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by

Sujith G. Kalarikkal
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Abstract of Thesis Presented to the Graduate School of the University of Florida in Partial Fulfillment of the Requirements for the Degree of Master of Science

FRACTURE TOUGHNESS OF GRAPHITE/EPOXY LAMINATES AT CRYOGENIC CONDITIONS

By

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The research presented in this thesis is an effort to better understand the interlaminar fracture behavior of graphite/epoxy composite laminates at cryogenic conditions. The double cantilever beam test was performed on different types of specimens, at room and cryogenic temperatures, and the fracture toughness was calculated from their load-displacement diagram. Neat resin fracture toughness was also measured using compact tension specimens. Additionally, the fracture toughness of some textile specimens and specimens treated with nano-particles was measured. It was observed that the all the specimens, excluding the textile specimen, showed a deterioration in fracture toughness at the cryogenic condition. Nano-treated specimens showed an improvement in fracture toughness, both at room and cryogenic temperatures. Finally, a finite element model was built and the fracture toughness calculated from the J-integral method and was compared to the experimental results.
CHAPTER 1
INTRODUCTION

Next generation space missions emphasize cutting the cost of launching payloads into orbit. One solution to this is the design and development of reusable launch vehicles (RLV) that would cut the cost of launching payloads into orbit from $10,000 per pound to $1000 per pound [1]. Weight is the most significant variable in the overall design of RLV. A decrease in structural weight will result in reduced fuel load, reduced cost, or increased payload. The major contributor to the mass of the launcher is the cryogenic propellant storage tank, which is the dominating component of the vehicle structure [2]. The weight of the tank can be reduced by the use of new and advanced materials with good strength to weight ratios. Hence, the application of lightweight composites over metals seems to be the best alternative [3]. The use of light-weight composites will also reduce the inertial loads.

The X-33 RLV, which was a joint venture of NASA and Lockheed Martin Aeronautics Company, utilized liquid hydrogen (LH2) tanks that were made of an advanced carbon composite. The walls of the tanks were made of two thin slabs of lightweight graphite composite around a Nomex honeycomb core. The two face sheets were bonded to the core using a layer of epoxy adhesive [4]. The X-33 liquid hydrogen tank failed when it was ground tested at Marshal Space Flight Center [1]. Just before the failure, the tank had undergone a test in which it was completely filled with liquid hydrogen. The failure occurred after the liquid hydrogen was drained out. The very low temperature experienced by the tank when it was filled with liquid hydrogen had caused
micro cracks in the matrix of the inner face sheet. The micro-cracks allowed hydrogen to leak into the core. When the liquid hydrogen was drained out, the tank warmed up closing the micro-cracks. The hydrogen trapped in the core started expanding and the pressure built up in the core resulted in the rupture of the outer composite skin.

**Composites**

The dictionary defines composite as a structure or an entity made up of distinct components. A composite material is a macroscopic combination of two or more materials, having a distinguishable interface between them. Depending upon the method by which the constituents are put together, the resulting composite materials may have all the properties of the constituents or significantly different properties than the individual constituents. Composites are used for their superior structural, electrical, and tribological, properties in a variety of environmental conditions. Modern day composites are usually optimized to accomplish a unique balance of properties for a specified set of applications.

Given the wide array of materials that can be perceived as composites it is difficult to concur upon a single, simple definition for composite. As a common practical definition, composite materials that are used for structural properties may be limited to those materials that contain a continuous matrix constituent that binds together and provides shape to an assemblage of stiffer, stronger reinforcement. The resulting composite material has a structural property that is superior to the individual constituent materials due to load sharing.
Classification of Composite Materials

There are two distinct levels by which a composite can be classified. The first approach is based on the nature of the matrix constituent. These include organic-matrix composites or polymer matrix composites (PMCs), metal-matrix composites (MMCs), and ceramic-matrix composites (CMCs) [5]. Organic-matrix composites include two classes of composites: polymer-matrix composites and carbon-matrix composites.

The second approach to classification is based on the reinforcement form. These include particulate composite, flake composite, fiber reinforced composite and laminated composite (Figure 1-1) [5]. The fiber reinforced composites are the most commonly used form of composite.

Figure 1-1: Classification of composites based on reinforcement

Fiber-reinforced Composites

Structural composites typically use fiber as the reinforcement because many materials are stiffer and stronger in the fiber form than when they are in the bulk form. The smaller the diameter of the fiber, the smaller the chance of any flaws surfacing...
due to fabrication. Also for very small diameters, the fiber strength is almost equal to the theoretical cohesive strength between the layers of atoms. Though there are numerous advantages of using a material in fiber form there are some disadvantages. Fibers have poor transverse mechanical properties compared to the longitudinal property. They also cannot support compressive loads in the longitudinal direction. Hence, fibers are held together with a binder or matrix material to form a structural unit. The matrix also protects the fibers from external damage and environmental attack.

Fiber reinforced composites can be classified into continuous fiber composite, woven fiber composite, chopped fiber composite and hybrid composite (Figure 1-2) [6]. Continuous fiber composites have lengths much greater than the cross-sectional dimensions. Individual continuous fiber composite laminae are oriented in the required direction and bonded together to form a laminate. They are extensively used for structural application, though they are susceptible to delamination because the interlaminar strength is matrix dominated. In a woven fiber composite, the fibers are braided or knitted to create interlocking fibers that often have orientations orthogonal to the primary to the structural plane. They do not delaminate but have low strength and stiffness compared to the continuous fiber composite. They are used in structures where there is a need to have a structural, thermal or electrical property in the out of plane direction. Chopped fiber composites are used in high-volume applications. They have significantly poorer mechanical properties compared to continuous fiber composites. Hybrid composites are made of chopped and continuous fiber composites, or different fiber materials.
Laminated Composites

Laminates are made by bonding together thin layers of materials. The individual layers of a composite laminate may be of different materials or the same material oriented in different directions. Most composite laminates used for structural applications are made of continuous fiber-reinforced composites. These laminates usually have plies with fibers oriented in different directions. Since the fibers are stiffer and stronger than the matrix, a ply is stiffer and stronger in the fiber direction. Hence, laminates with fibers oriented in the same direction would have poor mechanical properties in the fiber transverse direction.

Laminates are described by the direction of the fibers in the individual plies. This form of notation is called the stacking sequence. The stacking sequence lists the
orientation of the fibers in each ply of the laminate measured from a reference axis. The outer ply fiber direction is chosen as the reference direction.

**Damage in Laminated Composites**

In most laminated composites, the damage often starts with matrix cracking transverse to the principal loading direction. Matrix micro-cracking is followed by other modes of damage such as fiber breakage, fiber debonding from the matrix, and interlaminar separations usually known as delamination, possibly assisted by the stress concentration at the tip of the micro-cracks. The damage may be a result of the cure-induced residual stresses arising during manufacture, external impact damage, environmental effects, fabrication processes, or may develop in service. This damage causes the structure to fail well below the strength levels expected for a defect-free material.

As explained earlier, in the case of the X-33 RLV, its hydrogen tank failed due to the formation of micro-cracks at the very low temperature. The polymer matrix of the composite laminate has a coefficient of thermal expansion (CTE) many times greater than the CTE of the fiber; hence it will strain and shrink more when exposed to cryogenic temperature. The fiber, with almost a near zero CTE, tends to resist this strain and hence there will be a stress build up in the polymer matrix. Micro-cracks tend to appear if the stresses are sufficiently high. The micro-cracks and the delamination between the plies created a passage for the liquid hydrogen to permeate through the inner face-sheet into the honeycomb core.

Delamination is of particular interest as it is one of the most serious and common modes of damage in high performance laminated composites. The delamination in a composite laminate usually occurs at the interface of two different ply orientations.
Interlaminar delamination may be defined as the debonding of plies due to the interlaminar stresses present in the laminate. Delamination can considerably reduce the compressive strength of the composite structure by delamination buckling. Also as in the case of the X-33 RLV LH2 tank, the use of composites for cryogenic applications such as the liquid hydrogen fuel tanks, the delamination in the laminate can produce passage for the liquid hydrogen to seep through thus increasing the permeability of the laminate.

There are several different methods for increasing the delamination resistance of composite laminates. Most of these methods employ the introduction of translaminar reinforcement in the composite by stitching i.e., sewing threads or yarns, and by inserting pins called z-pins. The introduction of nano-particles such as aluminum oxide or zinc oxide in the composite laminate is an emerging technique to improve the structural property of the laminates including the delamination resistance.

**Objectives and Approach**

In this section the objectives of the current research and a brief outline of the research approach towards achieving the goals will be discussed.

**Objectives**

The main focus of this research is to determine the effect of cryogenic temperatures on the delamination resistance of composite laminates used for structural applications. A further objective is to explore techniques to increase the delamination fracture toughness of composite laminates.

This research also aims at developing a finite element model to predict the fracture toughness of the composite laminate at room and cryogenic temperature in conjunction with the experimental data.
Approach

This research involves experimentally determining the fracture toughness of the laminated composite and the polymer matrix. The double cantilever beam (DCB) test is a common test performed on composite laminates to determine the interlaminar fracture toughness. The DCB test will be performed on the composite laminate specimens to determine the fracture toughness. The compact tension test will be performed on the neat resin specimens to determine its fracture toughness at room and cryogenic temperatures.

For the fracture tests at cryogenic temperatures, a cryogenic test chamber has been designed and fabricated. The double cantilever beam test and the compact tension test have been performed on the composite laminate and neat resin specimens, respectively in the cryogenic chamber.

Fracture toughness tests have been performed on composite laminate specimens treated with nano-particles of alumina (Al₂O₃) to investigate the possibility of using nano-impregnated composites to improve interlaminar fracture toughness (delamination resistance). Also, textile composites will be tested for fracture toughness to compare their relative ability to withstand delamination.

A finite element model has been built using the commercial software ABAQUS® and the results are compared with the experimental results. It is important to input suitable mechanical properties, thus suitable experiments have been performed to find the mechanical properties of the composite.
CHAPTER 2
BACKGROUND

Fracture toughness is defined as the measure of damage tolerance of a material containing initial flaws or cracks. It is the ability of a material to resist the propagation of an already existing crack when subjected to external loads. There are two alternative approaches to fracture analysis: the stress intensity and the energy criterion approach.

**Stress Intensity Approach**

The stress intensity approach uses a constant known as the stress intensity factor $K_I$. The stress intensity factor completely characterizes the crack tip conditions in a linear elastic material. Figure 2-1 shows the stress distribution on an element ahead of a crack tip in an elastic material. Each stress component is directly proportional to the stress intensity factors as shown by Equations 2-1 to 2-3 [7].

![Figure 2-1: Stress distribution on an element ahead of a crack](image)

Figure 2-1: Stress distribution on an element ahead of a crack
\[
\sigma_{ss} = \frac{K_i}{\sqrt{2\pi r}} \cos \left(\frac{\theta}{2}\right) \left[1 - \sin \left(\frac{\theta}{2}\right) \sin \left(\frac{3\theta}{2}\right)\right]
\]

(2-1)

\[
\sigma_{yy} = \frac{K_i}{\sqrt{2\pi r}} \cos \left(\frac{\theta}{2}\right) \left[1 + \sin \left(\frac{\theta}{2}\right) \sin \left(\frac{3\theta}{2}\right)\right]
\]

(2-2)

\[
\tau_{xy} = \frac{K_i}{\sqrt{2\pi r}} \cos \left(\frac{\theta}{2}\right) \sin \left(\frac{\theta}{2}\right) \cos \left(\frac{3\theta}{2}\right)
\]

(2-3)

By knowing the stress intensity factor the entire stress distribution ahead of the crack tip can be computed using Equations 2-1 to 2-3. If we assume that the fracture occurs at some critical combination of stress and strain, it follows that failure occurs at some critical stress intensity, \(K_{IC}\) [7]. \(K_{IC}\) is a measure of the fracture toughness and is a material constant which is independent of the size and geometry of the cracked body.

**Energy Criterion Approach**

The energy approach predicates that a crack propagates when the energy available for crack growth is sufficient to overcome the resistance of the material. The material resistance may include plastic work, surface energy or other types of energy dissipation connected with a propagating crack. The energy available for the increment of the crack is called the energy release rate \(G\), which is also called the crack extension force or the crack driving force. For example, for a wide plate in plane stress with a crack of length 2\(a\) [7] the energy release rate is given by

\[
G = \frac{\pi \sigma^2 a}{E}
\]

(2-4)

The energy release rate \(G\) is defined as the rate of change in potential energy with crack area. It is an alternative measure of the fracture toughness. The energy release rate at the moment of crack propagation is called the critical energy release rate \(G_C\). \(G_C\) is the
material resistance to fracture. The critical energy release rate, \( G_C \), for a double cantilever beam is given by

\[
G_C = \frac{F^2 a^2}{B E I}
\]

where \( F \) is the critical force at which the crack propagates, \( a \) is the initial crack length and \( B \) is the width of the specimen and \( E I \) is the flexural rigidity of the specimen.

**Relation between Stress Intensity Factor and Energy Release Rate**

\( K_I \) is a local parameter and \( G \) is a global parameter. \( K_I \) and \( G \) are uniquely related for a linear elastic material. For an isotropic material the relation between \( G \) and \( K_I \) is given by

\[
G = \frac{K_I^2}{E'}
\]

Where \( E' = E \) for plane stress

And \( E' = \frac{E}{1 - \nu^2} \) for plane strain

**Modes of Fracture**

In Fracture mechanics there are three modes of loading as shown by the Figure 2-2. In mode I the forces act perpendicular to the crack. The Mode I loading condition is also called the opening mode. In Mode II the forces act parallel to the crack plane. One force is pushing the top half of the crack back and the other force is pulling the bottom half forward. This creates a shear crack. Since the forces do not cause the material to move out of its original plane this is also called in-plane shear. In mode III the forces are perpendicular to the crack and pull the top half left and the bottom half right or vice versa. This causes the material to separate out of its plane. It is also called out-of-plane shear.
In most practical applications materials are vulnerable to fracture by Mode I than by Mode II or Mode III. Usually, Mode II and Mode III loading do not lead to fracture and are significant only when they are applied to a weak interface in a material. In the case of fiber reinforced composites Mode II fiber/matrix debonding and Mode II delamination can occur.

In most real time applications a combination of these different loading modes leads to crack propagation. The combination of the loading modes, called the mode-mixity, is determined by a quantity called the phase angle “$\psi$”. The phase angle varies from 0° which is pure mode I to 90° which is pure mode II loading. The phase angle for a material under mode I and mode II loading is calculated as [7]

$$\psi = \tan^{-1}\left(\frac{K_{II}}{K_I}\right)$$  \hspace{1cm} (2-7)

The total energy release rate is the sum of the individual energy release rate from each mode. The crack traverses through the path of least resistance and hence need not be restricted to its initial plane. The crack will propagate in such a way to maximize the energy release rate for an isotropic or homogeneous material.

Figure 2-2: Modes of Loading
CHAPTER 3
LITERATURE REVIEW

Much research has been done in the area of interlaminar fracture toughness of carbon/epoxy composites. Most of them focus on the effect of high temperature and moisture on the fracture resistance. Little work has been done to study the effect of cryogenic temperature.

Davidson, Kruger and Konig [8] determined the effect of stacking sequence on energy release rate (ERR) distribution across the specimen width in multidirectional DCB specimens. They investigated eight different stacking sequences, with the delamination growth between $30^\circ/30^\circ$ interface and $30^\circ/-30^\circ$ interface. Their test did not favor any particular specimen and found that all specimens exhibited an asymmetry in the energy release rate distribution about the center of the specimen’s width and is a function of $D_{16}/D_{11}$. They concluded that the difference in peak ERR is more important than the small asymmetry in the ERR distribution.

Sun and Zheng [9] analyzed the distribution of strain energy release rate, $G$, at the crack fronts of DCB specimens by means of the plate finite element. They found a boundary layer phenomenon in the distribution of $G$ at the crack front, which causes the strain energy release rate to vary along the straight crack front. Sun et al. [9] found that the beam theory will underestimate $G_C$ for a curved crack front, which is observed in actual test specimens. They also found that for laminated composites containing angle plies the distribution of $G$ is skewed and the skewness depends on the lay-up sequence.
Delamination crack growth of unidirectional CFRP in thermo-mechanical fatigue was conducted by Nakai, Sakata, Kadowaki and Hiwa [10]. They performed the tests in air and water varying the test temperature. They found that the crack growth was unaltered for the isothermal fatigue test when the test temperature was changed from lower to higher. They also found that the crack growth was almost the same in water.

Asp [11] investigated the influence of moisture and temperature on the interlaminar delamination toughness of HTA/6376C carbon/epoxy composite. He found that critical strain energy release rate in pure mode I, is unaffected by changes in moisture content and was found to increase slightly at elevated temperatures. The enhanced fiber bridging due to increase in temperature and moisture content promotes R-curve behavior in mode I tests. He also found that in pure mode II the critical strain energy release rate drops with moisture content and increase with temperature. In mixed mode the critical strain-energy release rate also decreases with moisture content, but no general trends in dependence of temperature was observed.

Ashcroft, Hughes and Shaw [12] conducted mode I constant displacement rate tests on epoxy bonded CFRP joints at -50, 22 and 90°C. They observed that the temperature influenced the mode of fracture which progressed from stable, brittle fracture at low temperatures to slip-stick fracture at room temperature and finally to stable ductile behavior at elevated temperatures. Ashcroft et al. [12] too found that the critical strain energy release rate increased with temperature.

Mode I interlaminar fracture of carbon/epoxy cross ply composites was investigated by de Morais, de Moura, Marques and de Castro [13]. They performed mode I DCB tests on carbon/epoxy \([0°/90°]_{12}\) specimens with the starter crack between
the 0° and 90° mid-layer. They observed that the crack propagated along the
neighbouring 0°/90° interface and within the 90° mid-layer. They measured the
interlaminar critical strain energy release rate using a corrected beam theory and found
that the intralaminar $G_C$ is significantly smaller than the interlaminar $G_C$. This would
prevent pure interlaminar propagation in multi-directional specimens with high
interlaminar fracture toughness.

De Morais [14] performed the DCB testing of multidirectional laminates. He also
analyzed the DCB specimen’s using 3D finite element models. Using an interlaminar
stress based criterion, he predicted that the $G_C$ of multidirectional specimens is typically
10-40% higher than the $G_C$ of unidirectional laminates.

Wu et al. [15] investigated the mechanical, thermal and morphological properties
of glass fiber and carbon fiber reinforced polyamide-6 and polyamide-6/clay nano-
composites. They found that the effect of nanoscale clay on toughness was more
significant than that of the fiber. Other researchers like Becker, Varley and Simon [16]
also experimented with the use of layered silicates to enhance the fracture toughness of
high performance carbon epoxy composites. They found that the use of layered silicates
increased the maximum load by about 25% along with a 50% increase in the critical
strain energy release rate.
EXPERIMENTAL SETUP

Experimental Technique

To understand the effect of cryogenic temperatures on the fracture toughness of laminated composites it is pertinent that we perform fracture toughness tests on the laminated composite at cryogenic temperatures. There are different techniques by which one can measure the fracture toughness of a material. The most common tests are the drum peel test, double cantilever beam test (DCB) and four point bending tests. The double cantilever test was chosen because of its simplicity and the ease to prepare the specimens. It also has a relatively high accuracy when compared to the other tests. ASTM D-5528 describes the standards for performing the double cantilever beam test on unidirectional composite laminates.

The DCB test is a displacement controlled loading test. It gives rise to stable crack growth which makes it appropriate to measure the strain energy release rate $G$. Delamination can be predicted by knowing the strain energy release rate $G$ for a given structural geometry, loading condition and delamination location and comparing it with the critical value $G_C$.

The DCB test is performed on a specimen with an initial crack in the interface of the plies of interest. The specimen is loaded with a pure mode I as shown in the Figure 4-1.
The specimen is first loaded and the crack is allowed to propagate to a specified length ‘\( \Delta a \)’. The specimen is then unloaded. If the DCB specimen is loaded linearly to \( P_1 \) where the crack of size \( a_1 \) begins to grow and the load drops to \( P_2 \) while the crack extends from \( a_1 \) to \( a_2 \) (Figure 4-2), then the loss in strain energy due to crack extension is given by the area \( \Delta A \) between the loading and unloading curves.

The fracture toughness is measured as the energy release rate ‘\( G \)’, which is calculated from the load-displacement diagram for the specimen from the DCB test. The area method is used to calculate the fracture energy, which is given by

\[
G = \frac{\Delta A}{B\Delta a}
\]  

(3-1)

Where \( \Delta A \) is the area between the loading and unloading curves, \( \Delta a \) is the crack extension and \( B \) is the width of the specimen.
Figure 4-2: Load-Displacement Curve for a DCB test

Specimen Fabrication

Double Cantilever Beam Specimen

The specimens were prepared from Toray Composites unidirectional carbon fiber prepreg (material designation A50TF266 S6 Class E, Fiber designation T800HB-12K-40B, matrix 3631). First the stacking sequence of the composite laminate was decided and the prepreg was cut to size depending upon the dimensions and lay-up orientations. The prepreg was cut on a clean cutting table, to prevent unwanted impurities sticking to it, using a sharp-edged blade and a ruler. The prepreg is stored in a freezer at very low temperature, when it is brought to room temperature is becomes tacky and hard to handle. So it is important that the prepreg be cut very fast. The unused prepreg is put back into the freezer to increase its shelf life.

The next step in the specimen preparation process is to lay the cut prepregs according to the desired fiber orientations in the composite laminate. The cut prepreg is laid one over the other according to the desired stacking sequence. Each time a new layer
is added, it is pressed with enough pressure to make sure that no air bubbles are trapped between the plies. A roller was used for this purpose, so as to apply uniform pressure on the lay-up. Also, the prepreg is tacky when it is at room temperature, this helps in sticking. The pre-crack is created in the laminate by placing a non-porous Teflon® sheet between the designated plies. Once the lay-up is complete the next step is to cure the laminate.

The laminates were cured in a computer controlled autoclave. Before the laminate is put in the autoclave it is first vacuum bagged for applying vacuum to the composite laminate during the cure cycle. An 18”x 18” aluminum plate was used as the base tool. The surface of the aluminum plate was polished and cleaned with sand paper. A non-porous Teflon sheet is laid on top of the base tool, to avoid the sticking of the composite laminate to the base tool. The laid-up composite laminate is then placed on the non-porous Teflon and another layer of non-porous Teflon is laid on top of the laminate (Figure 4-3). Care is taken to avoid any wrinkles or air-bubbles being trapped between the laminate and the non-porous Teflon sheets. This is achieved by gently rubbing the top of the Teflon sheet with the base of your palm. Since we are using non-porous Teflon as the release film, excess epoxy does not permeate to the breather from the laminate. Finally a breather is placed on top. The breather helps in uniformly spreading the vacuum throughout the laminate. All the components are then placed inside a vacuum bag and edges are sealed with a bag sealant (Figure 4-4). A small slit is cut on top of the vacuum bag for the vacuum plug. Care was taken to place the vacuum plug on one edge of the plate and not on top of the laminate. If the vacuum plug is placed on top of the laminate, it could produce an impression on the laminate when it is cured. Precautions
were taken to prevent any wrinkles forming in the vacuum bag material when the open sides are sealed.

Figure 4-3: Laminate with the release film

Figure 4-4: Vacuum bag Lay-up

The vacuum bagged laminate is then kept in the autoclave and the vacuum pump is switched on (Figure 4-5). As the air inside the bag is evacuated the vacuum bag fits snugly around the composite laminate. Any wrinkles that are formed during this process
are smoothened out. Once the vacuum pressure inside the bag reaches 20 inches Hg the autoclave door is shut and the cure-cycle was started. The laminate is cured at 180°C (355°F) for 3 hours. Figure 4-6 shows the cure cycle.

Figure 4-5: Vacuum Bagged Laminate with vacuum pump on.

Figure 4-6: Cure Cycle
The part is removed from the autoclave once the cure cycle is complete and has reached room temperature. The composite laminate is then removed from the vacuum bag and cut into strips using a diamond wet tile cutter. Each specimen was 177.8 mm (7 inches) in length; 25.4 mm (1 inch) in width and approximately 3.05 mm (0.12 inches) in thickness. Different sets of unidirectional laminate specimens were fabricated by this process with the pre-crack between two 0° plies, two 90° plies and between a 0° and a 90° ply, and finally between two bi-axially woven textile plies. Table 4-1 shows the specimen designation, lay-up and the number of plies in each specimen. The “D” in the lay-up signifies the pre-crack. In future “0D0” will be used to refer the specimen with the pre-crack between two zeroes, “90D90” refers the specimen with the pre-crack between two nineties, and “90D0” represents the pre-crack between a ninety and a zero specimen.

Table 4-1: Specimen Lay-up and Designation

<table>
<thead>
<tr>
<th>Crack between</th>
<th>Specimen Denomination</th>
<th>Lay-up</th>
<th>Total no. of plies</th>
</tr>
</thead>
<tbody>
<tr>
<td>two zeroes</td>
<td>0D0</td>
<td>[(0/90)_d/0/D]_s</td>
<td>18</td>
</tr>
<tr>
<td>two nineties</td>
<td>90D90</td>
<td>[(90/0)_d/90/D]_s</td>
<td>18</td>
</tr>
<tr>
<td>a zero and a ninety</td>
<td>90D0</td>
<td>[(0/90)_d/D/0/(90/0)_d]</td>
<td>17</td>
</tr>
<tr>
<td>two zeroes with 20% nano</td>
<td>0D0 N 20</td>
<td>[(0/90)_d/0/D]_s</td>
<td>18</td>
</tr>
<tr>
<td>two zeroes with 9% nano</td>
<td>0D0 N 9</td>
<td>[(0/90)_d/0/D]_s</td>
<td>18</td>
</tr>
<tr>
<td>textile</td>
<td>TEX</td>
<td></td>
<td>12</td>
</tr>
</tbody>
</table>

The interlaminar surfaces of some the specimens were treated with nano particles (38 nm Al\_2O\_3) to study its effect on the interlaminar fracture toughness. A thin layer of the nano particles was uniformly painted on the plies before laying them up. Two sets of specimens were prepared by this method by varying the mass of the nano-particles sprayed between the plies. The mass of a single ply of the unidirectional composite was weighed and 9 % and 20 % by mass of the nano-particle was sprayed on the plies making
the interface of the pre-crack before laying them up. In this thesis the 9% nano-particle treated specimen will be represented by “0D0_N_9” and “0D0_N_20” will be used to refer to the 20% nano-particle treated composite.

Finally some bi-axially woven textile composite laminate specimens were also prepared to see the effect of cryogenic temperature on the fracture toughness. The textile composite laminates used 12 plies to equal the mass of the unidirectional composite specimens. The textile composite specimens will be represented by the word “TEX”.

After the specimens are cut to the required dimensions, two loading blocks were attached to the DCB specimens for the application of the opening force (Figure 4-7). The loading blocks were fabricated from a rectangular steel rod and had dimensions 27.94 mm (1.1 inches) in length, 20.32 mm (0.8 inch) wide and 15.24 mm (0.6 inch) thick. A 9.525 mm (3/8-inch) hole was drilled through the center of each loading block to allow the insertion of a loading pin. Epoxy 907 manufactured by Miller-Stephenson chemical company was used as the adhesive to attach the loading blocks to the DCB specimens for the room temperature tests. When the same adhesive was used for the cryogenic tests it was found that the steel-tabs peeled off the specimen for very small loads. So different techniques such as tying the steel-tab to the laminate using a cotton yarn was used to secure it to the laminate during the cryogenic tests (Figure 4-8). In later tests PR-1665 cryogenic adhesive from PRC-DeSoto International was used for the cryogenic test.

The surfaces of the steel tabs were cleaned with a solvent and contaminants such as dirt, grease etc. were removed prior to the application of the sealant. A progressive cleaning procedure was used using new tissues each time. The sealant was applied uniformly on the surfaces and the steel-tabs were fixed to the laminates. Great care was
taken while mounting the steel-tabs to the composite laminate to ensure uniformity in the specimens.

Figure 4-7: Loading blocks attached to the specimen for loading

Figure 4-8: Loading blocks attached to the specimen
Epoxy 907 is a two part adhesive. Equal parts by volume of part A and part B were mixed thoroughly for 3 minutes on a clean surface. A thin layer of the mixed adhesive was then applied evenly on the loading blocks. The loading blocks were then mounted on the composite specimen. Pressure was applied on the blocks to squeeze out excess adhesive to form a thin glue line. The excess adhesive was removed with a knife. Great care was taken while mounting the loading block to ensure symmetry of the specimen. The adhesive was allowed to cure for 24 hours at 24°C.

PR-1665 is a high tear and tensile strength cryogenic potting and molding compound. It is supplied in a two-part kit, Part A and Part B. Part A was heated to 125°C with constant stirring and allowed to cure before using. Part B was also heated to 60°C with constant stirring and was cooled before mixing. Using a mixing paddle, 35:100 ratio parts by volume of Part A and Part B was mixed thoroughly. The mixed adhesive is then applied uniformly on the loading blocks, which were then mounted on the composite specimens. A small pressure was applied on the loading blocks to squeeze out the excess adhesive which was then removed with a knife. The adhesive was then cured at 24 °C for 24 hours and at 54 °C for 40 hours.

Once the steel tabs are fastened to the laminate, the edges of the specimen were painted with silver permanent marker. Lines are drawn at an interval of 25.4 mm (1 inch) starting from the initial crack tip so as to monitor the crack tip location.

**Compact Tension Test Specimen**

The compact tension specimens, for the fracture toughness test of the epoxy matrix, were prepared using US Composites 635 thin epoxy resin. The 635 resin system is used for lamination of fiberglass, carbon fiber, Kevlar or any type of reinforcement. Three parts by volume of the epoxy was mixed with one part of the hardener in a clean
container. Then the mixed epoxy system was poured into a paper mould and allowed to cure. The mixed epoxy has a pot life of 20-25 min with a cure time of 8-10 hours at room temperature. The cured specimens (Figure 4-9) were then machined to the desired dimensions. After the specimens were machined the holes for the loading pins were drilled.

![Compact Tension Specimen](image)

**Figure 4-9: Compact Tension Specimen**

ASTM D-5045 describes the standards to prepare the compact tension specimen. Initially, a notch was machined using the diamond saw as the pre-crack. To initiate the natural crack a sharp edged blade was inserted and tapped with a small force. It was observed that the epoxy was very brittle and that a small force was enough to initiate the crack to propagate the whole length, hence making the specimen unusable. To prevent this, a fresh blade was slid with a single motion to make the natural crack.

**Cryogenic Test Chamber**

A cryogenic chamber was designed and fabricated to perform the DCB test at cryogenic ambience. Liquid Nitrogen was used to produce the cryogenic condition.
Liquid Nitrogen boils at a temperature of 77K. Hence, it is essential that the cryogenic chamber be leak-proof. The cryogenic test chamber basically consists of two concentric steel drums of diameters 457.2 mm (18 in.) and 355.6 mm (14 in.) with an insulated annulus. One inch thick styro-foam sheets were used for the insulation. A 1.5 in. hole was cut on the center of the drums for bottom shaft. A screwing mechanism was used to prevent leakage near the shaft opening. The fixtures for the DCB test were fastened to the bottom shaft (Figure 4-10). The cryogenic test chamber was tested for leakage and was made sure that it was leak proof. Three polyethylene bags were used to cover the inside of the chamber as an additional safety measure. Figure 4-11 shows the cryo-chamber and the liquid nitrogen tank.

Figure 4-10: Inside of the Cryo Chamber showing the screwing mechanism and the fixture
There are several precautions that need to be taken while handling liquid nitrogen. Liquid Nitrogen boils at 77 K, and hence can cause frost bite when exposed to skin. It can spatter while being poured into the chamber and damage one’s eyes, so safety glasses must be worn at all times. Also the face should be kept away from the chamber when opening since Liquid Nitrogen displaces oxygen from its surrounding and hence can cause suffocation [17].
CHAPTER 5
FRACTURE TESTS

The double cantilever beam tests and the compact tension tests were both performed on a 12,000 lb refurbished MTI Phoenix machine with a 5,000 lb interface load cell (see Figure 4-1). Both the displacement of the crosshead and the load detection of the load cell were verified before starting the tests. The loading of the MTI was controlled by a computer which also recorded the load on the load cell and displacement of the crosshead.

Figure 5-1: MTI machine.
Double Cantilever Beam Test

The double cantilever beam tests, both room and cryogenic temperature tests, were performed at a crosshead displacement rate of 0.04 in./min to simulate quasi-static conditions.

Room Temperature Test

Each room temperature (24 ± 2 °C) test specimen was cyclically loaded and unloaded four times. The crack was allowed to propagate approximately 25.4 mm (1 in.) during each loading cycle. The crack growth was monitored during the loading cycle by using a microscope. Figure 5-2 shows the room temperature tests being performed on a specimen. Once the crack propagates to a distance of 25.4 mm (1 in.), the crosshead was held at hold and the tip of the crack was marked with a sharp tipped marker before starting the unloading cycle process. This mark indicates the tip of the initial crack for the next loading cycle. The process was repeated during each loading cycle. A typical load-displacement graph is shown in Figure 5-3.
Figure 5-3: Typical Room Temperature Load-displacement Diagram

Cryogenic Temperature Test

The in-situ DCB cryogenic temperature tests were performed in the cryogenic chamber by keeping the DCB specimen fully immersed in liquid nitrogen bath during the whole testing cycle. This was done to ensure uniform distribution of the cryogenic temperature on the specimen and eliminate any effects due to temperature gradients. Liquid nitrogen is constantly supplied to the cryogenic chamber to compensate for the liquid nitrogen lost due to boiling. Each specimen was kept immersed in liquid nitrogen for 30 min before starting the loading-unloading cycle. Figure 5-4 shows the DCB specimen in the cryogenic chamber prior to the start of the test.
Figure 5-4: Cryogenic Temperature DCB Test

Since the DCB specimen is immersed in the liquid nitrogen bath during testing, it was not possible to monitor the crack growth, so each specimen was removed from the liquid nitrogen after each loading and unloading cycle. The distance through which the crack propagated was measured using the microscope and the crack tip was marked with a sharp tipped marker on both sides of the DCB specimen. The specimen was again mounted and the test was repeated. Each cryogenic specimen underwent two loading-unloading cycles. Figure 5-5 shows a typical loading-unloading cycle at cryogenic temperature.
Compact Tension Test

Compact tension tests were performed on neat resin specimens in order to understand the effects of cryogenic conditions on the fracture toughness of the epoxy matrix. All the compact tension tests were also performed at a crosshead displacement rate of 0.04 in/min to simulate quasi-static conditions.

For both the room and cryogenic temperatures tests shown by Figures 5-6 and 5-7, the specimens were loaded till the crack traverses the whole width. A typical load-displacement diagram for the compact tension test is shown by Figure 5-8. The cryogenic temperature tests were performed with the specimens fully immersed in liquid nitrogen. Each specimen was kept immersed in liquid nitrogen for 30 min before starting the loading cycle.
Figure 5-6: Room Temperature Testing of Compact Tension Specimen

Figure 5-7: Cryogenic Testing of Compact Tension Specimen
Figure 5-8: Typical Load-Displacement from Compact Tension Test
CHAPTER 6
RESULTS AND DISCUSSION

Double Cantilever Beam Test

The \( G_C \) value was calculated using the specimen width, the length through which the crack propagated and the strain energy loss (Equation 3-1). The strain energy for each specimen was calculated from its load-displacement diagram. The load-displacement diagram for each specimen at room and cryogenic temperatures can be found in Appendix A and Appendix B, respectively.

Room Temperature Test

Table 6-1 shows the average \( G_{IC} \) values of two specimens (eight cycles) at room temperature for each specimen type.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fracture Toughness ( G_{IC} ) in N/m (lb/in)</th>
<th>Coefficient of Variation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0D0</td>
<td>224.59 (1.28)</td>
<td>7.20</td>
</tr>
<tr>
<td>90D90</td>
<td>394.53 (2.25)</td>
<td>8.63</td>
</tr>
<tr>
<td>90T0</td>
<td>176.91 (1.01)</td>
<td>10.34</td>
</tr>
<tr>
<td>0D0 20% nano</td>
<td>215.73 (1.23)</td>
<td>15.07</td>
</tr>
<tr>
<td>0D0 9% nano</td>
<td>442.57 (2.53)</td>
<td>9.50</td>
</tr>
<tr>
<td>TEX</td>
<td>422.09 (2.41)</td>
<td>10.02</td>
</tr>
</tbody>
</table>

The comparison of fracture toughness of the two unidirectional laminated composites, i.e., 0D0 and 90D90 specimens, show that the 90D90 specimens with the pre-crack between two 90° plies, is greater (394.53 N/m) than the ones with the pre-crack between two 0° plies (224.59 N/m). This is partly because the fibers in the 90D90 specimen act as barriers for the crack to propagate. The crack changes its path each time
the fibers block its path, hence more energy is required to initiate crack propagation. The crack followed a zigzag route, traveling from one 0/90 interface to the other 0/90 interface adjacent to the pre-crack.

The 90D0 specimen, with the crack between a 90° ply and a 0° ply showed the least resistance (176.91 N/m) to crack propagation. It was observed that the crack stayed on the interface of the two plies and did not jump as in the case of the 90D90 specimen. This might be due to the fact that the laminate is not symmetric and hence the specimen may not be under pure mode-I loading.

The 20% nano-impregnated specimen showed poorer fracture toughness (215.73 N/m) compared to the non-nano 0D0 specimen. The reason for the poor performance of the 0D0_N_20 specimen might be due to the possible agglomeration of the nano particles which might have hindered the adhesion of the plies during curing. Figure 6-1 shows the Scanning Electron Microscope (SEM) photograph of the crack surface of the 20% nano treated specimen. We can clearly see that the nano particles are not uniformly distributed on the surface and there are areas of high and low concentrations of the nano particle.

The 9% nano particle treated specimen shows the highest delamination resistance (442.57 N/m) compared to all the other specimen types. The reason might be the fact that the nano particles act as barrier for crack propagation and hence increases the energy required or the fracture toughness. Figure 6-2 shows SEM photograph of 9% nano treated specimen crack surface. We can see that in this case the nano particles are more uniformly distributed and there are no areas of high or low concentrations.
Figure 6-1: SEM image of 20% nano treated specimen crack surface.

Figure 6-2: SEM image of 9% nano treated specimen crack surface.
The textile laminate too show higher fracture toughness (422.09 N/m) compared to 0D0 specimens. In fact, it shows the second highest fracture toughness next to the 0D0_N_9 specimen. Figures 6-3 shows the SEM image of TEX crack surface. We can see that there are a lot of broken fibers. The higher fracture toughness might be due to fact that more energy is required to break the fibers for the crack to propagate.

Figure 6-3: SEM image of textile specimen crack surface.

Table 6-2 shows the percentage variation in fracture toughness of each specimen types with the 0D0 specimen as the reference.

Table 6-2: Percentage variation in $G_{IC}$ at room temperature

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fracture Toughness $G_{IC}$ in N/m (lb/in)</th>
<th>Percentage Increase or Decrease in $G_{IC}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0D0</td>
<td>224.59 (1.28)</td>
<td>N.A.</td>
</tr>
<tr>
<td>90D90</td>
<td>394.53 (2.25)</td>
<td>75.67</td>
</tr>
<tr>
<td>90T0</td>
<td>176.91 (1.01)</td>
<td>-21.23</td>
</tr>
<tr>
<td>0D0 20% nano</td>
<td>215.73 (1.23)</td>
<td>-3.94</td>
</tr>
<tr>
<td>0D0 9% nano</td>
<td>442.57 (2.53)</td>
<td>97.06</td>
</tr>
<tr>
<td>TEX</td>
<td>422.09 (2.41)</td>
<td>87.94</td>
</tr>
</tbody>
</table>
Cryogenic Temperature Test

Table 6-3 shows the average $G_{IC}$ values at cryogenic temperature for each specimen type.

Table 6-3: Fracture Toughness of DCB specimens at cryogenic temperature

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fracture Toughness $G_{IC}$ in N/m (lb/in)</th>
<th>Coefficient of Variation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0D0</td>
<td>164.07 (0.94)</td>
<td>22.32</td>
</tr>
<tr>
<td>90D90</td>
<td>235.54 (1.34)</td>
<td>14.95</td>
</tr>
<tr>
<td>90T0</td>
<td>173.07 (0.99)</td>
<td>42.32</td>
</tr>
<tr>
<td>0D0 20% nano</td>
<td>189.87 (1.08)</td>
<td>11.92</td>
</tr>
<tr>
<td>0D0 9% nano</td>
<td>250.75 (1.43)</td>
<td>19.15</td>
</tr>
<tr>
<td>TEX</td>
<td>826.93 (4.72)</td>
<td>6.19</td>
</tr>
</tbody>
</table>

It was seen that the fracture toughness deteriorates for both the unidirectional composite specimens. The 0D0 specimen showed fracture toughness of 164.07 N/m at cryogenic condition and the 90D90 specimen showed a fracture toughness of 235.54 N/m. The decrease in fracture toughness might be due to the development of thermal stresses in the laminate and formation of micro-cracks in the laminate at the low temperature which leads to overall degradation of the material.

The nano impregnated specimens too showed decrease in fracture toughness at the cryogenic temperature. It is interesting to see that the 0D0_N_9 specimen showed higher fracture toughness (250.75 N/m) compared to the 0D0 specimen (164.07 N/m) and 90D90 specimen (235.54 N/m).

The textile laminate showed the most interesting behavior at cryogenic temperature. From Table 5-3 we can see that the fracture toughness of the TEX specimen increased (826.93 N/m) at cryogenic temperature. It was observed that the crack does not propagate in one plane but traverses through the plies.
Table 6-4 shows the percentage variation in fracture toughness at cryogenic temperature of each specimen with the 0D0 specimen as the reference.

Table 6-4: Percentage variation in $G_{IC}$ at Cryogenic temperature with respect to the 0D0 specimen

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fracture Toughness $G_{IC}$ in N/m (lb/in)</th>
<th>Percentage Increase or Decrease in $G_{IC}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0D0</td>
<td>164.07 (0.94)</td>
<td>0</td>
</tr>
<tr>
<td>90D90</td>
<td>235.54 (1.34)</td>
<td>43.56</td>
</tr>
<tr>
<td>90T0</td>
<td>173.07 (0.99)</td>
<td>5.49</td>
</tr>
<tr>
<td>0D0 20% nano</td>
<td>189.87 (1.08)</td>
<td>15.72</td>
</tr>
<tr>
<td>0D0 9% nano</td>
<td>250.75 (1.43)</td>
<td>52.83</td>
</tr>
<tr>
<td>TEX</td>
<td>826.93 (4.72)</td>
<td>404.01</td>
</tr>
</tbody>
</table>

Table 6-5 compares the fracture toughness of all the specimens at room and cryogenic temperature.

Table 6-5: Comparison of $G_{IC}$ at room and cryogenic temperature

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fracture Toughness $G_{IC}$ in N/m (lb/in)</th>
<th>Percentage Increase or Decrease in $G_{IC}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Room Temperature</td>
<td>Cryo Temperature</td>
</tr>
<tr>
<td>0D0</td>
<td>224.59 (1.28)</td>
<td>164.07 (0.94)</td>
</tr>
<tr>
<td>90D90</td>
<td>394.53 (2.25)</td>
<td>235.54 (1.34)</td>
</tr>
<tr>
<td>90T0</td>
<td>176.91 (1.01)</td>
<td>173.07 (0.99)</td>
</tr>
<tr>
<td>0D0 20% nano</td>
<td>215.73 (1.23)</td>
<td>189.87 (1.08)</td>
</tr>
<tr>
<td>0D0 9% nano</td>
<td>442.57 (2.53)</td>
<td>250.75 (1.43)</td>
</tr>
<tr>
<td>TEX</td>
<td>422.09 (2.41)</td>
<td>826.93 (4.72)</td>
</tr>
</tbody>
</table>

Effect of Lay-up on $G_{IC}$

Comparing the fracture toughness of the 0D0 specimen and the 90D90 specimen, it can be seen that the fracture toughness decreases by 26.95 % and 40.29 % respectively compared to the room temperature specimens. It is also observed that the fracture toughness of the 90D90 specimen at cryogenic temperature is higher (235.54 N/m) than the fracture toughness of the 0D0 specimen at room temperature. Fig 6-4 shows the comparison of the fracture toughness of the two unidirectional specimens.
Figure 6-4: Effect of Lay-up on $G_C$

Figures 6-5 and 6-6 show the 0D0 and 90D90 crack surfaces. We can see that in the case of the 0D0 specimen the crack stays in its plane while the crack growth in 90D90 is not planar. The crack grows to the adjacent 0° ply in the case of the 90D90 specimens. As mentioned earlier the increase in fracture energy might be due to this deviation from the expected crack path.

Figure 6-5: SEM image showing 0D0 specimen crack surface.
Effect of Nano particles on $G_{IC}$

It was observed that the fracture toughness of the 9% nano particle treated composite was better (442.57 N/m and 250.75 N/m) than the non-nano 0D0 specimens (224.59 N/m and 164.07 N/m) at both room and cryogenic temperatures respectively. The nano particles act as obstruction to the propagation of the crack. The impregnation of 9% nano particles increases the fracture toughness by nearly 97% at room temperature and by approximately 53% at cryogenic temperature. It can also be seen that the fracture toughness of the 9% nano treated specimen at cryogenic temperature (250.75 N/m) is higher than the fracture toughness of the non-nano 0D0 specimen (224.59 N/m) at room temperature. Figure 6-7 shows the comparison in $G_{IC}$ of the nano impregnated and non-nano 0D0 specimens at room and cryogenic temperatures.

Figure 6-8 shows the SEM photograph of the 0D0_N_9 specimen crack surface. At this magnification we cannot see much difference between the 0D0_N_9 specimen...
and the 0D0 specimen (see Figure 6-5) crack surface. A more magnified view of the crack surfaces is required to study the topographical difference between the two surfaces.

![EFFECT OF NANO PARTICLES ON \( G_{IC} \)](image)

**Figure 6-7:** Effect of nano-particles on \( G_{IC} \)

![SEM image showing 9 % nano treated specimen crack surface.](image)

**Figure 6-8:** SEM image showing 9 % nano treated specimen crack surface.
Comparison in $G_{IC}$ of Unidirectional and Textile laminates

The textile laminate specimen (TEX) showed higher fracture toughness (422.09 N/m) compared to both the unidirectional (0D0 and 90D90) specimens. At cryogenic conditions the fracture toughness of the TEX specimens increased by nearly 96 % compared to the room temperature specimen. It can be deduced from this observation that the textile laminates are best suited for cryogenic application. Figure 6-9 shows the comparison of $G_{IC}$ of the unidirectional laminates and textile laminate.

![Graph comparing $G_{IC}$ values of unidirectional and textile laminates at different temperatures](image_url)

Figure 6-9: Comparison in $G_{IC}$ of Textile and unidirectional specimen

Figure 6-10 shows the SEM photograph of the Textile specimen crack surface. We can clearly see that there are numerous broken fibers on the surface of the crack. As mentioned earlier the increase in fracture toughness might be due to the fact that more energy is required to break the fibers. In the case of the cryogenic specimens it was observed that the crack growth was more translaminar than interlaminar i.e. the crack growth was not planar and propagated through the adjacent plies.
Figure 6-10: SEM image showing TEX specimen crack surface.

**Compact Tension Test**

As mentioned earlier CT specimens were used to measure the fracture toughness of the neat resin both at room and cryogenic temperature. The stress intensity factor $K_{IC}$ was calculated from the test results using the relation given by Equation 6-1. The $K_{IC}$ represents the lower limiting value of the fracture toughness. It is a function of the testing speed and temperature.

$$K_{IC} = \left( \frac{P_{\text{max}}}{BW^{1/2}} \right) f(x)$$  \hspace{1cm} (6-1)

Where, $f(x)$ is given by

$$f(x) = \frac{(2 + x)(0.886 + 4.64x - 13.32x^2 + 14.72x^3 - 5.6x^4)}{(1 - x)^{3/2}}$$  \hspace{1cm} (6-2)

where $P_{\text{max}}$ is the maximum load at failure, $B$ is the specimen thickness, $W$ is the specimen width, $a$ is the initial crack length and $x = a/W$. 
Results

A total of four specimens were each tested at room and cryogenic temperature. The load-displacement diagrams for the compact tension tests at room and cryogenic temperature can be found in Appendix C. The calculated fracture toughness at room temperature in terms of the stress intensity factor is given in Table 6-6.

Table 6-6: $K_{IC}$ at room temperature

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Load at Failure (lb)</th>
<th>Fracture Toughness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>psi in $^{1/2}$</td>
</tr>
<tr>
<td>1</td>
<td>239.27</td>
<td>2764.58</td>
</tr>
<tr>
<td>2</td>
<td>273.36</td>
<td>3157.96</td>
</tr>
<tr>
<td>3</td>
<td>242.10</td>
<td>2881.29</td>
</tr>
<tr>
<td>4</td>
<td>269.01</td>
<td>2990.60</td>
</tr>
</tbody>
</table>

Figure 6-11 shows the room temperature test specimens after the test was performed. It can be observed that the crack growth was steady and remained in its plane during fracture in the room temperature tests. The average fracture toughness at room temperature was found to be 3.24 MPa. m $^{1/2}$ (2948.61 psi in $^{1/2}$).

Figure 6-11: CTT room temperature specimen after the test.
Table 6-7 shows the fracture toughness results obtained from the cryogenic temperature test. We can see that the maximum load to initiate fracture was far less than the maximum load required at room temperature.

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Load at Failure (lb)</th>
<th>Fracture Toughness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>psi in$^{1/2}$</td>
<td>MPa. m$^{1/2}$</td>
</tr>
<tr>
<td>1</td>
<td>67.034</td>
<td>841.40</td>
</tr>
<tr>
<td>2</td>
<td>139.5646</td>
<td>1508.66</td>
</tr>
<tr>
<td>3</td>
<td>57.5276</td>
<td>691.95</td>
</tr>
<tr>
<td>4</td>
<td>134.5949</td>
<td>1642.58</td>
</tr>
</tbody>
</table>

Since the cryogenic specimens were directly dipped in liquid nitrogen, it was observed that the specimens began to crack due to the thermal shock even before the load was applied. Figure 6-12 shows the fractured test specimens. We can see that the crack growth is unsteady and is not confined to its original plane. The fracture toughness was calculated and the average was found to be 1.29 MPa. m$^{1/2}$ (1171.15 psi in$^{1/2}$).
Figure 6-13 sows a comparison of fracture toughness at room and cryogenic temperature. Since there was damage in the specimen even before running the test, we cannot actually compare the room temperature results to the cryogenic temperature results.

![Comparison in KI](image)

Figure 6-13: room and cryo temperature fracture toughness comparison of CTT

It can be concluded that the cryogenic temperature produces cracks in the epoxy matrix due to thermal shock, which deteriorates the performance of the material. For testing the fracture toughness of the epoxy at cryogenic temperature, some method should be developed which will slowly reduce temperature of the material to the cryogenic temperature, to eliminate fracture due to thermal shock.
CHAPTER 7
FINITE ELEMENT MODELING

A finite element model of the DCB test specimen at room temperature was built using the commercial FE software ABAQUS 6.4. Only the 0D0 and 90D90 specimens were modeled because of the complexity involved in the nano treated and the textile specimens. The fracture toughness results from the finite element model were compared to the fracture toughness results from the experiments.

Model Development

All 2-D models (plane stress) of the DCB specimens were developed using CPS8R 8 node bi-quadratic plane stress elements. The test specimens were used as the basis for the FE model. The laminate was divided into two halves and each half was built separately. The orientation dependent material properties were then assigned to each plies in the laminate. Then the two halves were tied at the appropriate surface to produce the laminate with the pre-crack. The two steel tabs were then modeled and attached to the top and bottom surfaces of the laminate as shown in Figure 7-1. The material properties used in the FE model for the steel and the unidirectional plies can be found in Table 7-1.

Table 7-1: Material Properties in Msi

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_1$</th>
<th>$E_2$</th>
<th>$E_3$</th>
<th>$v_{12}$</th>
<th>$v_{31}$</th>
<th>$v_{23}$</th>
<th>$G_{12}$</th>
<th>$G_{31}$</th>
<th>$G_{23}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel</td>
<td>30.0</td>
<td>30.0</td>
<td>30.0</td>
<td>0.30</td>
<td>0.30</td>
<td>0.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T800/3631</td>
<td>16.5</td>
<td>1.1</td>
<td>1.1</td>
<td>0.34</td>
<td>0.34</td>
<td>0.25</td>
<td>0.64</td>
<td>0.64</td>
<td>0.44</td>
</tr>
</tbody>
</table>
Figure 7-1: FE model of DCB specimen

The next step in the modeling procedure was the assignment of the boundary conditions. As in the case of the actual experimental setup, the center of the bottom steel tab, where the pin sits, was restricted to move in both the vertical and horizontal direction but was allowed to rotate. Similarly the center of the top steel tab was not allowed to move horizontally. It was allowed move vertically and rotate about its center. In the case of the cryogenic temperature models an additional temperature boundary condition was applied to the FE model.

The critical load from the experimental data, at which the crack propagated, was the applied load in the FE model. The crack length incorporated in the model was also dependent on the experimental test data. It was later found that the crack length had to be increased by 0.05-0.3 inches in the FE model to match the deflection from the experiments. This can be justified by the fact that it is very difficult to see the actual crack tip because the crack has the shape of a thumbnail as shown in the Figure 7-2 and it
can be seen that the actual crack tip is ahead of the observed crack tip. Several FE
simulations were performed to find the most appropriate crack length.

![Figure 7-2: Shape of the crack](image1)

As we are only interested in the area in the vicinity of the crack tip, we can see
from Figures 7-3 and 7-4 that mesh was made more refined near the crack tip.

![Figure 7-3: Mesh refinement near the crack tip](image2)
Finite Element Analysis

The strain energy release rate is directly related to the J-integral for a linear elastic material. The J-integral method is a powerful technique for analyzing fracture in composites. It is related to the variation in potential energy and is path independent. The J-integral method was used to find the energy release rate and hence the fracture toughness of the specimen. ABAQUS directly generates the strain energy release rate related to each contour number. The $G$ value obtained by this method will be compared with the experimental results.

Results and Discussion

Table 7-2 shows the comparison of the fracture toughness from the FE model and the experimental test for the 0D0 specimen and 90D90 specimen at room temperature.
Table 7-2: Comparison of FE and experimental Fracture toughness

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Observed crack length from experiment (in)</th>
<th>crack length added to the FE model (in)</th>
<th>Fracture Toughness (N/m)</th>
<th>FE</th>
<th>Experimental</th>
</tr>
</thead>
<tbody>
<tr>
<td>0D0</td>
<td>1.176</td>
<td>0.30</td>
<td>197.58</td>
<td>211.94</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.316</td>
<td>0.20</td>
<td>236.29</td>
<td>182.86</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.152</td>
<td>0</td>
<td>277.10</td>
<td>237.16</td>
<td></td>
</tr>
<tr>
<td>90D90</td>
<td>1.2825</td>
<td>0.25</td>
<td>383.59</td>
<td>475.41</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.3105</td>
<td>0.10</td>
<td>539.31</td>
<td>545.79</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.3645</td>
<td>0.05</td>
<td>405.67</td>
<td>428.08</td>
<td></td>
</tr>
</tbody>
</table>

From the results we can see that the FE results are comparable to the experimental results when the crack length is increased by a certain amount. It is also observed that the magnitude of the compensated crack length decreases with the increase in the observed crack length.

Table 7-3 shows the percentage difference in the average fracture toughness between the FE model and experimental data.

Table 7-3: Percentage variation of Fracture Toughness

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fracture Toughness (N/m)</th>
<th>Percentage Difference (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>FE model</td>
<td>Experimental</td>
</tr>
<tr>
<td>0D0</td>
<td>236.99</td>
<td>210.66</td>
</tr>
<tr>
<td>90D90</td>
<td>443.46</td>
<td>483.09</td>
</tr>
</tbody>
</table>
CHAPTER 8
CONCLUSIONS

Double Cantilever Beam Tests were performed at room and cryogenic temperatures on composite laminates to investigate the effect of cryogenic temperature on the fracture toughness. Neat resin fracture toughness was also measured using CT specimens at room and cryogenic temperatures. A cryogenic chamber was designed and fabricated for the in-situ testing of composites at cryogenic temperature. Different types of DCB composite specimens of dimensions 177.8 mm × 25.4 mm (7 in. × 1 in.) with the pre-crack between, two 0° plies, two 90° plies, 0° and 90° plies, two textile plies were fabricated and tested at room and cryogenic temperatures using an MTI machine. Some of the specimens were treated with nano particles (38 nm Al₂O₃) at the ply interface, to determine its effect on the fracture toughness. Each specimen underwent four loading cycles at room temperature and two loading cycles at cryogenic temperature. The critical energy release rate, \( G_{IC} \), was calculated for each specimen using the area method. In the case of neat resin tests the stress intensity factor was calculated from the failure load and not the load-displacement diagram. The results from the room and cryogenic temperatures tests were compared. Finally, a finite element analysis was performed on the DCB specimens using ABAQUS and \( G_C \) calculated using the J-Integral was compared to the experimental results.

Comparing the \( G_C \) values of the unidirectional specimens, we find that the 90D90 specimens exhibited higher fracture toughness compared to the 0D0 specimens. Also,
both the specimens showed deterioration in fracture toughness at cryogenic temperature. The fracture toughness was least for the 90D0 specimens.

The nano-particle treated composite specimens showed an improvement in fracture toughness by nearly 97% at room temperature and approximately 53% at cryogenic temperature. Hence, the use of nano-particles to improve fracture toughness should be considered in future applications. More research needs to be done in future to optimize the quantity of the nano-particles that should be used and techniques to impregnate them into the composite.

Textile composites specimens exhibited good resistance to delamination at room temperature compared to the unidirectional specimens. The fracture toughness of the textile specimens almost doubles at cryogenic temperature. The use of textile composites for structural applications involving cryogenic conditions should be considered in future. Further research need to be done to understand the improvement in fracture behavior of the textile composites at the cryogenic temperature.

The compact tension tests result show that the epoxy matrix cracks due to thermal shock at cryogenic temperature. The fracture toughness deteriorates by nearly 60% at cryogenic temperature. For better understanding of the fracture behavior of the epoxy at cryogenic temperature the specimens should be exposed to the cryogenic temperature by slowly decreasing the temperature from room temperature.

The results of the FE model, shows good agreement with the experimental results. The fracture toughness can be predicted accurately using this model in conjunction with the experimental fracture load.
APPENDIX A
DCB ROOM TEMPERATURE TEST LOAD-DISPLACEMENT GRAPHS

The Double Cantilever Beam Test Load-Displacement graphs for all specimens at room temperature. DCB_0T0_1 indicates a DCB specimen with pre-crack between two 0° plies and the 1 indicates the specimen number. Similarly 0T0_N_9_1 indicates the first DCB specimen of the 9% nano-impregnated laminate.
Figure A-1: Load-displacement diagram for DCB_0T0_1.

Figure A-2: Load-displacement diagram for DCB_0T0_2.
Figure A-3: Load-displacement diagram for DCB_90T90_1.

Figure A-4: Load-displacement diagram for DCB_90T90_2.
Figure A-5: Load-displacement diagram for DCB_90T0_1.

Figure A-6: Load-displacement diagram for DCB_90T0_2.
Figure A-7: Load-displacement diagram for DCB_0T0_N_20_1.

Figure A-8: Load-displacement diagram for DCB_0T0_N_20_2.
Figure A-9: Load-displacement diagram for DCB_0T0_N_9_1.

Figure A-10: Load-displacement diagram for DCB_0T0_N_9_2.
Figure A-11: Load-displacement diagram for DCB_TEX_1.

Figure A-12: Load-displacement diagram for DCB_TEX_2.
APPENDIX B
DCB CRYOGENIC TEMPERATURE LOAD-DISPLACEMENT GRAPHS

The Double Cantilever Beam Test Load-Displacement graphs for all specimens at room temperature. DCB_0T0_CT_1 indicates a DCB specimen with pre-crack between two 0° plies, CT indicates the cryogenic temperature and the 1 indicates the specimen number. Similarly 0T0_N_9_CT_1 indicates the first DCB specimen of the 9% nano-impregnated laminate tested at cryogenic temperature.
Figure B-2: Load-displacement diagram for DCB_0T0_CT_1.

Figure B-2: Load-displacement diagram for DCB_0T0_CT_2.
Figure B-3: Load-displacement diagram for DCB_90T90_CT_1.

Figure B-4: Load-displacement diagram for DCB_90T90_CT_2.
Figure B-5: Load-displacement diagram for DCB_90T0_CT_1.

Figure B-6: Load-displacement diagram for DCB_90T0_CT_2.
Figure B-7: Load-displacement diagram for DCB_0T0_N_20_CT_1.

Figure B-8: Load-displacement diagram for DCB_0T0_N_20_CT_2.
Figure B-9: Load-displacement diagram for DCB_0T0_N_9_CT_1.

Figure B-10: Load-displacement diagram for DCB_0T0_N_9_CT_2.
Figure B-11: Load-displacement diagram for DCB_TEX_CT_1.

Figure B-12: Load-displacement diagram for DCB_TEX_CT_2.
APPENDIX C
COMPACT TENSION TEST LOAD DISPLACEMENT GRAPHS

The Compact Tension Test Load-Displacement graphs for all specimens at room and cryogenic temperatures. CTT_RT_1 indicates a compact tension specimen tested at room temperature and the 1 indicates the specimen number. Similarly CTT_RT_1 indicates the first specimen tested at cryogenic temperature.
Figure C-3: Load-displacement diagram for CTT_RT_1.

Figure C-2: Load-displacement diagram for CTT_RT_2.
Figure C-3: Load-displacement diagram for CTT_RT_3.

Figure C-4: Load-displacement diagram for CTT_RT_4.
Figure C-5: Load-displacement diagram for CTT_CT_1.

Figure C-6: Load-displacement diagram for CTT_CT_2.
Figure C-7: Load-displacement diagram for CTT_CT_3.

Figure C-8: Load-displacement diagram for CTT_CT_4.
LIST OF REFERENCES


BIOGRAPHICAL SKETCH

Sujith G. Kalarikkal received his Bachelor of Technology degree from the University of Calicut, Thrissur, India, in 2000 in mechanical engineering. During August 2000 to July 2001, he worked as a project associate in the Machine Design Section at the Indian Institute of Technology, Chennai, India. Since August 2001, he has been pursuing his M.S degree in the Department of Mechanical and Aerospace Engineering at the University of Florida in the area of composite technology.