EFFECT OF ELEVATED TEMPERATURE AND LOADING RATE ON DELAMINATION FRACTURE TOUGHNESS

J. R. Reeder
Mechanics and Durability Branch, NASA Langley Research Center,
Hampton, VA 23681

D. H. Allen
School of Engineering, Nebraska University, Lincoln, NE 68588

W. L. Bradley
Engineering Dept., Baylor University, Waco, TX 76798

ABSTRACT

The effects of temperature and loading rate on delamination growth were studied. The delamination fracture toughness of IM7/K3B was measured at 149°C, 177°C, and 204°C. At each temperature the tests were performed with a variety of loading rates so that the delamination initiated over the range of time from 0.5 sec to 24 hrs. The double cantilever beam (DCB) test was used to measure fracture toughness. The results showed that the delamination resistance is a complicated function of both time and temperature with the effect of temperature either increasing or decreasing the fracture toughness depending on the time scale. The results also showed that the fracture toughness changed by as much as a factor of three as the time scale changed over the five orders of magnitude tested.

INTRODUCTION

As composites are used in more and more demanding structural applications, understanding their damage tolerance becomes increasingly important. Delaminations can be formed from many types of damage events and are a primary failure mechanism for composites. When composites are considered for elevated temperature applications such as on a high speed aircraft[1], there is the additional concern that unexpected failures could occur after prolonged loading due to time dependent growth of delaminations. This is especially true in applications where the expected service life may be measured in decades. This paper investigates how the resistance to delamination growth is affected by temperature and time scale.

DCB specimens were used to measure Mode I fracture toughness in this study. The DCB test is a well established test used to measure interlaminar fracture toughness in fiber reinforced composite materials [2]. Toughness was measured at several elevated temperatures, and to investigate the effect of time scale, loading rates over a range of five orders of magnitude were used.

EXPERIMENTAL PROCEEDURE

An IM7/K3B composite material was used in this study. This composite material is specially formulated to perform at elevated temperatures and has been considered for high speed aircraft applications [1]. The composite is comprised of IM7, a fairly common high strength graphite fiber manufactured by Hercules, and K3B, an amorphous thermo-plastic polyimide resin. The K3B material can be used at elevated
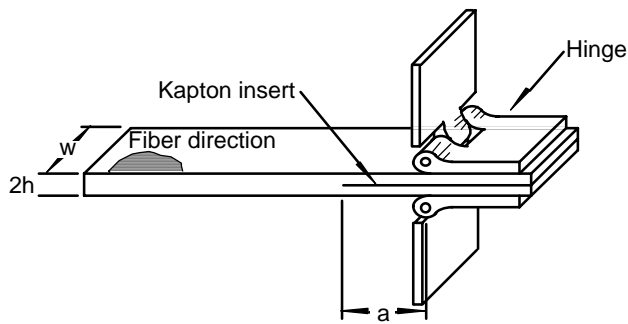
Figure 1. The DCB test specimen.

temperatures because of its high T_g (236^oC) [3]. The longitudinal, transverse and shear moduli of the composite are 159, 8.9, and 5.9 GPa, respectively [4].

The DCB specimens were cut from 24-ply unidirectional laminates that had been specially manufactured with a 0.05 mm Kapton layer at the midplane. These sheets of Kapton were sprayed with a mold release agent so that the Kapton did not bond to the composite during the curing process, thus creating an artificial delamination within the laminate. The layer of Kapton was placed in the laminate so that when the laminate was cut into test specimens, the Kapton would extend 5.7 cm in from one edge, as seen in Figure 1. The DCB specimens were cut to a width (w) of 25.4 mm, and a thickness (2h) of approximately 3mm. Load was applied to the specimen through aluminum hinges which were adhered to the specimen. An epoxy-phenolic film adhesive, designated HT 435 by the American Cyanamid Co., was found to perform adequately at the elevated temperatures. The hinges were bonded so that the crack length (a), which is the distance from the center of the hinge pins to the interior end of the Kapton film, measured 38 mm.

Before testing, the DCB specimens were dried and aged. This K3B material has been shown to change properties due to physical aging[3]. By aging the test specimens at 204°C for 66 hrs, the effect of further aging occurring during the test itself was kept small.

During the DCB tests, load, displacement, and temperature measurements were recorded. During some of the tests, video images of the delamination growth were also recorded. The DCB tests where conducted in a 100 kN hydraulic load machine, but load measurements where taken from a secondary 4.4 kN inline load cell. The displacement from the test was measured by an LVDT (linearly variable displacement transducer) located within the test machine. The controller is capable of controlling either the load or the displacement of the test machine. The test machine is equipped with an environmental chamber which surrounds the test section and is capable of creating test environments from -129°C to 316°C. The temperature inside the chamber and on the specimen surface was measured by 12 type-T thermocouples. All instruments were connected to the test computer. The test computer could send commands to the load frame controller while reading and recording the test data. The test machine connects to the test specimen through two grips. The top grip was a mechanical grip but the bottom grip was hydraulic and could be actuated from outside the environmental chamber. The connection between each grip and the load frame was water cooled to protect the equipment outside the chamber that could not withstand the elevated temperature. The experimental setup is shown in Figure 2.

During a few of the tests, delamination growth was observed using a microscope with a long focal length. This microscope allowed the crack tip to be observed from outside the environmental chamber. The microscope rested on a translation stage so that the advancing crack tip could be followed. The microscope was equipped with a video camera and the images were recorded. A time stamp was placed on the images so that images could be synchronized to load and displacement data after the test. Because a high level of illumination was needed to observe the crack tip through the microscope, a remote light source with a high temperature fiber-optic cable was used to focus light at the crack tip. In addition, a thin layer of white correction fluid was applied to the specimen in the vicinity of the crack tip so that delamination growth could be monitored more readily.

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Delamination toughness tests were performed at three temperatures: 149, 177 and 204°C. Tests were also attempted at 232°F, but these tests failed by compression on the surface of the specimen near the crack tip prior to delamination growth. These tests were therefore invalid and were disregarded. At each temperature, constant load rate tests were performed with a range of loading rates that produced failure times from 0.5 sec to approximately 24 hrs. The five loading rates were 267, 13, 0.53, 0.027, and 0.0013 N/sec. At each loading rate and temperature, five duplicate tests were performed.

To perform each test, the specimen was attached to the top mechanical grip. The environmental chamber was closed and the temperatures inside the chamber were allowed to stabilize. The bottom hydraulic grip was then closed from outside the chamber. The specimen was then ready to be tested. The tests had to be controlled in different ways depending on the load rate used for the given test. The 0.53 and 13 N/sec tests were performed using the hydraulic load frame controller under load control. Because the hydraulic load controller could not control at the slower loading rates, the 0.027 and 0.0013 N/sec tests were performed with the test computer controlling the applied load. At the fastest load rate (267 N/sec) the hydraulic load controller could not produce the desired load rate without going into unstable oscillation. Therefore, at the fast loading rate test, the specimens were loaded in displacement control.
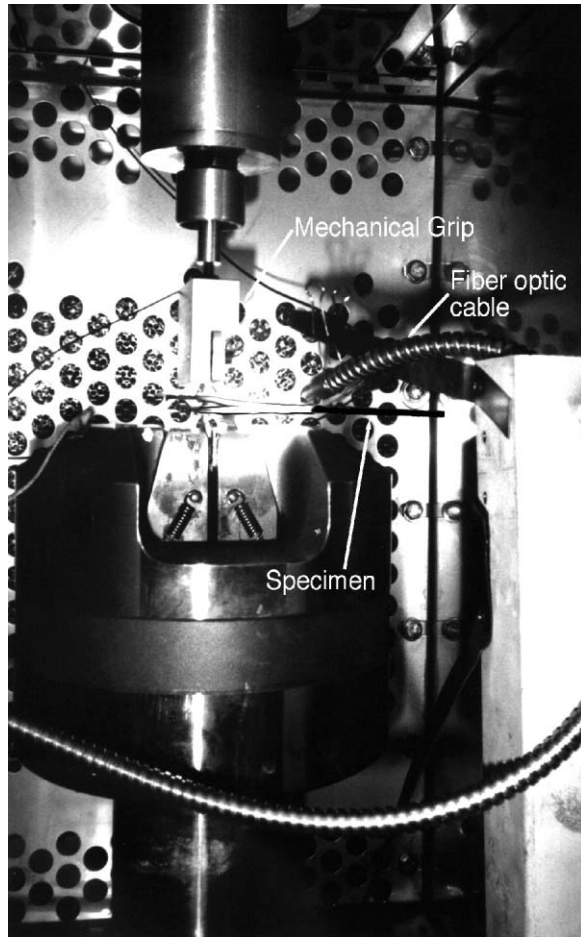
Figure 2. Photograph of a DCB test in progress.

There is no difference in displacement control and load control as long as the compliance of the specimen is constant. A displacement rate of 10 mm/sec was chosen to achieve a loading rate of approximately 267 N/sec for these specimens. The exact loading rate was determined from the measured load response of each test.

During each test, load and displacement were recorded along with temperature data. At least 100 data points were recorded while each specimen was being loaded to failure. To measure the initial delamination length, each specimen was broken open after testing, and the distance from the hinge pin to the end of the Kapton insert was measured. In this way, the initial delamination length was measured off of the delaminated face of the specimen where the end of the insert could be plainly seen. Measured load was then plotted versus displacement, as seen in Figure 3. From the graph, the slope of the initial linear portion (m), the load where the loading curve deviated from linear (P_{NL}), and the maximum load point (P_{MAX}) were recorded. The load values of each test were corrected by the zero load point reading taken from the load cell just after each specimen broke. For the fastest tests, a graph of load versus time was also plotted, and the slope of the initial linear portion was recorded as the loading rate.

The ASTM protocol for performing the DCB test[2] gives three options for calculating the fracture toughness from experimentally measured values. With each option the theoretical value for toughness given in equation 1 is modified to give more accurate. The modifications rely on the record of applied load (P), displacement (δ), and crack length (a), measured as the delamination grows.
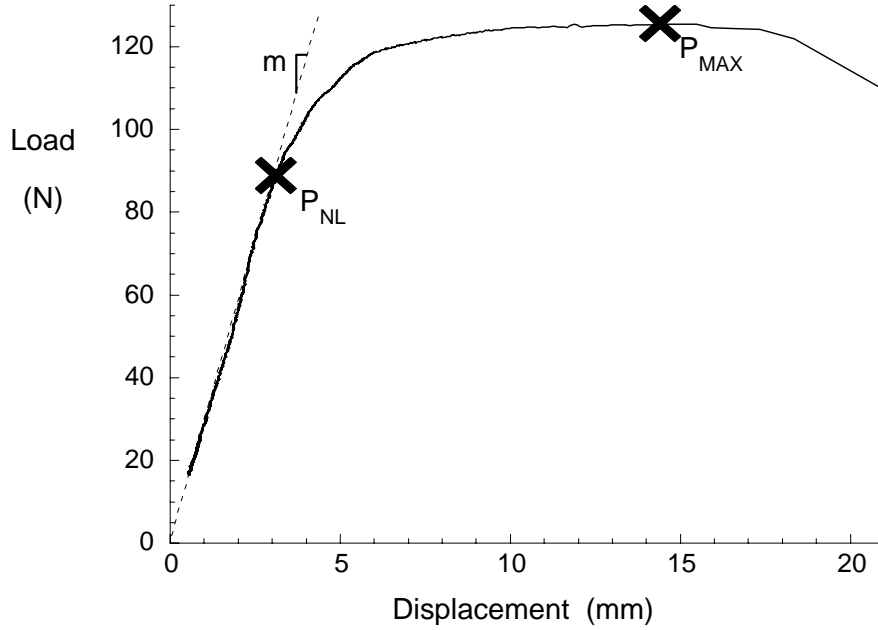
Figure 3. Typical load-displacement response from DCB test.

$$G_{Ic} = \frac{1}{w}\frac{dU}{da} = \frac{3P\delta}{2wa}$$

(1)

Unfortunately, because a record of crack extension could not be recorded under all test conditions, fracture toughness in this paper had to be calculated in a different manner. The ASTM protocol for the mixed mode bending (MMB) test [5], which is a very similar delamination test, uses a different calculation method. The change in calculation method is necessary because the delamination often grows unstably, and therefore, it is often not possible to obtain a record of P, δ and a as the delamination grows. The MMB toughness calculation equations were adapted for use with the DCB test. This formulation, shown in equation 2, is derived by expressing displacement as a function of elastic modulus, and by modifying the crack length by the χ parameter to account for deformations not modeled by basic beam bending theories.

$$G_{Ic} = \frac{12P^2(a + \chi h)^2}{w^2 h^3 E_{flex}}$$

(2)

where $\chi = \sqrt{\dfrac{E_{11}}{11\,G_{13}}\left\{3 - 2\left(\dfrac{\Gamma}{1+\Gamma}\right)^2\right\}}$ and $\Gamma = 1.18\dfrac{\sqrt{E_{11}\,E_{22}}}{G_{13}}$, and where E_{11}, E_{22} and G_{13} are the longitudinal,

transverse and shear moduli, respectively. The flexural modulus in this equation is back-calculated from the slope of the load displacement curve, m, using equation 3.

$$E_{flex} = \frac{2(a + \chi h)^3}{3\frac{wh^3}{12}}m$$

(3)

Substituting equation 3 into equation 2 produces equation 4 which was used to calculate fracture toughness from test data in this study.

$$G_{Ic} = \frac{3P^2}{2\,w\,(a + \chi h)\,m}$$

(4)

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Linear elastic fracture mechanics is assumed in equation 4 and in the use of the terminology G_{Ic}. This may seem strange when testing at elevated temperature and at time scales where a visco-elastic deformation may cause changes in the fracture toughness. In this case, using linear elastic fracture mechanics is believed to be valid because the load displacement curve will be shown to be linear up to the point of delamination propagation. Thus any significant visco-elastic deformation that may be occurring in a specimen is assumed to occur on a very local scale in the crack tip region as part of the crack growth process.

RESULTS

The ASTM standard for the DCB test indicates that three different critical loads may be used to determine fracture toughness [2]: P_{NL}, the load at which the loading curve deviates from linear; P_{VIS}, the load at which the delamination is observed to grow; and P_{MAX}, the load at which load reaches a maximum. (The standard actually limits the "MAX" value to be the maximum load that occurs within a 5% increase in compliance). Since multiple options were given, it was necessary to determine which of these loads was most appropriate for these tests. However, since crack growth could not be observed for every test, only P_{NL} and P_{MAX} were considered. As seen in Figure 3, a great deal of nonlinearity occurs between the nonlinear point and the maximum point. The P_{MAX} point is also significantly higher than the P_{NL} so the calculated toughness will depend greatly on which critical point is used. The nonlinearity in the loading curve could be due to either nonlinear deformation or delamination growth. If it is due to nonlinear deformation in the loading arms, then the "MAX" critical load might be the most appropriate, but the data reduction method described earlier would not be valid because it assumes linear fracture mechanics. If the nonlinearity in the loading curve is due to delamination growth, then the "NL" critical load is the most appropriate choice, and the data reduction method should be valid.

To determine the source of the loading curve nonlinearity, tests were conducted using a video camera to monitor the delamination growth studied where the delamination position was monitored with video
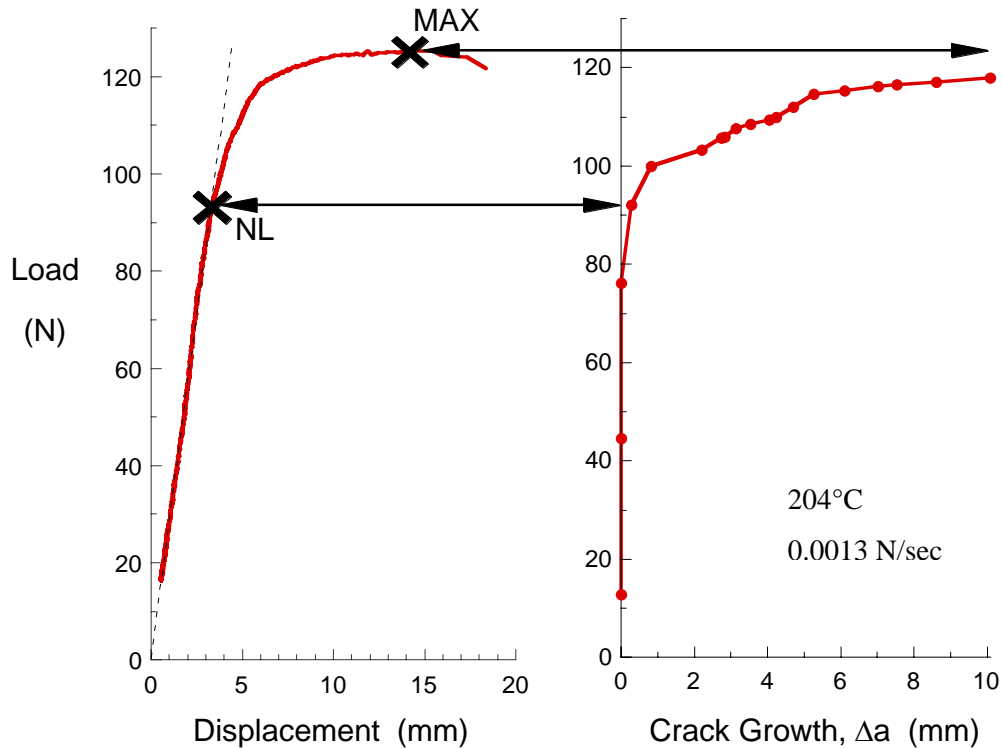

Figure 4. Displacement history compared to delamination growth.

images. Figure 4 shows the load displacement curve and the delamination growth response for a 0.0013 N/sec test performed at 204°C. This slow, high temperature test is expected to be the worst case for viscoelastic deformation in the loading arms. Figure 4 shows that the delamination begins to grow just as the loading curve deviates from linear, thus making the P_{NL} the appropriate choice for the fracture toughness calculation at delamination onset.   The maximum load point occurs after substantial delamination growth and when the crack is growing too fast to be followed by the camera. Since all but the fasted tests were run in load control, it is somewhat surprising that the delamination did not grow immediately to failure.   The fact that it grew stably as the load increased indicates that the fracture toughness increased significantly as the delamination grew. This response, called a "rising R curve", is most likely due to fiber bridging [6-8] and to viscoelastic effects. Any increase in fracture toughness that is due to fiber bridging is an artifact of the unidirectional test specimens used and would not be expected to occur in real structures.

The fracture toughness results calculated using the "NL" critical load point are presented in Figure 5. The data is plotted as toughness vs. load rate. Because these tests were at constant load rate and because the change in load to failure was small compared to the change in load rate, a plot of the same data vs. time-to-failure would be almost identical. Because it may be easier to think in terms of time instead of in terms of rate, the time-to-failure scale is shown above the graph. Notice that the time-to-failure scale is reversed so that the longer times are to the left and that both rate and time-to-failure are plotted on a log scale. Five specimens were tested at each condition, and the data at each temperature is fit with a best fit curve. The results show that delamination toughness is significantly affected by loading rate. The fracture toughness generally increases with increasing load rate (decreasing time-to-failure), but this trend appears to reverse itself at the lowest load rate condition for both the 177°C and 204°C test conditions. The sensitivity to load rate changes with the temperature. At 149°C, the rise in fracture toughness is
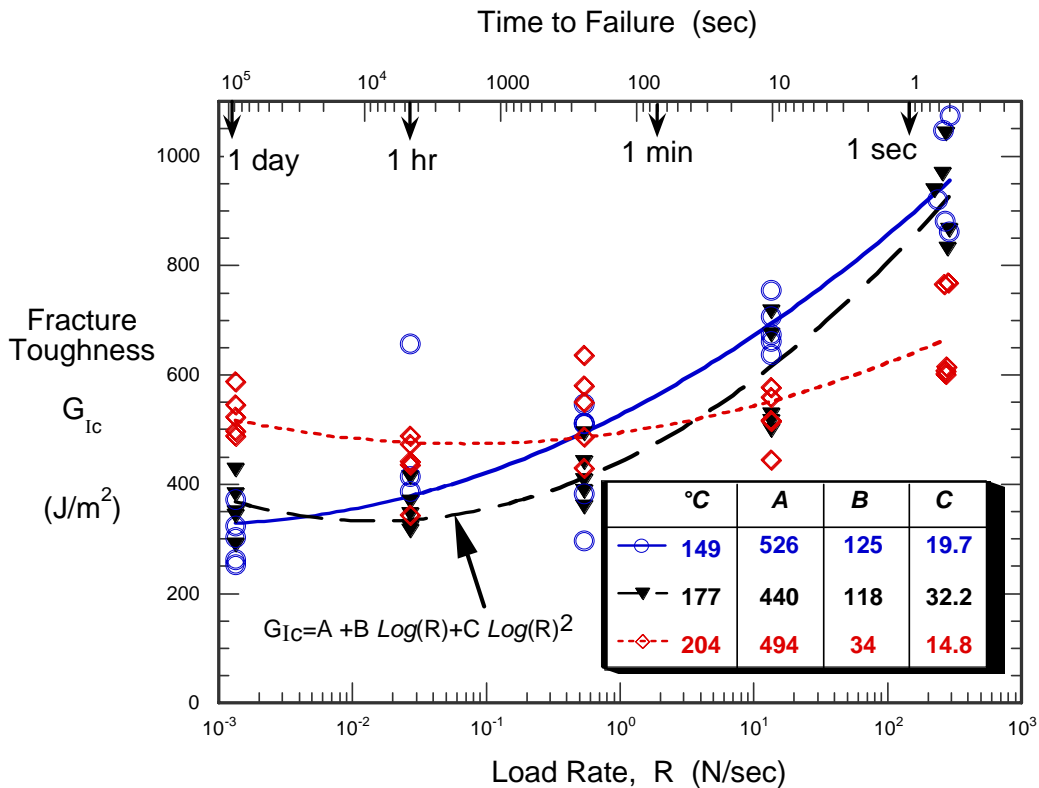

Figure 5. Delamination energy release rate results.

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nearly 3-fold over the range of loading rates tested, while at 204°C, the rise is fairly minor. Although an increase in temperature increases the sensitivity to loading rate, it does not always increase the fracture toughness itself. The effect of increasing temperature caused a decrease in toughness at higher load rates but an increase in toughness at lower load rates. The lowest fracture toughness and therefore the most critical condition in the range tested occurred at the lowest temperature (149°C) and at the slowest loading rate 0.0013 N/sec (failure in 24 hrs.).

DISCUSSION

The fracture toughness data clearly show that fracture is a complicated function of time and temperature. This experimental study was originally conceived as an attempt to explain why composite transverse strength data obeyed the same type of time-temperature superposition laws as the modulus data obeys [9, 10]. The thought was that if the strength data was actually controlled by small flaws in the composite then a change in observed strength might be due to a change in fracture toughness. Since fracture toughness is a complicated function of stiffness, the similarity in strength and stiffness (modulus) might be explained through the effect on toughness.

This attempt to explain the strength results was not successful because the fracture toughness results could not be shifted in time to form a single master curve. With both strength and modulus, a change in temperature could be shown to be equivalent to a shift in the time scale (usually plotted on a log scale) [3, 9]. However, the fracture toughness curves at different temperatures shown in Figure 5 intersect each other instead of lying parallel to one another. Therefore, it is clear that they cannot be shifted in time to form a master curve. If a master curve could have been formed from the fracture toughness data, longer tests at lower temperatures would have been equivalent to shorter tests at higher temperatures. Therefore, it would have been possible to predict fracture toughness under much longer time spans than were actually tested. Since a master curve was not formed, expensive long term testing will be needed to determine the effects of long term loading.

Because the fracture toughness data does not form a master curve, it is clear that the effect of time and temperature on fracture toughness is more complicated than can be explained by the effect on modulus alone. This is best exemplified with the effect of temperature at different time scales. At fast loading rates, an increase in temperature increases toughness whereas at slower loading rates an increase in temperature decreases fracture toughness. This complex response is believed to be due to a combination of three effects related to an increase in temperature: a decrease in modulus, an increase in strain to failure, and a decrease in material strength. Therefore an increase in temperature can cause an increase or a decrease in toughness, depending on which effect is dominant.

No model to explain this complex response can be offered at this time. The data itself are seen as valuable because it demonstrates the complexity of the response and shows that the magnitude of the effect of time and temperature can be quite significant. The toughness of the material changed by a factor of three over the range of temperatures and load rates studied here.

CONCLUDING REMARKS

The delamination fracture toughness of IM7/K3B was measured by the DCB test at 149°C, 177°C, and 204°C over a range of loading rates so that delamination initiated in the range from 0.5 sec to 24 hrs. The deformation response of the test specimen was found to be linear up to the point of delamination onset. Therefore, the use of linear elastic fracture mechanics and the fracture toughness terminology is believed to be valid, even at the elevated temperatures where viscoelastic deformation may be occurring in the matrix, particularly the matrix in the crack tip region. The material was found to have a large R-curve

effect where the toughness increased with crack growth. The R-curve effect was so large that stable crack growth was obtained even in these load control tests.

The effect of time and temperature on fracture toughness was found to be both significant and complex. At the lowest temperature, the toughness tripled as the load rate increased by 5 orders of magnitude and time-to-failure changed from 24 hrs to 0.5 sec. The effect of loading rate was much less significant at the highest temperature. The effect of increasing temperature caused a decrease in toughness at higher load rates but an increase in toughness at lower load rates. Unlike the strength and stiffness results for this material, the fracture toughness results could not be shifted using time-temperature superposition to form a master curve. Therefore, the complex effect of time and temperature on fracture toughness cannot be explained by simple viscoelasticity models.

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