Cryocycling and Mechanical Testing of CFRP for X-33 Liquid H_2 Fuel Tank Structure

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ABSTRACT

Driving down the cost of space payload has become an important topic in the aerospace industry. This paper addresses issues involved with introducing composite materials into space vehicles' fuel tanks that must endure several cycles of thermal loading at both extremes while maintaining structural integrity. The material studied in this project was IM7-977-2, a graphite fiber with toughened epoxy, which is being used to construct the X-33 by the Lockheed Martin. This research explores the degradation of the composite's mechanical properties due to cryocycling, and attempts to establish the mechanism behind the degradation through a non-destructive analysis.

INTRODUCTION

In today's technologically advancing and communications driven society, a need for cheaper access to space has arisen. Currently, it costs $10,000 to transport a pound of payload into orbit, which hinders the feasibility of most large ventures. Programs such as the international space station and the planetary probes have had their capabilities truncated because their designs were so weight driven. In response to an initiative put forth by NASA, the Lockheed Martin Skunk Works is currently developing a single-stage-to-orbit reusable launch vehicle (SSTO RLV) to resolve this issue. This vehicle, the X-33, capitalizes on many new and rediscovered technologies to reduce launch cost of payload by an order of magnitude. This SSTO vehicle uses a lifting body base to supply the majority of the lift, linear aerospike engines to provide thrust at a high efficiency, a metallic thermal protection system, and fabrication mostly of composite materials.

By combining these new technologies, many unexplored territories are entered. The fuel tanks for the linear aerospike engines provide an example of this. These engines operate using hydrogen mixed with an oxidizer, which needs to be stored at cryogenic temperatures around 20K. To minimize structural weight, graphite composite was selected as the material for the fuel tanks. This combination raises the question of how composite materials behave at very low temperatures; a field in which very little research has been done. Problems can also arise due to thermal cycling loads introduced by the flight cycle, as seen in **Figure 1**.

The fuel tanks, which originally are at 20K due to the cryogenic fuel, can quickly reach above 400K during re-entry while the vehicle is re-entering the atmosphere at mach 15 with empty tanks. Theoretically, this large change in temperature will result in cracks initiating through the tanks, as seen in **Figure 2**. This is expected of composites since they are composed of several ply layers that expand in a dominant direction, producing a high amount of strain energy that can easily be relieved with the formation of cracks. When this problem is coupled with cryogenic operating temperatures, the feasibility of a reusable launch vehicle becomes questionable.

A related work published [1] by McDonnell Douglas for the DC-XA RLV program discusses the cost advantages of composite tanks and their gas permeability. The conclusion of that paper was that the composite tanks provide over 30% savings in weight for the same strength. However, this study only analyzed the properties of the composite at held cryogenic temperatures, and did not account for extreme thermal cycling strains.

RESEARCH PLAN

Approach

This project explores how the thermal cycling of composite specimens at cryogenic temperatures affects their material properties and failure behavior. The attributes used for this comparison are the Young's Modulus, Poisson's Ratio and ultimate stress, as well as the material's general response to tension and compression. Groups of samples were thermally cycled pre-specified numbers of times in a custom-built cryogenic dewar, and then tested for strength and stiffness. Furthermore, an optical micro-crack analysis and permeability tests were performed to help detail the underlying mechanisms behind these material property degradations. Finally, the data was compiled into graphs to assist in predicting the life of this composite material under cyclic thermal loading.

Test Matrix

The Lockheed Martin Skunk Works (LMSW) has provided TELAC with 10 – 11"x11" panels of IM7-977-2 toughened graphite epoxy composite identical to the material which will be used on the X-33's fuel tanks. These .08" thick plates were then machined on a water-jet to 40 –9"x2" samples to be tested. The test matrix is shown in **Figure 3**. In the thermal chamber, groups of five samples were cycled at a time. Each group was cycled a desired number of times, representing multiple flights of the vehicle. Three samples from each group were used for mechanical testing, a single sample was necessary for optical observations, and a fifth specimen was cycled to replace any tensile specimens that broke at the grip. These specimens also were x-rayed to locate damage.

Cycles	Tensile	Spare	Optical
0	3	1	1
1	3	1	1
2	3	1	1
5	3	1	1
10	3	1	1

Figure 5: Test matrix for CFRP samples

Cryocycling

A customized thermal cycling dewar, seen in **Figure 4** has been designed with the aid of the Cryogenics Laboratory at MIT, and was fabricated by Precision Cryogenics, Inc. A more detailed figure can be seen in **Appendix A**. This chamber was designed to simulate the operating environment of the X-33's composites fuel tanks. A single cycle as demonstrated in **Figure 5**, consisted of a cool-down from 300K to 20K, a heat-up to 400K, and then back to 300K, thereby simulating one flight cycle of the X-33. Both the heating and cooling rates were fixed at 14K/min as specified by Lockheed, and the target temperatures was held for 5 minutes to guarantee that the samples had reached the same temperature as their environment. Expanding liquid nitrogen was used to pre-cool the specimens to about 80 K, after which liquid helium was dribbled in manually to reach the target temperature. For heating, first compressed helium was passed through a pipe heater to avoid the problems of accidentally freezing air, and then above 150 K shop air was used to warm the samples by forced convection. Both of these gasses were first passed through a wall-mounted flowmeter to ensure a proper flow rate (8 CFPM for air and 2 for He) so not to burn out the heating elements. The temperature, with the exception of the LHe flow, was controlled by an electronic cryostat custom-built by Eurotherm. It used an E thermocouple probe that was bolted on the rack next to the samples in order to determine temperatures and rates. It was also necessary to mount a K thermocouple on the bottom of the chamber to aid in LN_2 detection.

Mechanical Testing

After being cycled, the groups of samples were mechanically tested in tension until destruction in order to determine their material properties. This test, as well as the strain gauging technique, adhered to the methods outlined in the TELAC manufacturing notes [2], and were performed to ASTM standards. For these tests, as seen in **Figure 6**, the samples were mounted vertically in a MTS servo-hydraulic testing machine, and pulled hydraulically with stroke control with large steel grips. Two perpendicular strain gauges were mounted on the samples, which read the strain in the X and Y directions, and a load cell measured the stress applied. From this data, the tensile stiffness and Poisson's Ration could be calculated. Then when the specimen failed, that ultimate strength was also recorded. The final outcome of these experiments was placed into a spreadsheet to calculate the Young's Modulus and Poisson's Ratio versus number of cycles exposed.

Optical Analysis

One sample from each set of cycled specimens was reserved for optical testing. The goal of this analysis was to progressively observe the mechanism of material property degradation of the coupons as the cycles were increased. These untested samples had one edge polished thoroughly with increasingly finer grits of sandpaper, and then finally smoothed out on a buffing wheel. High-resolution microscopes were used to count average crack densities for each set of cycles, as demonstrated in **Figure 7**. These cracks were to appear as slots in the otherwise smooth surface of the matrix, seen in the transverse plies. Then a crack density was to be calculated and compared to the number of cycles exposed, as well as to their corresponding strengths and stiffness. The second non-destructive test used to analyze the specimens was x-ray observation. The spare samples were polished slightly to remove any excess epoxy, and then wiped with an x-ray sensitive die penetrant along their sides. The samples were exposed to x-rays for 3 seconds, and the images were captured on a 550 black & white Polaroid film backing.

Permeability

Gas permeability testing determines the relative porosity of a material by applying a back pressure of a gas on a sample and measuring the pressure loss over time. The permeability of a material correlates with the crack density since a high number of cracks will produce a larger flow path through the thickness of the material. However, even severe cracking in interior plies will not allow any flow if the surfaces are unblemished, and therefore may go undetected. Thus, this test was used in to complement the X-ray observations.

For these tests, eight 1¼" diameter circular specimens were water-jetted from the IM7, four of where were cryogenically cycled 7 times, and the other four left as controls. The TELAC gas permeability chamber is shown in **Appendix B** in exploded view. The selected sample was first placed between two rubber washers which have been sealed with a thin layer of Dow Corning High Vacuum Grease, being careful not to contaminate the test areas of sample. Next the top plate was bolted tightly in place, and a multimeter was connected to the transducer input leads to tune the voltage source to 12.00V. After the nitrogen canister was opened and adjusted to 70 Psi, the needle valve was then opened to allow gas to enter the testing chamber. The multimeter was then connected to the transducer output leads to record the output voltage. Finally the needle valve was closed tightly, and data points were taken as necessary.

This procedure was conduced on a total of five samples: one aluminum control, two uncycled IM7 samples and two thermally cycled IM7 samples. The tests showed no significant change in pressure in both the control and the test specimens over time periods of as high as two week. These results indicate there is no through-thickness crack damage due to thermal cycling in IM7, qualifying it as suitable selection for a fuel tank material.

DATA REDUCTION

After all the data was collected, spreadsheets were formatted on Microsoft Excel. This program has a data reduction package, which will linearly regressed all the data, and provide reliability statistics such as the standard deviation and the t-value. The values considered were extracted from the most linear section of the data, from 0 to 20 KSI. The final results are in the sections below, presented as 3 plots all versus the number of cycles. These show the reduced properties of the Young's Modulus in tension, Poison's Ratio, the Ultimate Strength for tension. Additionally, there was supposed to be a 4^{th} plot versus cycles of average crack density, as well as the crack density plotted against both the regressed moduli and the observed ultimate strengths on the same plot. This was to show the correlation between the cracks and the corresponding material properties. However, since no cracks were observed, these graphs were not generated. All of these plots are lines fitted to the data, with calculated standard deviation boundaries around them.

These experimental plots were also compared to theoretical plots formulated from TELAC cracking codes and data provided by Lockheed. A few different codes were used at TELAC, some provided by Prof. McManus, and others by different graduate students. The predicted pre-cycled results for all of these modes came out to within 1% of our actual control data, thereby verifying the quality of our setup. These numbers were also within 1% of the Lockheed data, which gave them confidence that this project would produce usable results. Accurate thermal cycling models were unavailable though, due to lack of appropriate data from the proprietary Lockheed material.

DISCUSSION OF RESULTS

Young's Modulus

The control data for this composite yielded an E of around 10.1 MSI, which is an average value for a mid-range graphite-epoxy. This is found from the slope of the stress-strain regressed curve. As thermal cycling is introduced to the laminate, one would expect for the value of young's modulus to decrease if cracks were forming, since the bonds holding the material together are breaking apart effectively making it less stiff. As seen in **Figure 8** below though, no reasonable trend is detectable in the data. The fitted line slopes slightly positively, although this is within the error bars of the data. This is the first evidence that there may not be any crack growth up until the 10 cycles tested.

Poisson's Ratio

From the data collected during the tensile tests, the ratio of the two recorded stains for the control data gave a ν of about 0.46. If cracks were to propagate due to thermal stress, one would expect this the value of ν to increase as the fibers are able to move around more freely without having to strain the matrix as well. Again, **Figure 9** below shows that there is no significant trend across the cycles in the Poisson's Ratio, and in fact a slightly large variance in results, bound between .4 and .6 mostly. This graph also suggests though that there is no evidence of cracks.

Ultimate Stress

The ultimate stresses were the recorded values of the load at the instant the specimen failed during tensile tested, adjusted in terms of pounds per square inch. The initial value found for the control specimens was 150 KSI, which is on the same order of strength as many alloys of titanium or steel. Surprisingly, this is about the same ultimate strength as AS4-3501-6, a much cheaper composite. The reason for this is because the toughened epoxy system in the IM7 makes it more durable to repeated loading and thermal cycling, while the ultimate strength is fiber dominated, mostly by the fibers running in the direction parallel to the loading. For this reason, it is also expected that if cracks do initiate, the ultimate strength of the composite should remain the same. Even if there were no epoxy left, the dominant fiber bundles would still have the same failure stress. The recording of the ultimate stress was performed mostly for material characterization and to verify that there were no unexplainable results inconsistent with the above theory. As seen in **Figure 10** below, all of the ultimate stresses captured are tightly bound around 150 KSI, and therefore confirms that the thermal cycling is not affecting the individual fibers in any unexpected way, such as thermally shocking them into different material properties.

Crack Density

After the mechanical testing and permeability results, there was strong evidence to support the theory that there were no cracks present after the 10 thermal cycles. Therefore it was crucial to perform a very methodical optical search for cracks for a final confirmation. Several different techniques were used to look for the cracks or delaminations, and many opinions were taken from experienced graduate students, although none were found under 120x magnification. From this view, individual fibers could be seen, but no gaps were apparent in the surrounding matrix. To even further investigate this finding, a series of x-rays were taken of the samples. Again, no cracks could be seen, so two samples of AS4 with similar layups were placed under the x-ray for comparison. **Figure 11** shows thermocycled samples of AS4 and IM7 side by side. As seen in the picture, the AS4 sample has distinct cracks running in the 45° orientation, while the IM7 sample shows nothing but the fluid on the surface. This leads to the conclusion that there are no cracks of detectable size in any of the Lockheed samples. These results were also confirmed by infrared thermography.

CONCLUSION

After reviewing all of the data, the conclusion was that the advanced composite IM7-977-2 does now exhibit any crack growth after 10 cycles of the X-33 flight, and is a suitable material for its chosen task. There was however one property could not be quantified that did vary across the different number of cycles—failure mode. A significant observed phenomenon was the fact that as the different cycled samples were tested, the failure modes became more violent. For example, most of the control specimens broke with a clean break down the center, while the 10 cycled specimens violently shattered into several pieces and appeared to have failed first at a 45° ply. Since there is no data to support any cracks being present of substantial size, one possibility is that of crack beginnings in the center of the specimen. However it is peculiar that whatever damage was done did not have an effect on any of the other properties obtained. From preliminary testing this toughened composite seems very suitable for cryogenic fuel tanks. To continue a more thorough investigation of this material, higher numbers of cycles should be tested to find where the material does begin to crack. Also, a more in depth crack analysis may be necessary such as with an electron scanning microscopes to look for even smaller cracks to explain the changes in failure mode. A more elaborate series of gas permeability experiments could also be performed using gasses with smaller particles such as He or testing at various temperatures. A set of parallel tests should be performed on beam specimens, which is a more likely candidate for fuel tank structures. Combined loading and thermal cycling could also be a possible area of interest. Composite fuel tanks for cryogenic applications have many versatile and exciting possibilities, however there is still much research to be done to make them a viable option in the future.

ACKNOLEDGEMENTS

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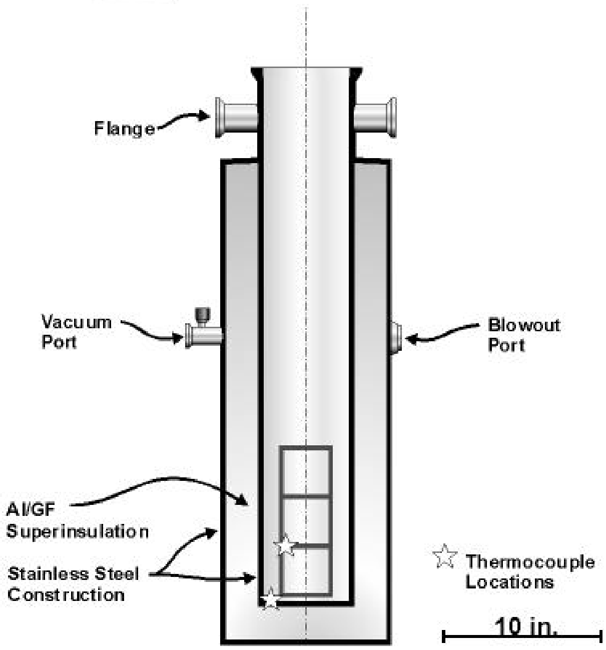
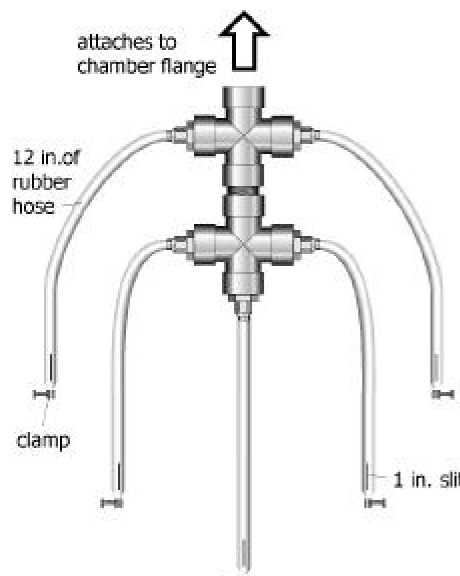
2. Beaumont, Brewer, Lagace, Varnerin. "TELAC MANUFACTURING COURSE CLASS NOTES," Technology Laboratory for Advanced Composites, September 1991.

APPENDIX A: Cryocycler Assembly

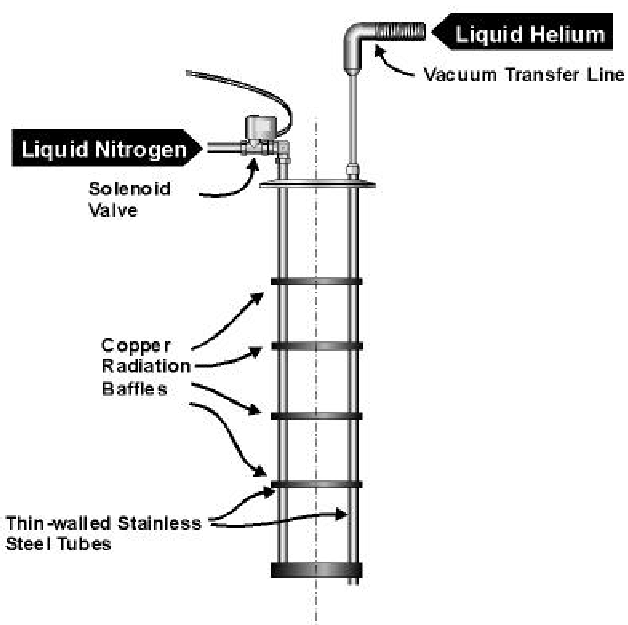
Cryogenic Dewar



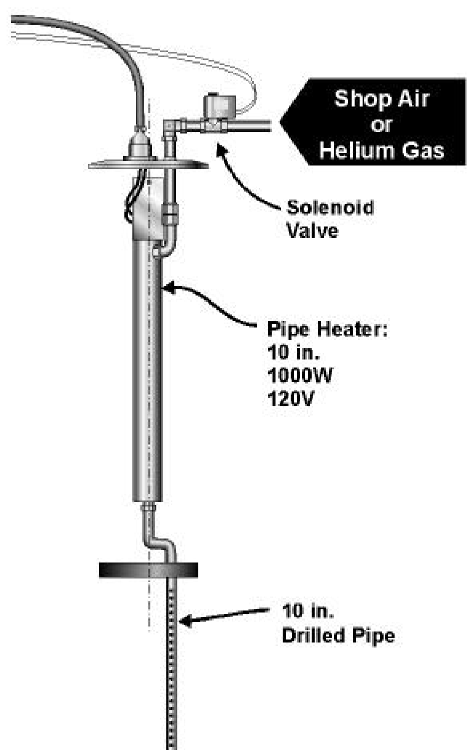
Pressure Relief



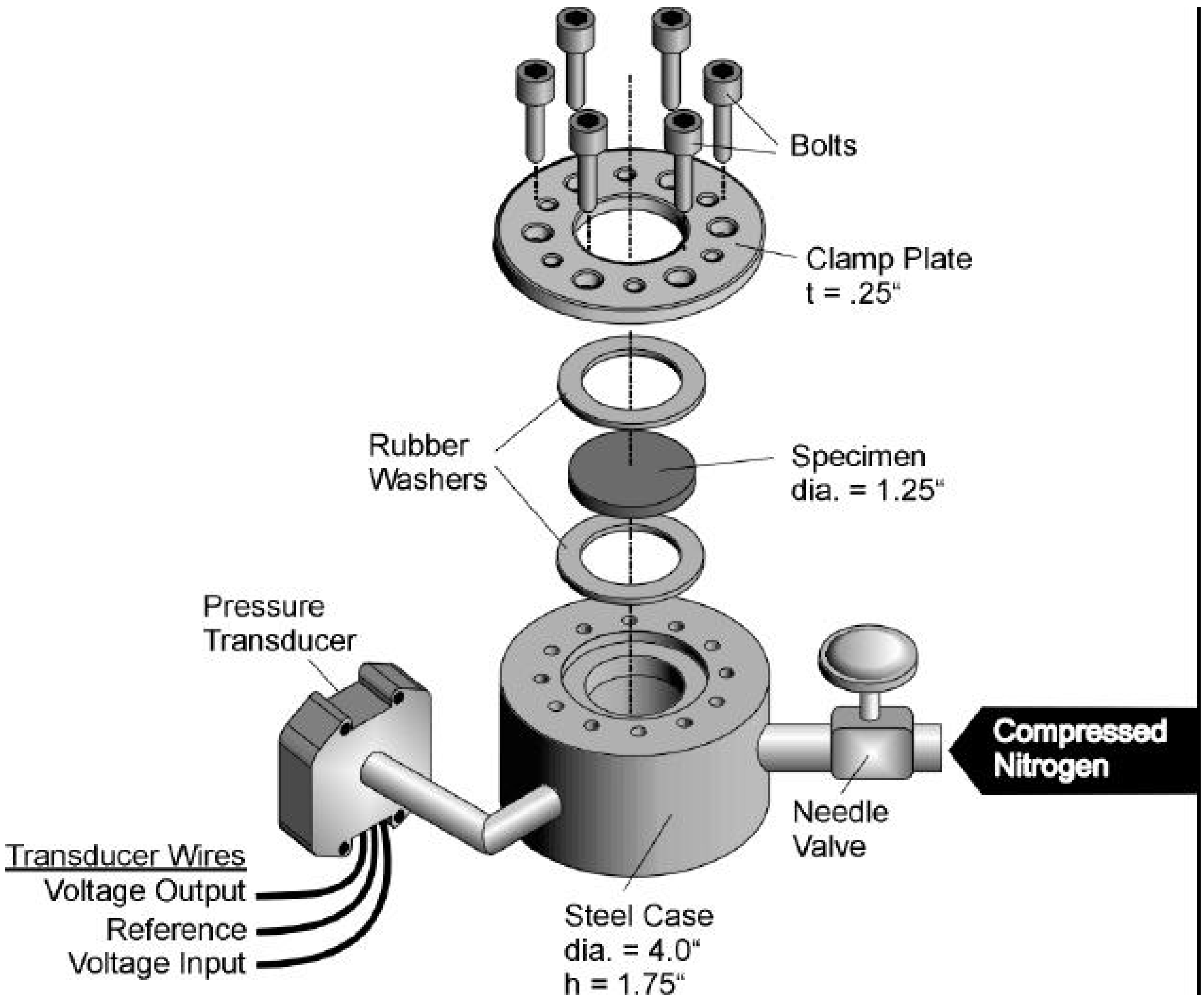
Cooling System



Heating System



APPENDIX B: Permeability Set-Up



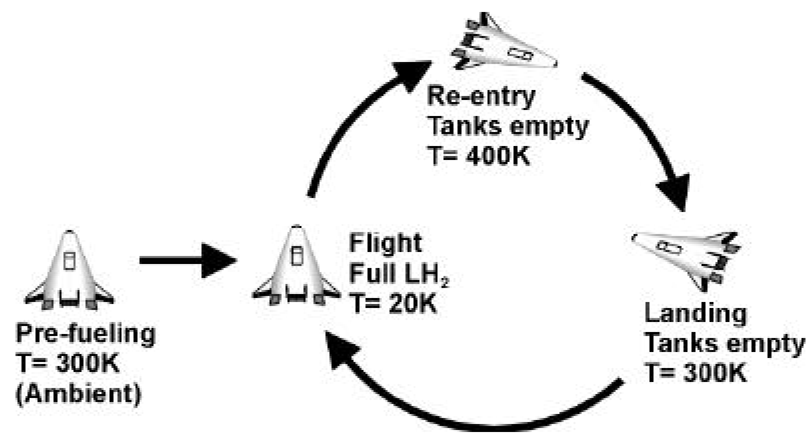


Figure 1: Flight cycle of the X-33 vehicle

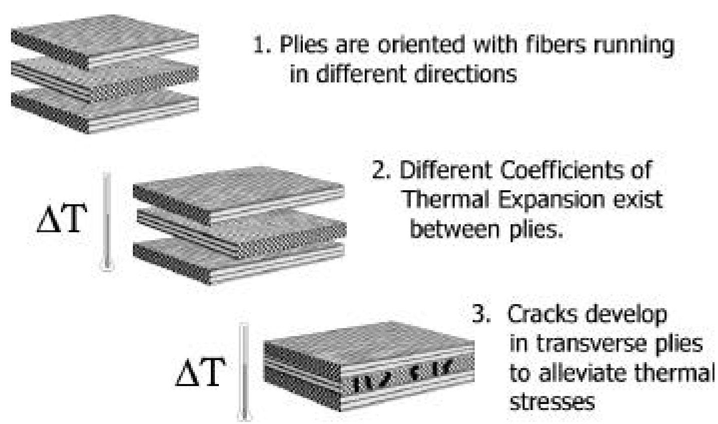


Figure 2: Crack development in composite laminate

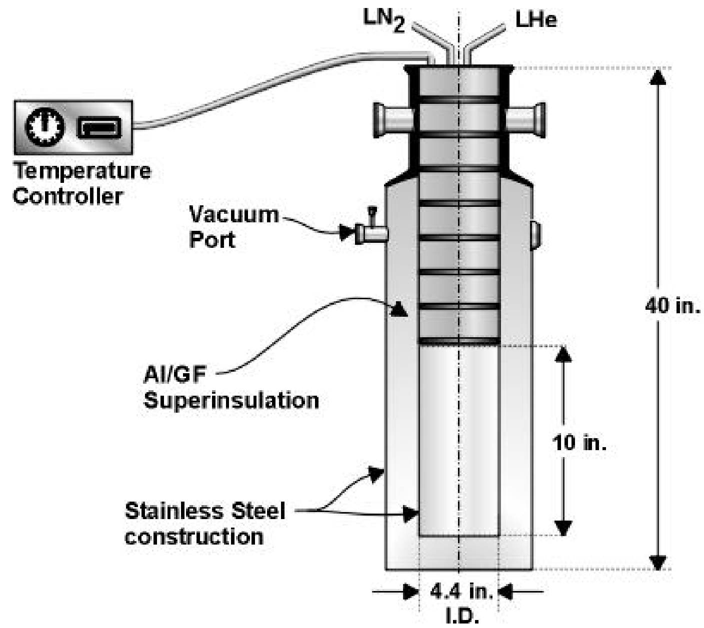


Figure 3: Cryogenic thermal cycling chamber

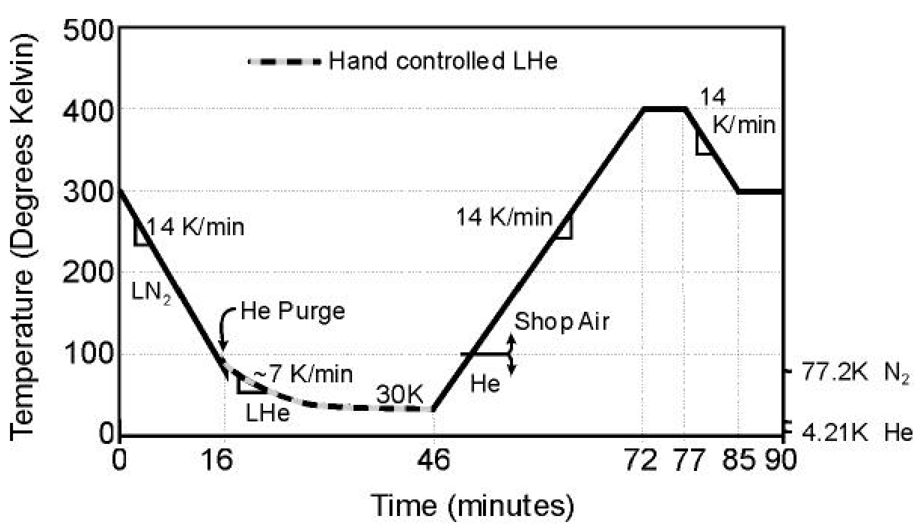


Figure 4: Temperature cycle timeline used in experiments

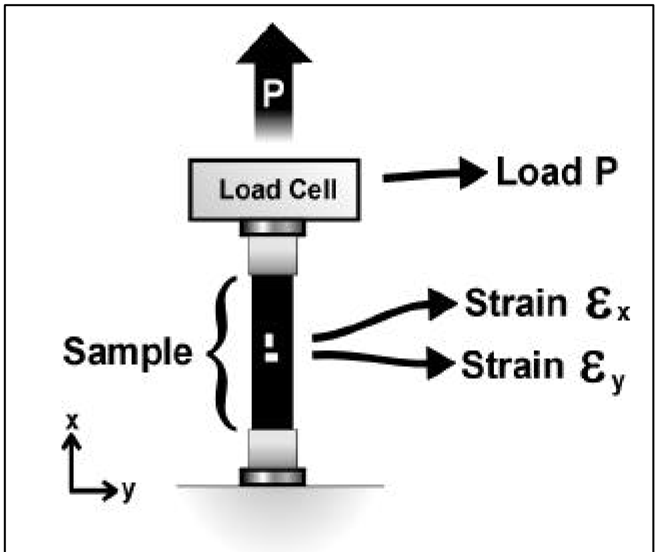


Figure 6: Tensile loading test setup

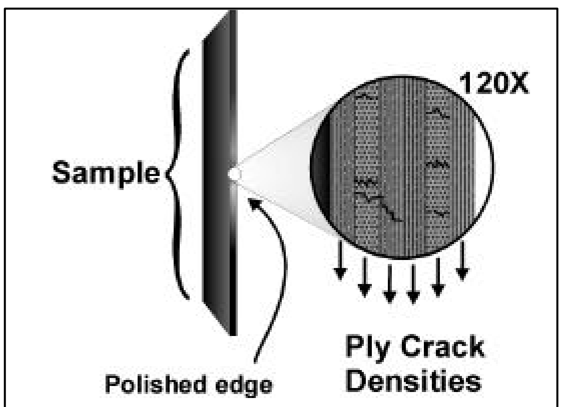


Figure 7: Crack density count

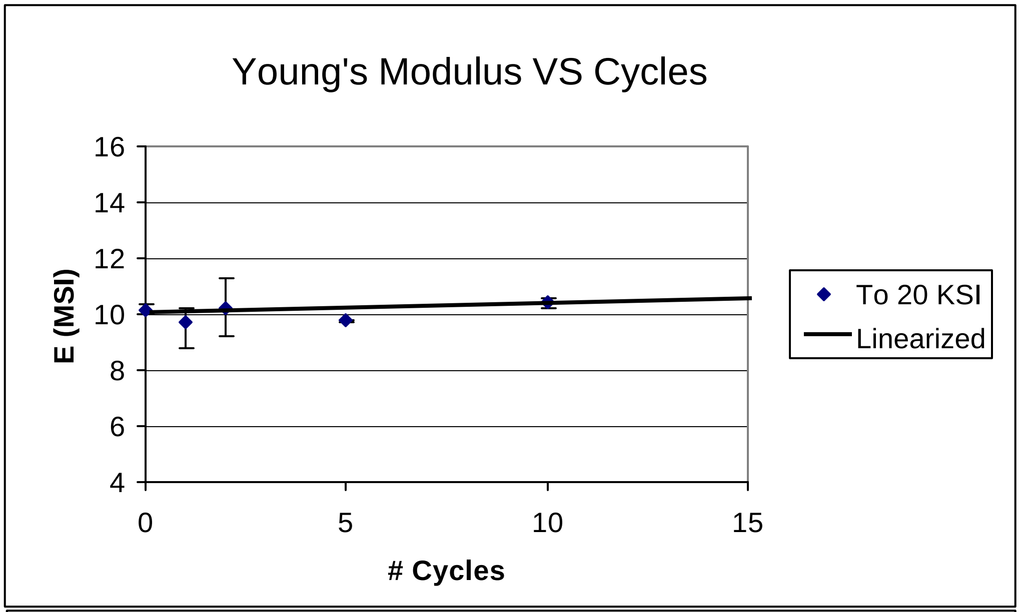


Figure 8: Young's modulus VS cycles

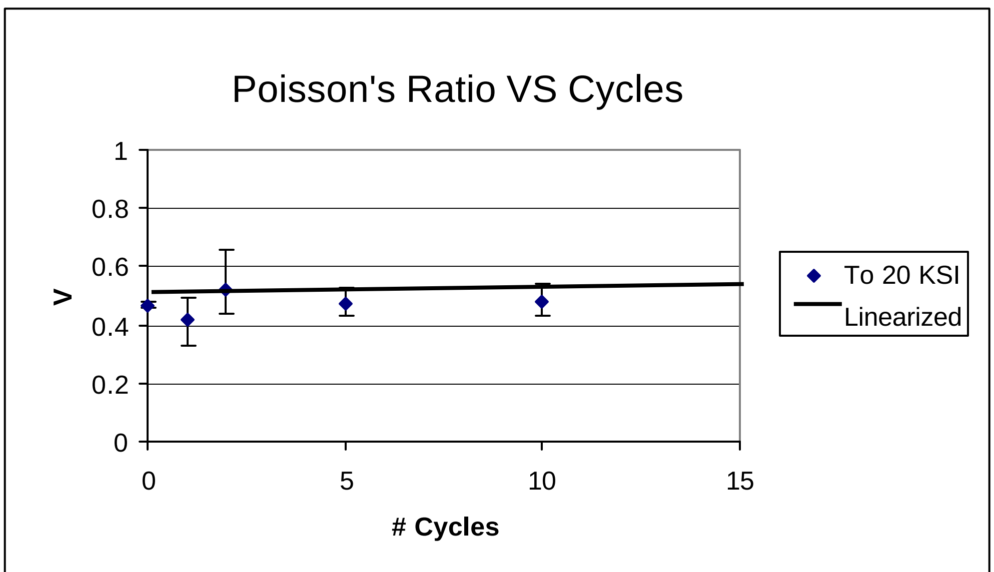


Figure 9: Poisson's Ratio VS Cycles

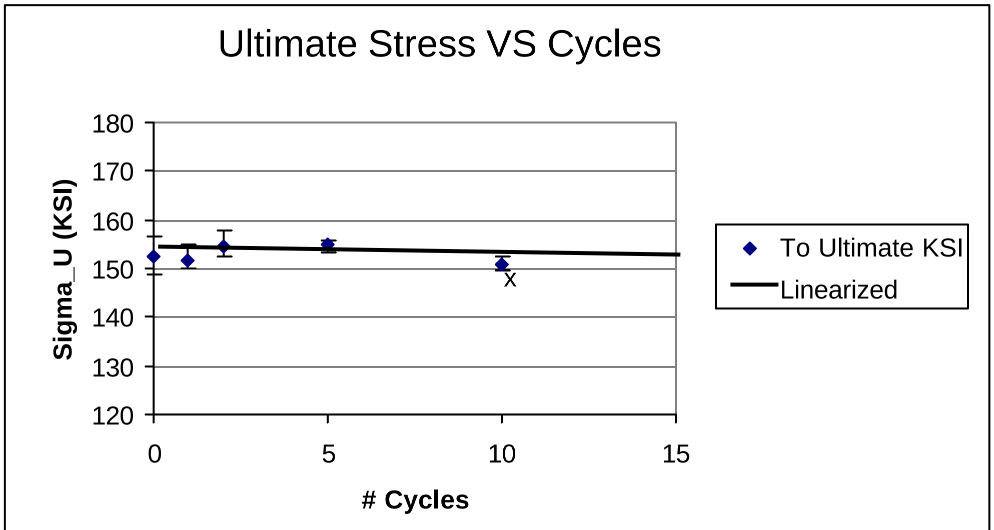


Figure 10: Ultimate stress VS cycles

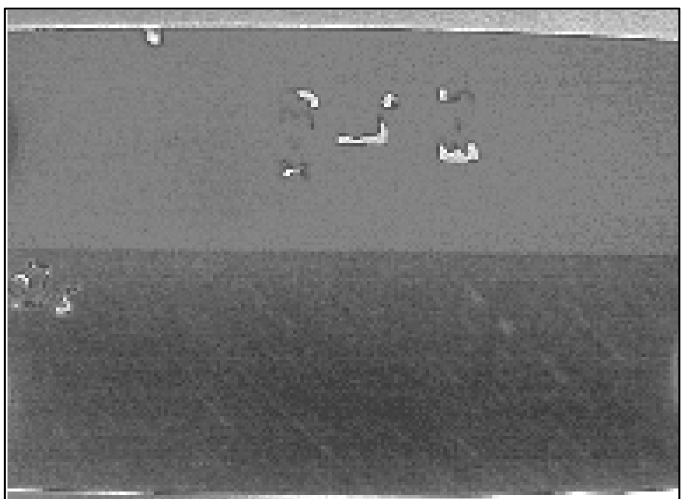


FIGURE 11: NDE crack comparison