in the range of 6,000–60,000.

Also keep an eye on the histogram on the right side (Fig. ??). Ideally, the curve should lie towards the center of the spectrum. Pixels on the right edge are oversaturated and on the left edge are completely black. Double click on the red line to add drag points. Double click on a point to remove it. This curve is typically manipulated so that it is steepest over the gray values where most of the pixels are found. This increases the contrast for pixels in this range.

Figure A.13: Pixel information panel

Figure A.14: Histogram
A.4.2 Background Subtraction

Use this tool to remove any background noise from an x-ray image

1. Click the "BG Subtraction" button, above the histogram in the top right section of the window

2. Move the sample out of the way so only the background remains.

3. Either click the red button or the image averaging button to capture a still image of the background

4. Click Set (1) to set this image as the background (See Fig. ??)

5. Click Apply (4) to apply the background subtraction processing

6. Typically "Image minus background" is selected (5)

7. Click Remove (3) to remove the effects of image processing and remove the current background image

Figure A.15: Background subtraction tool
A.5 CT Acquisition

- Object must remain in view at all angles. Rotate the object to make sure none of the region of interest passes out of view.

- Object center of rotation must be as close to the stage’s center of rotation as possible. When you enter the Centre of Rotation tab, the manipulator will automatically move to x=0.

- The sample must be penetrated at all angles. It also cannot be saturated at any angles. Check to see that the thinnest and thickest views are properly penetrated, with gray levels between 6,000 and 60,000.

- To start a CT inspection, click on the CT tab

![Figure A.16: CT Tab](image)

A.5.1 Sample Setup

- Choose the number of projections to be used. More projections result in higher detail, but take more time. Click optimize to have the software choose the number needed for optimal detail. This will likely take several hours. It depends on the amount of detail in the object of interest, but usually any 180 projections and up will produce acceptable tomograms.

- The frames per projection are the number of images that will be averaged together to produce each projection. Typically 4 images is a sufficient value. Remember that each increase in this value will double the overall scan time.
A.5.2 Reference Images

- When you get to this tab, move the sample out of the way of the x-ray source. It will automatically return to the same position when you move on to Center of Rotation.

- Select a number of frames above the recommended minimum to obtain the black and white reference levels. If the black or white image is too old, check the box and click Acquire. Once you have done this, the images can be reused if another CT is run with the same kV and W/uA settings. This is saved for 3 hours.
A.5.3 Center of Rotation

- The CoR needs to be determined precisely for a high quality image. Leave the "Calculate Automatically" box checked and click "Acquire". The line represents the cross sectional slice that will be used to calculate the CoR. Move it to a reasonable place on the sample.

Figure A.19: Center of rotation acquisition

A.5.4 Reconstruction Setup

- The settings in section (1) (See Fig ??) are used to reconstruct the volume, if the "Reconstruct" option on the Acquisition tab is selected. If automatic reconstruction will not be used, then there is nothing to change on this tab.

  - A good practice is to run one CT scan without automatic reconstruction. After the scan is done, reconstruct it manually using CT Pro on the other computer. Take note of the beam hardening and noise reduction settings. Then if a similar scan is run in the future, the proper settings may be selected on this screen to do an automatic reconstruction. The auto process is less of a hassle, once the proper reconstruction properties are known.

- Regardless if auto reconstruction will be selected or not, press the Acquire button in the Volume of Interest section (2). Then select the volume of interest around your sample, giving some free space along the sides. The volume inside these borders will be the volume that is reconstructed.
Section (3) shows information such as voxel size and required disk space.

Figure A.20: Reconstruction settings

A.5.5 Acquisition

- Name your data, choose the proper filter if one is used, and select Reconstruct if auto reconstruction is desired

- Ring artifacts can be minimized, but the process adds to the scan time. If time is a limiting factor, uncheck the box and see if there are any ring artifacts in the final product.

Figure A.21: Acquisition window
A.6 Reconstruction

If auto reconstruction hasn’t been selected, the volume will have to be reconstructed with the CT Pro software on the reconstruction computer. Start by opening the .xtekct file.

A.6.1 Image

• Nothing needs to be done on this screen, but information is given in the Result box about the quality of the projection set.

• Click on the Compare button at the bottom to see a frame by frame view of the first few projections around the starting frame.

Figure A.22: Image window
A.6.2 Center of Rotation

- Move the red line up and down to select a suitable slice to compute center of rotation from.

- Change to Dual selection to compute the center of rotation from 2 slices.

- If the calculation fails, uncheck the Fast option in the Accuracy of Determination section.

- If the calculation fails again, change from Standard accuracy to Fine or Ultimate.

- The manual process is a good option if the above steps fail. It’s a visually-led process. Change the distance until the cross section shown in the window is aligned and clear.

Figure A.23: Center of rotation window
A.6.3 Setup

- To change settings on this screen, select the Reconstruct All option next to the setting and click Start. Once the process has finished, click on the option buttons to determine which results in the clearest, noise free image.

- Interpolate is almost always left on "Interpolate".

- Find the best option for Beam Hardening (1-6).

- Find the best option for Noise Reduction (1-6).

Figure A.24: Setup window
A.6.4 Calibration

For normal use, nothing needs to be done on this screen.

A.6.5 Volume

- Adjust the volume of interest in the Selection section if necessary
- Volume space and voxel size are noted
- Change the output file name if desired, and click Start to construct the 3D volume, which is written as a .vgi file, to be opened by VGStudio.

Figure A.25: Volume window
A.7 Post-Reconstruction Processing

Reconstruction post-processing is discussed for VGStudio MAX software. Other options exist for this work, such as Avizo Fire.

- The reconstruction process returns a .vgi file. Save to a .vgl file when you open the file in VG Studio, so that your post-reconstruction manipulations are saved.

- The first thing to do is click Object→Calibrate object. You can let the program choose automatically or indicate manually what parts of the image are background and what are material. The software will segment the gray scale and hide the background noise.

- Next create an aligned clipping box from the button on the top toolbar. This is useful to crop out artifacts on the edges of the volume, or areas that are not relevant to the study. The limits of the clipping box can be changed by dragging the yellow handles on the 3D view. An easier way is to change their numerical values by looking at the windows on the right side. Under Clipping, select the Aligned clipping box tab and change the boundary values there.
Appendix B

Deben 5kN Tensile Stage Guide

B.1 Tensile Testing

B.1.1 Mounting a Sample

- For tensile testing, samples should be mounted to ensure that the jaws can be expanded far enough to complete the test.

- Standard stages have a travel of approximately 10 mm, limited at each end of travel by software limits.
  - For elastic samples this will mean that the jaws should be fully closed, with a 10 mm gap, before starting the test otherwise the limit may be reached before completion.
  - For brittle samples the jaws will not need to be closed fully. The best position will be in the center of the allowable range. Calculate the breaking strain of the given material to determine if the jaw extension will be sufficient.

- The geometry for a tensile sample is shown in Fig. ?? The most important dimensions in this diagram are the pin hole diameters and spacing. The geometry
in the necked section may be modified. The length may also be increased to values larger than 34 mm. This, however, decreases the amount that the lower jaw may be extended. Lengths smaller than 34 mm can also be utilized.

Figure B.1: Tensile sample geometry

Procedure

- Screw the brass sample support screws into the system and undo the top two M6 caphead screws, shown in Figure ??, Remove the top cap from the system.

- Undo the four M3x50 cover fixing screws, shown in Figure ?? and remove the top cover and tube assembly.

- Remove any sample if previously fitted.

- Verify that mode is Tensile

- Using the software, offset the force to 0N with Tools → Set Loadcell zero offset (Ctrl + L).
Figure B.2: Removal of M6 caphead screws

Figure B.3: Removal of top cover
• Fit the sample using the sample support screws to set the correct jaw spacing in order to accommodate the sample. Observe the force reading during this procedure (especially with low force loadcells) to ensure that the force does not go overrange or underrange.

• Measure the gap between the jaws (sample gauge length) and use the ”Goto mm (absolute)” function to move the jaws to a distance that is just under the sample gauge length. For example, for a gauge length of 12mm, move to 11.5mm.

![Figure B.4: Gauge length measurement](image)

*Note: The nominal microtest travel is 10mm to 20mm. In order to accommodate variations in sample length it is recommended that the minimum sample gauge length is 11mm.*

• Replace the top cover. Ensure the location arrows are aligned, as shown in Figure ???. Do not tighten the four M5x50 fixing screws yet.

• Tighten the top two M6 caphead screws (Fig. ??). When tightening the screws, the transition between loose and tight should be almost immediate. If the screws begin to tighten slowly, then check for any trapped components before continuing. The system alarm will begin to sound at this step because the load is now underrange. This is normal.
Using the "Goto" function on the software, select Goto 0N. The extension will increase slowly.

When the force reaches 0N, the stage will stop. Now tighten the four M3 screws. Monitor the force reading to ensure that undue force is not being applied to the sample.

Click Displacement → Set Current Position, and set the current position equal to the gauge length measured before.

Click "Clear data" in the software.

Remove the sample support screws.

The system is now ready to run a test.
Table B.1: Maximum test duration for different sampletimes

<table>
<thead>
<tr>
<th>Sampletime</th>
<th>Maximum Test Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 ms</td>
<td>7 hours 6 minutes</td>
</tr>
<tr>
<td>200 ms</td>
<td>14 hours 13 minutes</td>
</tr>
<tr>
<td>500 ms</td>
<td>1 day 11 hours</td>
</tr>
<tr>
<td>1 s</td>
<td>2 days 23 hours</td>
</tr>
<tr>
<td>2 s</td>
<td>5 days 22 hours</td>
</tr>
<tr>
<td>5 s</td>
<td>14 days 19 hours</td>
</tr>
</tbody>
</table>

B.1.2 Running a Tensile Test

- If the mode is shown as Compression, then click on Setup → Mode → Tensile to set the system to tensile mode.

- Enter sample details in Setup → Sample Details. This allows the software to display the current stress and strain during the test, and record the sample details to the saved file after testing is over.

- Set the Sampletime and MotorSpeed. For highly elastic samples, use a longer sample time and a faster motor speed. For brittle samples, use a slow motor speed and a fast sample time (100mS). Make sure that the selected sampletime will allow enough time for the test to complete (See Table ??).

- Set the gain if necessary. Calculate the maximum load that you plan to reach, and choose the smallest range that includes this load. This will ensure the highest gain resolution for this range. If the load exceeds this range during the test, the gain will automatically scale, but a few data points will be lost at the point where the resolution changes.

- There are several options for testing strategies, including prescribing extension or load, or manually starting and stopping the motor. For full options, see Section ??.
• Save file to disk. CSV file format is recommended. This format can be opened as a data array in Excel, as well as in Microtest, including all start/stop markers. The sample and test details are stored in the file header. MTR files can only be opened by Microtest.

B.2 Compression Testing

B.2.1 Mounting a Sample

*Note: Travel is 5-15mm using the standard compression jaws.*

**Procedure**

• Undo the four M3x50 cover fixing screws. If tensile jaws are fitted, the top two M6 caphead screws need to be removed as well. Remove the top cover.

![Figure B.7: Top cover](image)

• If tensile jaws are already fitted, remove the tensile clamps. Attach the lower compressive jaw.

• Assemble the top jaw assembly as shown in Figure ???. Clamp the top jaw together using two M4x16 screws.
• Unscrew the collar assembly from the top cover (Fig. ??).

• Place the collar onto the top jaw assembly as indicated in Figure ??.

• Fix the top cap to the top jaw assembly and secure using two M6x25 caphead screws (Fig. ??).

• Screw the top jaw assembly into the microtest stage (Fig. ??).

• Replace the top cover and secure using the four M3x50 caphead screws.
To load a sample, remove the top cover, leaving the two M6 caphead screws in position in the top assembly.

If the mode is shown as Tensile then click on Setup → Mode → Compression to set the system to compression mode.
Note: The jaws cannot move if the force reading is 'Underrange'. If this happens during setup, adjust zero offset to continue.

- Set the minimum extensiometer position to 15 mm in Setup → Module Setup. Click "Apply". Press "Open jaws". This will allow the jaws to open to the hardware limit.

- Once the module has reached its limit of travel, press "STOP" and change the minimum to 5 mm and press 'Apply' and 'OK'. Set the current position to 15.992 mm in Displacement → Set Current Position.

- Measure the height of the sample and use the "Goto mm (absolute)" function to move the jaws to a distance that is the sample height plus 0.5mm. For example, if the sample height is 10mm, move the jaws to a distance of 10.5mm.

- Place the sample on the bottom jaw and offset the force to 0N with Tools → Set Loadcell zero offset.

- Refit the top cover and tighten the four M3 screws. If a force increase is seen during this step, then the jaws may be too close together and contacting the sample. Use the "Goto mm(absolute)" function to increase the distance between the jaws.

- Using the software, click "Goto" and enter a force between 1N to 5N. This will move the jaws so that contact is made with the sample. When this is complete, the motor will stop automatically.

- Click "Clear Data".

- The stage is now ready for a compression test.
B.2.2 Running a Compression Test

- Verify that mode is Compression.

- Set the Sampletime and MotorSpeed. For highly elastic samples, use a longer sampletime and a faster motor speed. For brittle samples, use a slow motorspeed and a fast sampletime (100mS). Make sure that the selected sampletime will allow enough time for the test to complete (See Table ??).

- Set the gain if necessary. Calculate the maximum load that you plan to reach, and choose the smallest range that includes this load. This will ensure the highest gain resolution for this range. If the load exceeds this range during the test, the gain will automatically scale, but a few data points will be lost at the point where the resolution changes.

- There are several options for testing strategies, including prescribing extension or load, or manually starting and stopping the motor. For full options, see Section ??.

- Save file to disk. CSV file format is recommended. This format can be opened as a data array in Excel, as well as in Microtest, including all start/stop markers. The sample and test details are stored in the file header. MTR files can only be opened by Microtest.

B.3 Motor Controls

B.3.1 Manual Controls

Start Motor

Starts the motor and data acquisition. The motor will start and the force, extension and elapsed time will be displayed. The speed of the motor can be changed in
Figure B.13: Manual motor controls

"MotorSpeed".

**Stop Motor**

Stops the motor but allows data acquisition to continue, allowing relaxation of the sample to be monitored. To continue recording data click on "Start Motor" again.

**Constant Load**

This button is only enabled while the motor is running. Pressing this will start the constant load mode. In this mode, the Microtest software will attempt to maintain the current load on the sample for an indefinite period. The motor will run for short periods in both directions as necessary in order to take up slack or to back off slightly. A prompt will appear on the graph to indicate that this mode is active. It may not always be possible to maintain a constant load in cases where the sample exhibits high deformation, or is in the breaking transition. Be careful, since breaking characteristics will be changed, as the module will attempt to maintain the decreasing force found during fracture.
Ramp Load

This feature aims to exert a steadily increasing (or decreasing) force upon the sample. The mode is invoked by clicking on the "Ramp Load" button on the motor control panel. This then brings up a dialogue box in which the user can enter the rate of change of load on the sample. This value is entered in N/min and can range from 1N/min up to the maximum value of the loadcell. If this value is positive, the force increases with time. If it is negative, the force decreases. Clicking on "OK" activates the mode. A status box shows the current force, the target force, and the error. If the force reaches maximum, or zero, the module is stopped. The module can be stopped at any time by clicking "STOP" or the "Ramp Load" button.

Clear data

This clears the current data from the acquisition display area and resets the extension reading to zero. The Start position will also be updated to match the current Absolute position.

Close jaws

Clicking "Close jaws" will move the stage until the jaws are as close together as allowed by hardware and software limits. If the force goes overrange or underrange during this moves, the motor will be stopped automatically.

STOP

At any time during any action, clicking the red STOP button will stop the motor immediately, as well as data recording.
Open jaws

Clicking "Open jaws" will similarly move the jaws as far apart as allowed. If the force goes overrange or underrange during this moves, the motor will be stopped automatically.

B.3.2 Go to Controls

You can select to move to a specific force, absolute position, or extension, or for a specific duration. These target values can be above or below the current equivalent value (except for time moves), which means that the stage will move in either direction to achieve the requested result whilst still recording data. All Goto movements are carried out at the current specified motor speed. To perform a Goto function, simply select the desired units from the top four items (N, absolute, extension, time) and then enter the target value into the box above. Finally, click "Go to" and the stage will move accordingly. If the requested target value is unachievable, a message will be displayed. Pressing the red "STOP" button at any time will abort the current move.

The following five options allow various actions to be performed once the target value has been reached:
Stop

When the target is reached, the motor and data acquisition will stop. This is equivalent to pressing the red STOP button.

Measure

When the target is reached, the motor will be stopped but acquisition will continue. This is equivalent to pressing the Stop motor button.

Constant load

When the target is reached, the software will automatically switch to constant load mode, as described in Section ??.

Step

If this action is selected then the adjacent "Set" button must be used to enter a value before clicking "Go to". The step value can be positive or negative and this allows the force or position to be moved in specified steps. The motor and acquisition are stopped after each movement. This allows a complete test to be performed in a number of discrete steps which enables images or measurements to be taken as required. Clicking "Go to" will move to the next step.

Cyclic test

If this action is selected then the adjacent "Set" button must be used to enter the desired values for the cyclic test before the "Go to" button is clicked. As soon as the target is reached during the goto move, a cyclic test will automatically start.
B.4 Important Software Features

B.4.1 Setup Menu

Module Setup

You can change the software displacement limit in this window (Extensometer- Minimum (mm) ). This should be the only setting you will need to change in everyday operation, unless the PID values need to be adjusted.

Mode

Switch between Tensile and Compression modes.

Screen

Choose the elements to be displayed on the graph, including line style, start and stop markers, and text information.

Sample Details

Enter height, width, and thickness of a rectangular sample or diameter and thickness of a circular sample. This information is used to compute stress and strain, and is recorded at the top of any saved data file.

B.4.2 Xscale

Changes what parameter is displayed on the x axis: points, actual distance, extension distance, or time.
B.4.3 Sampletime

Change the rate at which data points are recorded. Faster sampling rates are generally better, but a maximum of 256,000 points can be recorded. See Table ?? for maximum test duration for each sampling rate.

B.4.4 MotorSpeed

The displacement rate at which the motor will move.

B.4.5 Gain

Changes the hardware gain of the ADC force reading. For x 1 (full scale), the 16 bit ADC resolution is distributed across 0-5000 N. For x 2 the resolution is distributed over 0-2500 N, down to x 50 for 0-100 N. Compute the maximum planned force beforehand, and choose the gain that will give the finest resolution over that range. If the load exceeds this range during the test, the gain will automatically scale, but a few data points will be lost at the point where the resolution changes.

B.4.6 Tools

Cursors

Attaches a cursor to the load curve, displaying the force and the reading for the chosen x parameter. This is useful for determining what range of data points to look at in Excel. Right click to jump to the maximum load data point.

Zoom

Zooms in on a section of the load curve. Drag the shaded rectangle on the main graph to move the zoom region. When the track box is enabled, the window will automatically move as new data is recorded.
Stress/Strain

Displays the real time stress and strain in the sample, if the correct sample details have been entered.

Break Detection

For unattended tests, it can be helpful to stop the system if the sample fractures. This normally causes the measured force to drop below a certain value and this drop can be detected using the Break detection function. When enabled, the load must first rise above the top dotted line on the graph (Force + Hysteresis). Once this line has been passed, break detection will activate once the load falls below the bottom dotted line (Force). When this happens, both the motor and data recording are stopped.

Set Loadcell zero offset

Before inserting a sample, the loadcell amplifier zero offset must be set. This can be done manually or automatically. The easiest method is to press Auto, then drag the bar right or left until the force reading is as close to zero as possible. When you insert the sample and tighten the clamps the force may change slightly because the sample is now under stress. This could be a positive or negative stress, so the reading could be positive or negative. Since the loadcell zero was set with no sample fitted, the reading should now be showing the true force being applied to the sample.

Show jog controls

Manually position the motor. First press a speed, and then drag the slider left or right. Left moves the jaws together, right moves them apart.
B.4.7 Displacement

Set Current Position

During sample setup, this is the window where you enter the distance between the jaws.
REFERENCES


