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Strain Rate Sensitivity of Polymer-Matrix Composites Under Mode I Delamination

M. D. Kistner Structural Composites Branch Nonmetallic Materials Division

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Materials Laboratory Wright Research and Development Center Air Force Systems Command Wright-Patterson Air Force Base, Ohio 45433-6533



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Post failure fractograp	hic examinations we	re utilized to c	orrelate significant
variations in mechanica	l properties with i	ractographic fea	tures. Mechanical test
results were summarized	i via data plots wit	th error bars to	show which effects
were statistically sign	uricant.		
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FOREWORD

This technical report was prepared by Mr. M. D. Kistner of the Structural Materials Branch, Materials Laboratory, Wright Research and Development Center, Wright-Patterson AFB, Ohio, under Project 2419 "Nonmetallic and Composite Materials." The period of this work was from June 1984 to April 1987.

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SECTION I

INTRODUCTION

An area of concern under the Task. Durability of Composites and Adhesives, in Work Unit Directive (WUD) 45 is the durability of thermoplastic composite materials. The recent development of solvent resistant thermoplastics such as polyetheretherketone (PEEK, ICI Industries, 1981) has increased interest in using thermoplastic matrix composite materials in more structurally demanding applications. This trend requires that the Air Force understand more of how these thermoplastic materials behave under rate of applied loads or displacements, environmental effects (moisture, ultraviolet light, etc.), temperature, and other factors affecting structural performance.

This study will gain a better understanding of how a damaged structure will behave under the combined effect of temperature and crack opening strain rate. The objective of this program is not to produce design data, but to examine if the design philosophy of using a strain rate independent fracture toughness valid for AS4/3502 is (graphite/epoxy) and AS4/APC-2 (graphite/PEEK). AS4/3502 will be used as baseline a thermosetting matrix composite system which shows "stable" crack growth. AS4/APC-2 will be used as a thermoplastic composite system which has been shown to exhibit "unstable" crack growth at ambient conditions under "quasi-static" crack opening displacement rates (0.05 In/Min). The difference between "stable" and "unstable" crack growth is discussed by Kinloch and Williams (Ref. Fig. 1 shows the difference between 1). continuous "stable" and discontinuous "unstable" crack growth for a constant width double cantilever beam.





The continuous growth system (Fig. 1) may ideally be characterized by a constant G_{Ic} value where the unstable growth system requires two values. In reality, the fracture toughness may vary along the length of the specimen due to strain rate effects, specimen geometric factors, fiber bridging, etc. With the AS4/APC-2 system, the crack growth behavior may vary between

these two modes depending upon the testing temperature and applied crack opening displacement rate as shown later in the report.

SECTION II

EXPERIMENTAL METHODS

1. PANEL FABRICATION

AS4/3502 and AS4/APC-2 unidirectional prepreg materials were received from Hercules, Inc. and ICI Industries respectively. In the batch of AS4/APC-2 prepreg received was noted we noted the prepreg layer having some regions of fiber which were not fully This caused some problem in processing the wetted out by resin. The size of the panels produced are 6 inches by 9 material. inches and 12 inches by 18 inches for AS4/APC-2 and AS4/3502 respectively. The fiber directions are in the 6-inch and 12-inch directions with the panels consisting of 34 plues of 0° layers. The AS4/APC-2 panel is laid up by aligning and spot welding AS4/APC-2 prepreg layers until a 17-ply package is formed; two of these packages are formed for each laminate. Between these packages, a 1.25 inch folded household grade aluminum foil (~1 mil thickness) coated with Frekote release agent is placed along the 9-inch direction of the 6-inch by 9-inch laminate lay-up. AS4/AFC-2 is then consolidated in a press using a steel The matched metal die. A diagram of the molding operation is shown in Fig. 2.



Figure 2. Press Molding Lay-Up

The consolidation cycle for the AS4/APC-2 material is:

- A. Ambient to 720° F at the maximum press heat up rate (~12 F/min) while under contact pressure.
- B. Apply 150 psi when the mold temperature reaches 720° F.
- C. Hold 720° F 750° F under 150 psi for 45 minutes.

D. Cool to under 200° F under pressure. (cooling rate achieved 3-5 F/minute from 720 F to 400° F with forced ambient temperature air and $20-30^{\circ}$ F/minute from 400° F to 200° F with water coolei platens).

E. Remove the laminate from the mold at a temperature of 180° F or less.

For the AS4/3502 material, the prepreg layers have sufficient tack to easily lay up one ply on top of the prior plies at ambient conditions. The center starter crack is formed by using 1-mil nonportus teflon coated glass sheets along each of the 18-inch sides 1.5-inches into the laminate. This laminate is cured in an autoclave using the lay up shown in Fig. 3.



Figure 3. Autoclave Lay-Up for AS4/3502 Panels

The cure cycle is as follows:

Ambient Conditions to 270° F at $3-5^{\circ}$ F/Min. Hold at 270° F for 15 Min. Apply 85 PSI Hold for 45 Min. at 85 PSI and 270° F 270° F to 355° F at $3-5^{\circ}$ F/Min. Hold 355° F for 2 Hours Cool to 120° F or lower at $3-5^{\circ}$ F/Min. Release Autoclave Pressure and Remove Cured Laminate

After the panels were consolidated or cured, they were ultrasonically C scanned and stored in a desiccator.

2. TEST AFPARATUS DEVELOPMENT

The Double Cantilever Beam test used in this project requires the measurement of crack lengths, loads, and crack opening displacements at specific points during the test to calculate fracture toughness values. Past fracture toughness tests which were ran under 'quasi-static' strain rates allowed the observer to visually monitor and record the crack length as the test proceeded. This project which used crack opening displacement rates up to 20.0 inches per minute required a method to rapidly electronically or optically record and correlate the crack length with the data from the test machine.

Some initial studies investigated using single strain crack detection gages produced by Micro-Measurements along with the crack detection system of the test machine to electronically record the crack growth data. This method of crack detection was ultimately abandoned due to the tendency of the crack to tunnel under rather than failing the gage. This method of failure occurred for all trials at elevated temperatures examined under this project. Another problem occurred at elevated temperatures. We used a GA-61 high temperature strain gage adhesive system (manufacturer's use range of -100 to 600°F for strain gage applications) to bond the testing tabs to the specimen surface, but found that the tabs tended to debond from the specimen by ductile bond failure.

To eliminate the problem of using strain gages, we used a high-speed videotaping method to record the test data. The videocamera was set with a shutter speed of 1/1000 of a second per frame and recorded 200 frames per second. This speed was sufficient to record all data except for the fast crack propagation speed for AS4/APC-2. Under the fast crack propagation speeds, jumps up to slightly greater than 1 inch occurred in less than the 1/200-second time interval between consecutive videotape frames. No method was found to successfully record these fast crack propagation velocities, but two potential methods were identified.

> 1. High Speed Photography - This method has the advantage of increasing the rate of photographing the crack growth event to several thousand frames per second. The disadvantages are that the camera requires some time to get up to operating speed and has a narrow window for recording the crack growth event. This method was not attempted due to the difficulty in timing the camera start without knowing when a rapid crack growth event will occur.

> 2. Use of a Digital Oscilloscope and Crack Detection Gages - The advantage of using a digital oscilloscope is that the data may be recorded at the rate of a few microseconds per data point. Another advantage is the data before and/or after a chosen event may be recalled from memory and stored via a floppy disk. Also the operator has the option of changing the characteristics of this event marker during the run. For instance, if single strain crack propagation gages are electronically coupled to form a parallel circuit, the resistance and voltage level will change as the gages fail. The digital oscilloscope, for instance, may be set to trigger upon a decrease in voltage below some critical value. Bv changing this voltage level after each crack detection gage which fails in the slow growth region and keeping the data slightly before and after the first gage which

> > 4

fails in the fast growth region, then the rapid crack growth velocity may be obtained. The major difficulty with this method is finding a crack propagation gaging system which may be used at elevated temperatures as discussed. Some future experimental previously development may be to develop a method to apply an electric circuit directly to the edge of the specimen without using a Kapton backing material. This would involve electrically insulating the specimen edge and applying a crack detection circuit of conductive material on this edge.

To eliminate the problem of tab bond failure, an innovative mechanical method to attach the T-tabs to the specimen surface was developed. This consisted of a metal backing plate which is attached to the tab by two metal screws. See Fig 4.



Figure 4 Modified T-tab Specimen Attachment

This method requires the specimen to be sawed with a 0.060-inch thick cut along the separator ply for one-half an inch into the specimen as is shown in Fig. 5.





A simple metal fixture was used to aid in the sawing of this 0.060-inch cut. This fixture consisted of a metal bar with the curvature of the diamond saw blade drawn on it. The end of the Double Cantilever Beam with the separator ply is clamped one-half an inch inside the line along the radial direction (Fig. 9).



Figure 9. End Groove Cutting Fixture

The depth of the cut is important. Too much depth will decrease half beam bending stiffness and cause error in the the crack opening displacement. If the depth is too shallow, thegrip surface of the tabs will be reduced and can cause the tabs to The curvature of the saw blade does not cause error in the slip. fracture toughness because the crack initiation film is one-half an inch further into the specimen. The thickness of the Modified bottom plate needs to be carefully designed to accommodate T-tab the specimen beam thickness. In the area of the screw connection, a thickness will cause excessive deflection small of the too center of the bottom plate and may thus lead to some initial crack opening of the beam; too large a thickness w111 cause insufficient clamping force allowing the T-tab to slip. Using method proved to be very reliable for all test temperatures this

and rates. Only one failure out of the approximately 100 tests may be directly attributed to the method by which the tabs were applied. This failure resulted from the diamond saw cut along the back of the specimen. This AS4/APC-2 specimen tested at ambient temperature, and a strain rate of 20.0 inches/minute applied crack opening displacement rate showed crack growth at the midplane (as would be expected for the unidirectional laminate) for about 1.5 inches into the specimen, then a secondary delamination started from the corner of the saw cut, and the crack growth continued from this secondary delamination as is shown in Fig. 6.





3. SPECIMEN PRODUCTÍON

The specimen type and size chosen for this study is the Double Cantilever Beam with dimensions of 0.5 inch (width), 5.5 inches (length), and 34 plies thick. The total thickness of the specimens are about 170 mils for AS4/3502 and 180 mils for AS4/APC-2. The specimens were cut using a table saw with a water cooled diamond impregnated blade. The specimen orientation is shown in Fig. 7. Density measurements were taken using a water displacement method. Fiber volumes were calculated by using an acid digestion method and these results were verified by direct measurement off a 37.5X optical photograph of an "average" void content polished cross section. These physical properties are presented in Table 1.

Material	Panel #	Fiber Volume (%)	Void Vo (۴)	olume	Interlaminar Resin Layer Thickness
			Photo	Acid	(10E-3 in)
AS4/3502	1	64.3	-	-	0.51
AS4/APC2	1 2	63.8 61.3	0.9 0.4	1.0 0.9	0.49 0.50
	3	62.8	0.6	0.6	0.54
	4	62.9	1.0	1.0	0.57
	5	61.1	0.4	0.8	0.55

Table	1.	Laminate	Physical	Properties
aule	1.	Lammate	FIIYSICAL	rioperilea





Cutting Diagrams for (a) AS4/APC-2 and (b) AS4/3502 Test Panels

Figure 7.

8

The interlaminar resin layer thickness did not consist of a pure resin layer. Often fibers migration occurred, and at some points occurs to such an extent that the resin layer may not be distinguished as is shown in Fig. 8.



Figure 8.

Interlaminar Resin Layer

This fiber migration has a significant effect upon the Mode I delamination behavior of both material systems examined. It was visually evident during the Mode I delamination testing that a significant amount of fiber bridging was occurring. The effect of fiber bridging may be to artificially increase the fracture toughness value obtained, since delamination in multidirectional laminates may exist between angle ply layers where the fiber migration will be far less than in the unidirectional layers used for this project. Also, in thick composite structures, the crack opening displacement may be much less than the relatively thin laminates used in this project; the effect of this lower crack opening displacement is to reduce the effect of fiber pull-out as an energy absorbing mechanism.

4. MECHANICAL TESTING

The mechanical testing setup consisted of a screw driven Instron Tensile Machine Model *LWS along with a 10,000-pound Load Frame Model #TTC and a 2-inch-high strain extensometer arranged such to do Mode I testing. The Instron chart has a respondence time of 500 microseconds across 90% of the full scale chart. To this test setup was added a high speed videocamera setup with two videocameras synchronized to record both the data off the Instron chart and the crack length via a split screen image. These high speed videocameras produced frame speeds of 1/1000 of a second per frame and 200 frames per second. This speed was found to be sufficient for recording the crack growth behavior for both AS4/3502 and AS4/APC-2 with the exception of recording the fast also crack growth velocity for AS4/APC-2. The test setup was modified by using the modified T-tab specimen attachments as

previously described in this report. These tabs have a pinned attachment to the Instron grips to allow free rotation of the Also, a simple specimen chamber was developed for specimen ends. performing elevated temperature tests. This chamber consists of an aluminum frame and a glare-free glass front. The chamber is heated via an adjustable temperature hot air blower. By using an baffle, the temperature is able to be uniformly air maintained throughout the length of the specimen. The temperature was controlled to within a limit of 5° F across the specimen length and usually varied by only 2-3 F. The typical cycle for the highest temperature took 15 minutes: approximately 10 minutes to heat and stabilize the specimen surface temperature and a 5minute soak at temperature. The test chamber setup is shown in Fig. 10.



Figure 10. Mode I Test Chamber Setup

A dual camera videotaping system was used to record the necessary data. By using a split screen, the data from the Instron chart and the crack length were able to be correlated for each increment of time recorded. The videotape data for AS4/APCwhich showed stick/slip discontinuous crack growth was checked 2 for crack length after the test by observing the easily visible transitions from slow to fast growth regions. The slow growth regions appear as lighter areas. The transition from slow to fast growth is accompanied by a rapid drop in applied load which can be seen on the Instron load/crack opening displacement chart. Thus, all the necessary data to calculate the value of Mode I toughness may be taken by correlating the crack fracture length with the corresponding event on the Instron load/crack opening displacement chart.

5. DATA REDUCTION

The method of data reduction used in this study is the Generalized Empirical Method as described in Refs. 2 and 3. This method of data analysis assumes the relationship

where

is the crack opening displacement
R is an effective empirically determined compliance term
P is the applied load
a is the crack length
n is an empirically determined slope

The empirically determined values of R and n are determined from a least squares fit of the $\log(P/d)$ versus $\log(a)$. By using the equation of the form

 $\log(P/\delta) = -\log(R) - n\log(a)$

the slope determines the value of n and the y-intercept determines the value of R. The critical strain energy release rate is then calculated by using

 $G_{Ic} = nP_c \delta_c / 2ba$

where

 P_c is the critical applied load O_c is the critical crack opening displacement b is the laminate width.

The effect of shear deformation was assumed to be small, since relatively large crack lengths of 2 to 4 inches are being used. Also, this method of calculation assumes inelastic effects are minimal. By examination of the loading and unloading curves, the viscoelastic effects appear to be limited to a small area around the crack tip, i.e. only small residual crack opening displacements remain after growing the delamination and returning the applied load to zero, thus validating the test as mostly elastic effects. From the resulting G values, a statistical analysis was performed on each of the modes of crack propagation, i.e. slow, fast, and continuous crack growth, for each condition examined. The statistical error was calculated by using an error limit of one standard deviation from the median value.

III RESULTS AND DISCUSSION

The summarized G values are presented for AS4/3502 in Table For AS4/3502, only continuous crack growth behavior 2. 15 observed (Fig. 11). The data show no significant trends with strain rate at a constant temperature. The only data point which shows some significant variation is at 250° F and 0.02 inch per minute applied crack opening displacement rate. These data are further plotted as a function of temperature in Fig. 12. The data generally show increased G_{Ic} values with temperature. An exception to the trend is the data point at 0.02 inch per minute applied crack opening displacement and 250°F and a very slight statistically insignificant drop for 0.20 inches per minute and 350⁰ F. and Also, the G_{Ic} fracture toughness increases by a factor of 1.7 to 1.8 from ambient conditions to the maximum test temperature of 350°F.

The summarized G_{Tc} values for AS4/APC-2 are presented in Table 3. The G_{IC} values are reported for both continuous and discontinuous crack growth behavior. The discontinuous crack growth behavior may be characterized by transitions from slow to rapid crack propagation. The value of G increases under slow growth until a critical value is reached which is called the initiation value, then a rapid crack initiation occurs. The lower value of G under which the fast crack growth stops is called the For continuous growth, the value of G_{Ic} was arrest value. calculated for every one-fourth of an inch for crack lengths from 1.75 to 3.75 inches. The error in the data was calculated by taking all data points of each type of crack growth behavior and calculating the standard deviation. The general data trends showed very high scatter for crack initiation with a reduced scatter for crack arrest and continuous growth crack respectively. The effect of increasing the crack opening strain rate as is shown by Fig. 13 is to transition from continuous to discontinuous crack growth. Again, the scatter in the data tends to eliminate any strain rate effects which are seen. Fig.14 shows the effect of temperature. An increase in temperature tends to favor continuous crack growth. The effect of increasing temperature is noted to have a strong effect upon increasing the crack initiation fracture toughness, but, has little effect upon the crack propagation values except at the highest applied crack opening displacement rates. Also, note that the fracture toughness for continuous crack growth tends to be of the same order as that for crack initiation. The results of this report show very little effect of applied crack opening displacement rate upon all the modes of crack propagation. The room temperature crack initiation data are verified by Smiley (Ref. The data from Smiley showed an almost constant value of G 3). for crack initiation in the "Ductile" and "Transition" modes for AS4/APC-2 at room temperature.

The post-testing evaluation consisted of examination of the fracture surfaces via a Scanning Electron Microscope (SEM). For

	Table	2		
Summary	of G _{Ic} Values	for AS4/3502		
	Cross	shead Displac	ement Rate	(inches/minute)
0.02	0.20	2.00	20.0	

Temp. (^O F) RT	* 1.42+/-0.18	1.40+/-0.09	1.20+/-0.09	1.27+/-0.27
250	3.55+/-0.36	2.03+/-0.26	2.12+/-0.15	1.84+/-0.35
300	2.01+/-0.26	2.49+/-0.40	2.12+/-0.28	1.98+/-0.30
350	2.51+/-0.23	2.44+/-0.30	2.19+/-0.23	2.18+/-0.34

* units for $\boldsymbol{G}_{\mbox{Ic}}$ are inch pounds per square inch

Table 3

Summary of G_{Ic} Values for AS4/APC-2

	Crosshe	ad Displaceme	nt Rate	(inches/minute)
0.02	0.20	2.00	20.0	-

Temp. $(^{\circ}F)$

RT

Arrest Continuous	5.11±0.42 [*] 14.20±1.83	6.98±2.55 14.47±0.89	6.65±2.22 10.92	5.62±0.45
Initiation	13.26±2.89	14.39±2.73	11.34±2.41	11.71±1.41
200				
Arrest		6.01±1.53	6.10±1.90	5.46±0.93
Continuous	17.79±0.70	17.57±2.49	18.25±2.04	17.36±1.89
Initiation		15.44±4.05	14.21±4.19	15.74±4.80
250				
Arrest		6.42±0.82	7.31±1.23	8.29±0.83
Continuous	11.74±0.39	20.07±3.23	24.20±4.05	21.78±1.68
Initiation		21.36±2.88	20.21±7.73	15.66±8.58
300				
Arrest			6.44±3.01	10.36±3.75
Continuous	23.74±0.66	25.97±3.02	25.80±1.20	29.60±3.61
Initiation			27.69±1.70	24.87±8.32

* units for $\boldsymbol{G}_{\mbox{Ic}}$ are inch-pounds per square inch



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AS4/3502, the energy deformation mechanism consisted of the formation of hackles along with fiber pull-out. By examination of Figures 15 and 16, a much greater number of hackles may be seen on the fracture surface for AS4/3502 tested at 0.02 inch per minute applied crack opening displacement rate and 250 F. This result is also seen in the calculated Mode I fracture toughness which is about 75% higher for this condition. By comparison of Figures 16 and 17, the effect of increasing the strain rate at 250° F tends to dramatically decrease the number of hackles and reduce the Mode I fracture toughness by approximately 50%. For AS4/APC-2, a dramatically different fracture surface is seen. In the slow crack growth regions in Figures 19, 21, 22, and 23, the matrix resin may be seen to be stretched between the fracture By comparison, Figures 18 and 19 showing fast growth surface. areas have much less resin deformation and yield a much smoother fracture surface. Comparison of the differences between fracture surface shown in Figures 20 and 21 to that of 18 and 19 show that at higher strain rates less difference is seen between the fast and slow growth fracture surfaces. By comparison of Figures 19 and 22, the amount of resin stretching and extent of fiber pullout may be much greater at 300 F. As the strain rate is increased at 300 F, the mode of failure changes from continuous crack to "stick-slip" discontinuous crack growth growth. The morphology in the slow growth region (Fig. 21) compares fairly well with that in the continuous growth region (Fig. 19). The growth region in Fig. 24 shows much less resin ductility fast than either the slow or continuous growth regions (Figures 19 and 23).

IV CONCLUSIONS

This report has looked at the combined effect of temperature and applied crack opening displacement rate upon the Mode I fracture toughness of AS4/APC-2 and AS4/3502. The results for AS4/3502 show significant changes in fracture toughness with temperature. The general trend is increases in temperature yield, increases in fracture toughness, the exception to this general rule is the data point at 250 F and 0.02 inch/minute applied crack opening displacement rate. At a given temperature, the effect of strain rate is smaller than the scatter in the data; the only exception is the data at 250 F and 0.02 inch per minute.

For the AS4/APC-2 system, the effect of increasing test temperature or decreasing test speed is to cause a tendency for the crack to grow in a continuous rather than discontinuous (stick-slip) type of crack propagation. Also, increases in test temperature for a given strain rate tends to increase the crack initiation but not the arrest Mode I fracture toughness, only at the highest applied crack opening displacement rate was a significant increase in arrest Mode I fracture toughness seen. Thus, except for the highest applied crack opening displacement rate, the effect of increases in temperature causes a larger difference in the arrest and initiation Mode I fracture



Figure 15 - AS4/3502 Mode I Fracture Surface: Tested at ambient conditions and 0.02 inch per minute applied crosshead speed.



Figure 16 - AS4/3502 Mode I Fracture Surface: Tcsted at 250°F and 0.02 inch per minute applied crosshead **speed**.



Figure 17 - AS4/3502 Mode I Fracture Surface: Tested at 250°F and 20.0 inches per minute applied crosshead speed.



Figure 18 - AS4/APC-2 Mode I Fracture Surface: Fast crack propagation area tested at ambient conditions and 0.02 inch per minute applied crosshead speed.



Figure 19 - AS4/APC-2 Mode I Fracture Surface: Slow crack propagation area tested at ambient conditions and 0.02 inch per minute applied crosshead speed.



Figure 20 - AS4/APC-2 Mode I Fracture Surface: Fast crack propagation area tested at ambient conditions and 20.0 inches per minute applied crosshead speed.



Figure 21 - AS4/APC-2 Mode I Fracture Surface: Slow crack propagation area tested at ambient conditions and 20.0 inches per minute applied crosshead speed.



Figure 22 - AS4/APC-2 Mode I Fracture Surface: Continuous crack propagation are tested at 300°F and 0.02 inch per minute applied crosshead speed.



Figure 23 - AS4/APC-2 Mode I Fracture Surface: Slow crack propagation area tested at 300°F and 20.0 inches per minute applied crosshead speed.



Figure 24 - AS4/APC-2 Mode I Fracture Surface: Fast crack propagation area tested at 300° F and 20.0 inches per minute applied crosshead speed. toughness.

Future investigation upon the strain rate effects for AS4/3502 or AS4/APC-2 should focus on expansion of the strain rate range and development of faster methods to record the crack velocity. Also, future investigation may involve model development to better understand the combined effect of time and temperature upon the Mode I fracture toughness.

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