CRYOGENIC TEMPERATURE EFFECTS ON PERFORMANCE OF POLYMER COMPOSITES

David Hui* and P.K. Dutta**

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* Dept of Mechanical Engineering, University of New Orleans, New Orleans, LA 70148
** U S Army Engineer Research and Development Center, CRREL, Hanover NH 03755
1. INTRODUCTION

The objective of this study is to evaluate the low temperature behavior of polymer composites down to the cryogenic temperature range. This would be accomplished by study of its behavior in several ways. First we would study the microfracture growth by observing the acoustic emission as the temperature is lowered. We would also note any damage growth by ultrasonic velocity testing applying the pulse echo method. Effects of such low temperature would then be studied by examining the shear properties by the short beam shear test, and also the fracture toughness properties over a wide range of strain rate and temperature. At present these studies are continuing. The limited data obtained from these studies are reported in this report.

2. BACKGROUND

In near future the lightweight composites will be used in the NASA re-entry vehicles. The structural systems of such re-entry vehicles must withstand rapid loading, vibration, high acceleration at take off from launching platforms in most severe environments, while internally containing liquid oxygen and hydrogen at cryogenic temperatures. Polymer composites, a multiphase material will be subjected to extremely high differential stresses within the phase components itself. In the past, careful manufacturing condition under controlled pressure and temperature in the autoclave systems provided reasonably satisfactory control of development of micro cracks during curing and subsequent applications in severe temperatures. In the new low cost, high volume manufacturing process, called the VARTM (Vacuum Assisted Resin Transfer Molding) process, control of the development of residual stress induced micro fracture will be extremely difficult. Research is needed to characterize such microstructures, monitor development and progression of the micro cracks under cryogenic temperature and service conditions, and finally assess the influence of such fracture growths on the performance of such structures by fracture mechanics studies. The current research program address some of these issues.

2.1 Failure modes

The primary concern for composites being used in the new generation of the re-entry vehicles is not only premature failure, but that they must not leak excessively even after multiple launches. Composites in space applications, whether used as rocket shell, satellite structures, or sensor platforms involve extremely high temperature and load variation at extremely high rates not only on its surface, but also through the thickness. Thick-section composites typically fail at stresses and strains that are well below the expected failure limits. Delamination is a common mode of failure. When cold, they fail with very small amount of strain, with more violence and high-energy release. This early failure is often attributed to the existence of critically sized processing and/or material defects and interfacial problems in the interphase region between the matrix and the reinforcing phase (Drzal 1983, 1986; Sottos 1990; Palmese 1992; Skourlis...
Evaluation of interphasial mechanical properties has been carried out experimentally (Sottos 1990; VanLandingham 1997) and theoretically (Palmese 1992; Chu and Rokhlin 1996). The extent of the interphase region in composites is significant (Hughes 1991). For instance, a 1 cm³ of a composite when filled with a fiber volume content of 60% contains as many as 3 million single filaments. The total area of the fiber surface is 3,400 cm². As a result, the matrix and its ability to adhere to a fiber are paramount to the effective transfer of the mechanical load in the composite (Erikson and Plueddemann 1974; Drza11983, 1986; Fishman 1991; Piggott 1991). The large surface area plays a direct role in the load transfer from the matrix to the reinforcing constituent. The way the interphase interacts with the matrix and with the fibers is quite important in determining damage initiation in composite materials and its ability to maintain sufficient impermeability to liquids and gases.

### 2.2 Strain-rate Effect and Microstructural Failure

Microstructural study by Dutta et al. (2000) of Gr/Ep composite fragments at CRREL, Hanover, NH and at the Air Force Wright Laboratory, Dayton, Ohio, has clearly shown that both temperature and the rate of loading significantly influence the interphase stress transfer mechanisms and final fracture that can influence the permeability as well as reliability in the performance of multiple launch. Reinforcing fibers and particles themselves may serve as stress raisers and lead to interfacial cracking (Eshelby 1957). Fiber-matrix debonding and cracks may significantly reduce the load transfer between matrix and the fibers and cause cracking in composites (Sottos, Li, and Agrawal 1994; Budiansky, Hutchinson, and Slutsky 1995). Interfacial damage (Keer, Dundurs, and Kiattikomol 1973; Hashin 1991; Pan, Green, and Hellman 1996) or material inhomogeneity of interphases also affects the elastic properties of composites (Jasiuk and Kouider 1993; Lagache et al. 1994; Low et al. 1995; Theocaris and Demakos 1995; Lutz and Zirnmennan 1996), the residual stresses (Jayaraman and Reifsnider 1993), and their macroscopic behavior (Tsai, Arocho, and Gause 1990; Kharik 1997; Kim and Mai 1991, 1998).

### 2.3 Residual Stress of Low-Temperature

Manufacturing of composites involves the use of a thermosetting polymeric material as the matrix phase. The polymeric matrix in the presence of a catalyst, heat, and pressure solidify through an irreversible exothermic chemical reaction (cure). Before curing, the polymer phase is a viscous fluid. It flows under pressure. As a result of curing, the polymer forms a covalently bonded three-dimensional molecular network with increasing viscosity and gel formation. The flow ceases at this stage but reactions continue to form a tightly cross-linked structure with characteristics of glassy solid (Rosen, 1993). The problem in composite cure is the problem of controlling the reaction exotherm and heat transfer so that uniform cure and minimum residual stresses are achieved (Bogetti et al. 1992). During the curing stage as the chemical reactions proceed residual stresses are developed with progressive changes in modulus and thermal expansion coefficients, and volume shrinkage of the resin. At the microstructural level
influence of low temperature on the induced stresses at the matrix/fiber interfaces, within
the matrix, and in the interlaminar layers has been analyzed and experimentally
investigated by many authors (Jones 1975, Lord and Dutta 1988, Dutta 1988, Dutta and
Lampo 1993). These results have shown that a difference between the curing temperature
and the operating temperature may be as large as 200°C (392°F) in usual cold
environment, and the residual stresses may be sufficiently large to cause microcracking
within the matrix and matrix/fiber interfaces. The computation of residual stresses using
the Tsai and Hahn method (1975) for unidirectional composites in longitudinal direction
as:

\[ \sigma_{mL} = (V_f E_f E_m)(\alpha_f - \alpha_m)(T - T_0)/(V_f E_f + V_m E_m) \]  

shows that at the cryogenic temperature of -180°C the matrix stresses could be as high
as 12000 psi. Thus, the large residual stresses induced at lower temperatures become
potentially damaging for polymer matrix composites with curing temperature
environment. The damage may begin with the formation of microscopic cracks in the
matrix or at the fiber/matrix interface. When these cracks develop to a certain density
and size, they will tend to coalesce to form macroscopic matrix cracks (Wang, 1986).
Transverse matrix cracking in composites affect stiffness, strength, dimensional
stability, and fatigue resistance.

### 2.4 Fracture Toughness of Fiber/Matrix Interphase

An understanding of the failure process at the interphase at the cryogenic

temperatures is essential to develop optimal performance capability at those
temperatures. For this, one must closely examine the polymer matrix and its interaction
with the interfacial surfaces (Wool 1995; Hrivnak 1996). Many studies, as reviewed
by Cantwell and Morton (1991), have concluded that composites are particularly
susceptible to damage by delamination, which is particularly dangerous because it is
often not visible from surface. The composites property measured in the fracture
mechanics study of the resistance to delamination is the critical energy release rate, or
fracture toughness, which is a measure of the energy consumed during the creation of
unit area of fracture surface during delamination. Three modes of crack loading can
occur, namely mode I (tensile opening), mode II (in-plane shear) and mode III (out-of-
plane shear). In practice, modes I and II and combinations of mode I and II are the most
important.

Test methods for measuring the interlaminar fracture toughness (\(K_C\)) at slow rate
in mode I, II and mixed I/II are well established and several standards exist for mode I
(ASTM D5528, ASTM E399, ISO CD 15024 version 97-02-24, and JIS K 7086 of
1993). Various test methods are currently being pursued for the other modes. However,
currently no appropriate high rate-loading test, especially under cryogenic conditions
exists, and all previous attempts to extend the slow speed test methods to high rates
have met with significant obstacles (Blackman and Williams 1998). The first obstacle is
in experimental test equipment to be capable of rapidly accelerating the test specimen

and then accurately recording the forces applied and the deformation occurred. Second, the dynamic effects are invariably induced at the high rate tests and it is critical that these effects are carefully considered, and accurately accounted for, if accurate and valid $K_C$ values are to be measured. Indeed, this probably accounts for the conflicting nature of some of the test results reported in the literature. For example, Smiley and Pipes (1987) pointed to very large reductions in the values of $K_{IC}$ of $K_{IIIC}$ for brittle epoxy as well as for thermoplastic polyether etherketone (PEEK) composites, as the test rate was increased from a few mm/min to about 1m/s. On the other hand, Beguelin et al (1991) reported mode I results of a PEEK matrix carbon composites only a small reduction in the value of $K_{IC}$ as the test rate was similarly increased. In a third study by Aliyu and Daniel (1985) on similar materials, increasing followed by decreasing values of $K_{IC}$ was reported as the test rate was increased. The differences in experimental results reported were further highlighted in a recent review by Cantwell and Blyton (1998). Their review indicated that the rate sensitivity of the composites was dominated by the toughness of the matrix, with brittle matrix composites exhibiting much less of a rate effect than tough matrix composites.

3. EXPERIMENTAL STUDY

We have undertaken the experimental study to determine how the composites behavior change as we approach the cryogenic temperature range by performing five series of tests: (1) Shear response at cryogenic temperatures, (2) Microfracture growth monitoring by acoustic emission as the temperatures are reduced, (3) Modulus degradation evaluation by ultrasonic wave transmission (pulse-echo) method, (4) Shear property degradation by short beam shear property evaluation, and by (5) fracture properties over a wide strain rate and temperatures.

3.1 Shear Response at Cryogenic Temperatures

These tests were performed to study the effects of temperature on the interlaminar shear resistance. The range of temperature was varied from $-100^\circ$C to $80^\circ$C. The results showed a drastic reduction of interlaminar shear strength with the temperature rise from $50^\circ$C to $80^\circ$C. However the increase in the shear strength with decreasing temperature is more gradual. The test specimens were prepared from a pultruded glass fiber reinforced composite square bar of 0.5 in. $\times$ 0.5 in. section. From this stock the specimens were machined with the fibers oriented in longitudinal direction. The rectangular specimens had a dimension of 1.5 in $\times$ 0.5 in $\times$ 0.25 in. Figure 1 shows a typical specimen. These specimens were then tested for interlaminar shear strength using the ASTM D2344-84 (The ASTM standard D2344-84 specifies the span to thickness ratio of 5. In our case the ratio is 3.26.

3.1.1 Testing
The tests were performed in an environment chamber which could be cooled with liquid nitrogen, or heated by a heating coil. The cooling system involves a supply of liquid nitrogen from the commercially available liquid nitrogen tank through a control valve which releases the evaporated liquid nitrogen into the environment chamber. A feedback loop of temperature sensed by a thermocouple controls the release of liquid nitrogen so that the temperature inside the chamber is maintained steady within +/- 1 °C.

The chamber could also be heated to a higher temperature by the heating coil mounted inside the test chamber. Figure 2 shows the test chamber with the heating coil. Again a feed back loop control using the thermocouple controls the temperature of the chamber.

For testing at temperatures other than the room temperature the specimens were soaked at that temperature for a minimum of 45 minutes. The short beam shear test was performed in a MTS machine using the Wyoming test fixture for three point bending.

3.1.2 Test Results

Table-1 shows the results of the test. Figure 3 shows the variation of the shear strength with temperature. Figure 4 shows the force displacement curves at different temperatures. Figure 5 shows the displacement at peak load at different temperatures.

3.1.3 Discussion

From Figure 3 we see that there is a drastic decrease of shear strength with increasing temperature from 23°C to 80°C. Possibly the higher temperature softened the matrix of the composite. From 23°C to -100°C we observe that the shear strength increases linearly. However this increase is more gradual. The increase in strength with temperature reduction can be modeled by the following equation:

$$S_H = -15.7494 T + 8935.36$$

Where $S_H =$ Shear Strength (psi), $T =$ Temperature (°C)

Figure 4 gives the force-displacement curves. Figure 5 shows that the displacements at peak load decreases with decreasing temperature. Also the peaks are not sharp at higher temperatures (Figure 4). The sharp peaks at lower temperature denote brittleness of the material. We conclude that (1) Shear Strength decreases drastically with increasing temperature. (2) At low temperatures shear strength increases almost linearly. (3) The material becomes more brittle at lower temperatures as seen by reduced deflection and sharp peaks.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>No. of Samples</th>
<th>Shear Strength $S_H$ (psi)</th>
<th>Displ. at peak load (in)</th>
<th>Standard Deviation</th>
</tr>
</thead>
</table>

Table 1. Temperature effect on Shear Strength in Quasi Isotropic test
<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>-100</td>
<td>6</td>
<td></td>
<td>10510.8</td>
<td>0.020</td>
<td>349.7</td>
</tr>
<tr>
<td>-5</td>
<td>6</td>
<td></td>
<td>9014.6</td>
<td>0.021</td>
<td>365.9</td>
</tr>
<tr>
<td>23</td>
<td>6</td>
<td></td>
<td>8309.8</td>
<td>0.022</td>
<td>288.0</td>
</tr>
<tr>
<td>50</td>
<td>6</td>
<td></td>
<td>4926.2</td>
<td>0.031</td>
<td>195.0</td>
</tr>
<tr>
<td>80</td>
<td>6</td>
<td></td>
<td>2721.8</td>
<td>0.051</td>
<td>96.2</td>
</tr>
</tbody>
</table>

Figure 1 The view of interlaminar shear test specimen

Figure 2 The test chamber with the heating coil
Figure 3. Variation of the shear strength with temperature

Figure 4. Force vs displacement at different temperatures

Figure 5. Variation of displacement at peak load with temperature
4. CRYOGENIC FRACTURE TOUGHNESS

The plain strain fracture toughness is the stress concentration at the crack tip under conditions of plane strain, and is regarded as the basic material property. The fracture toughness was determined by applying bending load to the notched specimens as shown in Figure 6.

4.1 Specimen Dimensions

The material of test specimen is a commercial pultruded composite product, which uses E-glass fiber and isophthalic polyester resin. The details of the test material are given in Table 2 and Figure 7.

![Figure 6 Test configurations for the Fracture Toughness Test](image)

*Figure 6 Test configurations for the Fracture Toughness Test*

<table>
<thead>
<tr>
<th>Table 2 The specification of test material</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>E-glass FRP composite</strong></td>
</tr>
<tr>
<td>Used Process: Pultrusion method</td>
</tr>
<tr>
<td>Reinforcing Fiber: E-glass</td>
</tr>
<tr>
<td>Matrix: Polyester</td>
</tr>
<tr>
<td>Density: 0.071 lb/in³ (0.971 g/cm³)</td>
</tr>
<tr>
<td>Volume Fraction of Fiber: 0.593</td>
</tr>
<tr>
<td>Notch angle: 90 degree</td>
</tr>
<tr>
<td><strong>Dimensions:</strong></td>
</tr>
<tr>
<td>Length: 1.511in (38.38mm)</td>
</tr>
<tr>
<td>Width: 0.494in (12.55mm)</td>
</tr>
<tr>
<td>Thickness: 0.521in (13.23mm)</td>
</tr>
</tbody>
</table>
4.2 TESTING

The specimens were tested both quasi statically and dynamically using the ASTM D70 three point-bending test.

4.2.1 Quasi-static Tests

Quasi-static tests were performed on the glass fiber specimen of 90-degree fiber orientation at room temperature (23°C) and at low temperature (-30°C) with a servo controlled hydraulic testing machine driving the loading platen at a speed of 0.01 inch/min. An environmental chamber was used for all low temperature tests. The chamber was cooled with chilled nitrogen gas slowly vented through a regulating valve controlled by a temperature sensor near the test specimen. Tests were performed only when a stable temperature was established for about 15 minutes within the chamber. Load and displacement were recorded using the load cell and the LVDT transducer attached to the testing systems and the data were automatically transferred to the same digital data acquisition system.

4.2.2 Dynamic Test

4.2.2.1 Experimental Approach

We performed a study of the interlaminar Mode I fracture toughness, $K_{IC}$, of unidirectional composites at room and cryogenic temperatures using a modification of Split Hopkinson Compression Bar Apparatus (SHBA). By using SHBA we had overcome the difficulties encountered by the researchers in measuring loads and deformation while using the traditional servo hydraulic machines at rate above 1 m/s. SHBA would allow a rate up to 100 m/s. The past researchers did not take into account the problems of low temperatures associated with high rate loading.
Our approach to measure the cryogenic high-strain-rate fracture toughness for composites is new and unique. The details of the proposed system, a preliminary model of which has already been developed, is shown in Figure 8(a) and 8(b). The typical stress waves used for computation of the force-displacement curves are shown in Figure 8(c). As shown in this Figure, we modified the SHBA interface to represent a three-point loading system of a single notch prismatic composite sample, which is mounted in between the two interfaces of the SHBA. The intact and the fracture samples are shown in Figure 9. Figure 10 shows the sample in the SHBA set up, and Figure 11, the cryogenic cooling with liquid nitrogen. The entire fracturing process would be performed in a cryogenic chamber built around the SHBA interfacial impact zone. We plan to measure the crack opening force and the corresponding displacements exactly the same way as we do in a standard Hopkinson Bar by integrating the incident, reflected and transmitted strain waves in each bar (Dutta 1987). The system will allow $K_{IC}$ to be measured with samples in which fiber orientation is parallel to the notch axis.

(a) Split Hopkinson pressure bar system schematic

(b) CRREL Split Hopkinson Bar apparatus
4.2.2.2 Analytical approach

For a specimen in three point loading (Figure 3), $K_{IC}$ is calculated using the expression of Brown and Strawley (1966),

$$K_{IC} = \frac{6M}{Bd} \sqrt{aF(a/d)}$$

where $F(a/d)$ is the finite width correction factor given by $F(a/d) = 1.99 - 2.47(a/d) + 12.97(a/d)^2 - 23.17(a/d)^3 + 24.80(a/d)^4$.

where $a = \text{crack length, } d = \text{specimen depth, } M = \text{ultimate bending moment, } P = \text{applied load, } l = \text{shear span, and } B = \text{specimen width.}$

4.2.3 RESULTS AND DISCUSSION
Quasi static test at room temperature gave the $K_{IC}$ value of 6.9 ksi√in. The batches for dynamic test of the notched samples (Figure 9(a)) were tested at different temperatures. A representative specimens which failed under the dynamic tests is as shown in Figure 9(b).

![Sample before test](image1.png) ![Sample after test](image2.png)

*Figure 9 GFRP samples before and after dynamic test*

![Loading position of the specimen](image3.png)

*Figure 10 Loading position of the specimen*

![The chamber used to keep the test specimen cold](image4.png)

*Figure 11. The chamber used to keep the test specimen cold*
It is clear that there are multiple cracks along the fracture path in the test specimens. No significant differences in the crack patterns were observed between high temperature and low temperature fracturing. The force-displacement characteristics and then the energy absorbed to develop the crack were determined. The absorbed energy versus time plot at different temperatures are shown in figure 12.

From Figure 12 we observe that the energy absorbed within the system is higher at –30 C and room temperature as compared to –50 C and 80 C.

The force-displacement curve showed a dramatic increase in stiffness and brittleness of both specimens in dynamic fracturing. The displacement at the peak force was assumed as the fracture initiation point. Because of the visco-elastic nature of the composite matrix, we do not always find any sharp failure point. At this point, there is considerable amount of scatter for the peak force. This is expected in Extren, which is a pultruded composite in which fiber volume percentage is low and many resin-rich areas occur. The peak force was used to calculate the fracture toughness, $K_{IC}$. The calculated values of $K_{IC}$ are summarized in Table 3 and plotted in Figure 13.
Table 3: Summary of result of fracture toughness

<table>
<thead>
<tr>
<th></th>
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</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>20.29</td>
<td>0.01259</td>
<td>1321.0</td>
<td>6.114</td>
</tr>
<tr>
<td>50</td>
<td>12.38</td>
<td>0.01217</td>
<td>1398.7</td>
<td>6.000</td>
</tr>
<tr>
<td>24</td>
<td>19.82</td>
<td>0.00630</td>
<td>1359.7</td>
<td>6.900</td>
</tr>
<tr>
<td>-30</td>
<td>14.03</td>
<td>0.01349</td>
<td>1126.6</td>
<td>5.307</td>
</tr>
<tr>
<td>-50</td>
<td>11.82</td>
<td>0.00821</td>
<td>893.4</td>
<td>5.146</td>
</tr>
<tr>
<td>-100</td>
<td>13.61</td>
<td>0.00515</td>
<td>1476.4</td>
<td>4.381</td>
</tr>
<tr>
<td>-150</td>
<td></td>
<td>0.00561</td>
<td>1262.5</td>
<td>3.746</td>
</tr>
</tbody>
</table>

$y = 6.1431e0.0029T$  
$R^2 = 0.9599$

Figure 13 Fracture Toughness at different Temperatures

The $K_{IC}$ values were plotted against the test temperatures. It is seen from the graph that fracture toughness varies with temperature. $K_{IC}$ increase with increase in temperatures. As
stated before, many factors contribute into the value of $K_{IC}$ for a given material. The strain rate data is summarized in the Table 4.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Avg. Maximum Force (lbs)</th>
<th>Strain</th>
<th>Strain Rate (Strain/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>1320.9</td>
<td>3591.75</td>
<td>49.885</td>
</tr>
<tr>
<td>50</td>
<td>1398.6</td>
<td>3803.03</td>
<td>52.819</td>
</tr>
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<td>24</td>
<td>1359.6</td>
<td>3696.94</td>
<td>51.346</td>
</tr>
<tr>
<td>-30</td>
<td>1126.6</td>
<td>3063.24</td>
<td>42.545</td>
</tr>
<tr>
<td>-50</td>
<td>893.4</td>
<td>2429.15</td>
<td>33.738</td>
</tr>
<tr>
<td>-100</td>
<td>1476.3</td>
<td>4014.31</td>
<td>55.754</td>
</tr>
<tr>
<td>-150</td>
<td>1262.5</td>
<td>3432.73</td>
<td>47.676</td>
</tr>
</tbody>
</table>

It is concluded that the low temperature reduces the fracture toughness of the composites. However fracture toughness increase with increase in temperature with the average strain rate of $40 \sim 50$ strain/sec. The strain rate was calculated by assuming that the maximum force reached the value at approximately 72 microseconds. The maximum value of force was considered applied to load a bending. Low temperature influence the energy absorption characteristics of the GFRP, i.e. it absorbs less energy at low temperatures as compared to high temperatures. After the fracture of the specimen, multiple cracks were observed along the fracture path but it was found that there is no significant difference in crack pattern between low and high temperatures.

5. Acoustic Emission Study

We hypothesized that the growth of microcracks in composite are likely to happen when the temperature is lowered to the cryogenic range.

5.1 Procedure for Acoustic Emission test

The procedure for the acoustic emission test is described below.

A 50-ply carbon test sample of size 4”length, 0.75”width and 0.25” thickness is taken and a transducer is placed on the sample. Vacuum grease is used as a fluid between the sample and the transducer to ensure the proper transformation of signals from sample to transducer. The transducer is then clamped tightly (Figure 14) with sample so that it does not move during the test. A thermocouple is clamped with specimen to note the
temperature of sample. The thermocouple is connected to a datalogger, which reads the temperature of specimen for every 2 or 4 sec and sends the data to a computer. The software used for this is called CAMPBELL SCIENTIFIC software.

Figure 14 Clamping of the transducer for acoustic emission test

The specimen is kept in a test chamber which was cooled by liquid nitrogen. The transducer collects the data of the time and accumulated events whenever an event occurs on the specimen (i.e. when ever the specimen has micro cracks).

The software used to record the events data is MISTRAS software of PHYSICAL ACOUSTIC EMISSIONS CORP. The sample is subjected from room temperature to low temperatures (23, -5, -50, -100, and -150°C) and kept at each of these temperatures for approximately 10 minutes, then the temperature was raised from -150°C to room temperature. The data of time, events and temperature is recorded throughout.

Graphs were plotted between the time vs temperature and time vs accumulated events (Figure 15). We observe that the rate of events increases from room to low temperatures but from -150°C to room temperature, the rate of increase of events is very low. This indicates that the cracks produced were high during the first half of the test and less during the second half of the test.
5.2 Results

Maximum no. of events counted for each sample is given in Table 5.

*Table 5 Maximum no. of acoustic emission events*

<table>
<thead>
<tr>
<th>Samples</th>
<th>Temp (°C)</th>
<th>Max no. of events on lowering of temperature</th>
<th>Max no. of events on warming to room temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-161</td>
<td>1492</td>
<td>---</td>
</tr>
<tr>
<td>2</td>
<td>-154</td>
<td>1930</td>
<td>33</td>
</tr>
<tr>
<td>3</td>
<td>-150</td>
<td>1280</td>
<td>186</td>
</tr>
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<td>4</td>
<td>-150</td>
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</tr>
<tr>
<td>5</td>
<td>-150</td>
<td>3495</td>
<td>249</td>
</tr>
</tbody>
</table>

6. Ultrasonic test evaluation at cryogenic temperature

6.1 Liquid nitrogen immersion test
In the first series of tests composites samples were immersed in liquid nitrogen (Temperature, –196°C) for different durations. The samples, immediately (within 5 to 10 seconds) after removal from the liquid Nitrogen bath were tested for determining the ultrasonic velocities (Figure 16(a)). Figure 16(b) shows the velocity measurement by the ultrasonic technique. The same samples were again tested for velocities when they achieved the room temperature after a day. Table 6 shows the immersion times vs velocity and Young’s modulus. The results are also shown graphically in Figure 17. Figure 18 and 19 shows the Young’s modulus of each specimen.

Table 6 Immersion and rapid test data

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Immersed Time (min)</th>
<th>Velocity at cryogenic temp immediately after removing from Liq Nitrogen (m/s)</th>
<th>Velocity at room temperature long after removing from Liq Nitrogen (m/s)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>60</td>
<td>2559</td>
<td>2248</td>
</tr>
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<td>45</td>
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<td></td>
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<td></td>
<td>5</td>
<td>2464</td>
<td>2368</td>
</tr>
<tr>
<td>Room (24°C)</td>
<td>None</td>
<td>2376</td>
<td>2376</td>
</tr>
</tbody>
</table>

(a) Ultrasonic testing machine
(b) pulse-eco signals in the ultrasonic testing

Figure 16 Ultrasonic testing technique

Figure 17 Change of velocities with different immersion times in Liq Nitrogen. The point at right shows the room temperature velocity data.
Figure 18 The Young’s modulus of CFRP specimens just after the immersion test

Figure 19 The Young’s modulus of CFRP specimens that kept it at room temperature for 2 days after the immersion test
6.2 Room and Liquid Nitrogen Comparison Test

In the second series of tests one composite sample was first tested for the velocities at room temperature. And then the sample was immersed in liquid nitrogen, and kept immersed for one hour. Then the sample was taken out and tested for acoustic emission till the specimen achieved room temperature. The specimen was again tested for velocity. Figure 20 shows the acoustic emission results. Figure 21 shows the results of the velocity and Young’s modulus of CFRP sample after immersion test.

Figure 20 The acoustic emission results

Figure 21 The results of velocity and Young’s modulus after immersion test
6.3 Ultrasonic evaluation of cryogenically exposed samples

In this series of tests ultrasonic velocities were determined for all samples, which were subjected to the acoustic emission tests. The results are given in Table 7. Note that there is a small change in the velocities after exposure to the liquid nitrogen, which can account for the microcrack generated emissions from the composites when cooled.

Table 7 Velocities after the exposure to cryogenic temperatures

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Velocity (m/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2713</td>
</tr>
<tr>
<td>2</td>
<td>2721</td>
</tr>
<tr>
<td>3</td>
<td>2721</td>
</tr>
<tr>
<td>4</td>
<td>2713</td>
</tr>
<tr>
<td>5</td>
<td>2713</td>
</tr>
<tr>
<td>Average</td>
<td>2716</td>
</tr>
</tbody>
</table>

Note that before immersion, the room temp velocity was 2737 m/sec (average of 3 samples). The average velocity after the exposure was 2716, a reduction of 21 m/sec for the damage growth.

7. CONCLUSIONS

Cryogenic exposure of graphite epoxy and other composites show that residual stresses at the fiber matrix boundary and at the interlaminar zone increase significantly. The increase may be sufficient to develop microcracks, which can be monitored by acoustic emission. Degradation of interlaminar shear strength is not sufficient, rather the low temperature increased the strengths. However, the fracture toughness decreased with lowering of temperatures. The degradation was also noticed by decrease in ultrasonic velocities.

The results presented here are mostly preliminary. As the testing are going on, more data are expected and characterization at the cryogenic temperatures will be more certain.

7. ACKNOWLEDGMENT

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Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Aeronautics and Space Administration.

8. REFERENCES


