T. Kevin O'Brien[1] *and Roderick H. Martin*[2]

Round Robin Testing for Mode I Interlaminar Fracture Toughness of Composite Materials

REFERENCE: O'Brien, T. K. and Martin, R. H., "**Round Robin Testing for Mode I Interlaminar Fracture Toughness of Composite Materials,**" *Journal of Composites Technology & Research*, JCTRER, Vol. 15, No. 4, Winter 1993, pp. 269–281.

ABSTRACT: This report summarizes the results of several interlaboratory "round robin" test programs for measuring the Mode I interlaminar fracture toughness of advanced fiber-reinforced composite materials. Double cantilever beam (DCB) tests were conducted by participants in ASTM Committee D-30 on High Modulus Fibers and Their Composites and by representatives of the European Group on Fracture (EGF) and the Japanese Industrial Standards Group (JIS). DCB tests were performed on three AS4 carbon fiber-reinforced composite materials: AS4/3501-6 with a brittle epoxy matrix, AS4/BP907 with a tough epoxy matrix, and AS4/PEEK with a tough thermoplastic matrix. Difficulties encountered in manufacturing panels, as well as conducting the tests, are discussed. Critical issues that developed during the course of the testing are highlighted. Results of the round robin testing used to determine the precision of the ASTM DCB test standard are summarized.

KEYWORDS: composite materials, double cantilever beam (DCB), interlaminar fracture toughness, delamination, standard test method

The data contained herein were generated by voluntary participants using the double cantilever beam (DCB) test (Fig. 1). The DCB test consists of a unidirectional fiber-reinforced laminate, manufactured with a thin insert implanted at the mid-plane near one end to simulate a sharp crack, and loaded such that the delamination forms at the insert in an opening mode (Mode I). Specimens were cut from panels manufactured using prepreg voluntarily supplied by several material suppliers. A list of participants is included in the Appendix. A chronology of the activity is documented in Ref *1* in the form of excerpts from ASTM meeting minutes from 1986 to 1992.

Early discussions (prior to 1985) resulted in limiting the DCB test to 0° unidirectional laminates to prevent the initial delamination from branching to interfaces away from the midplane [2]. The width-tapered DCB configuration [*3*] was abandoned because of the added complexity of machining this configuration and the tendency for 0° unidirectional width tapered laminates to split at the juncture between the narrow and tapered regions. Furthermore, the phenomenon of fiber bridging between the two 0° plies on either side of the delamination [*4–6*] was first observed during this phase.

Since 1986, five distinct rounds of testing were conducted. The first round yielded useful data for AS4/BP907. However, little data were obtained for the other two materials because of problems that were experienced in obtaining sufficiently thin or completely disbonded inserts for starting the delaminations. The second round of testing yielded useful results for AS4/3501-6, although with fewer labs participating. However, problems were again encountered with the manufacture of AS4/PEEK panels with good quality inserts. The third round of testing was conducted in conjunction with the European Group on Fracture (EGF) and the Japanese Industrial Standards (JIS) group. Although sufficient AS4/PEEK panels were manufactured to conduct a thorough test matrix, specimens obtained from these panels had problems with torn and folded aluminum inserts. The fourth round of testing, consisting of static tests from a DCB fatigue round robin, yielded more data on AS4/PEEK specimens with the Kapton inserts. The fifth round of testing yielded sufficient data on AS4/PEEK specimens with thin Upilex inserts to determine the precision of the DCB standard test method.

Background

The DCB test consists of a unidirectional continuous fiber-reinforced laminate, manufactured with a thin insert at the midplane near one end, and loaded such that the delamination forms at the insert as a Mode I, or opening mode, fracture. The parameters that were investigated in the round robins were: (1) the method of introducing the opening load, (2) specimen thickness, and (3) insert type and thickness.

Figure 1 shows two configurations of the DCB where the load is introduced via piano hinges (Fig. 1*a*) or loading blocks (Fig. 1*b*). A variation on the loading block configuration designated "T-tabs" was also used (Fig. 1*c*). In the first round of testing, load introduction was accomplished using either piano hinges or T-tabs. Piano hinges were used exclusively in the second round. By the third round, correction factors for loading blocks and tabs had been developed, with specific guidelines for when they were required. Hence, both piano hinges (Fig. 1*a*) and end loading blocks (Fig. 1*b*) were used in rounds 3 through 5.

Specimens in the first two rounds were 25-mm (1-in.) wide. In the third round, however, 20-mm-wide specimens were tested, but a limited number of 12.5, 25.0, and 37.5 mm (0.5, 1.0, and 1.5 in.) wide specimens were also tested. Because no significant width effect was discovered (see Ref *1*), 20 to 25-mm wide specimens were tested in the fourth and fifth rounds.

In the first and second rounds, specimens consisted of 24-ply laminates for the AS4/3501-6 and AS4/BP907 tests, and 36-ply

[1]U.S. Army Research Laboratory, Vehicle Structures Directorate, NASA Langley Research Center, Hampton, VA 23681.
[2]Analytical Services and Materials, Inc., Hampton, VA 23666.

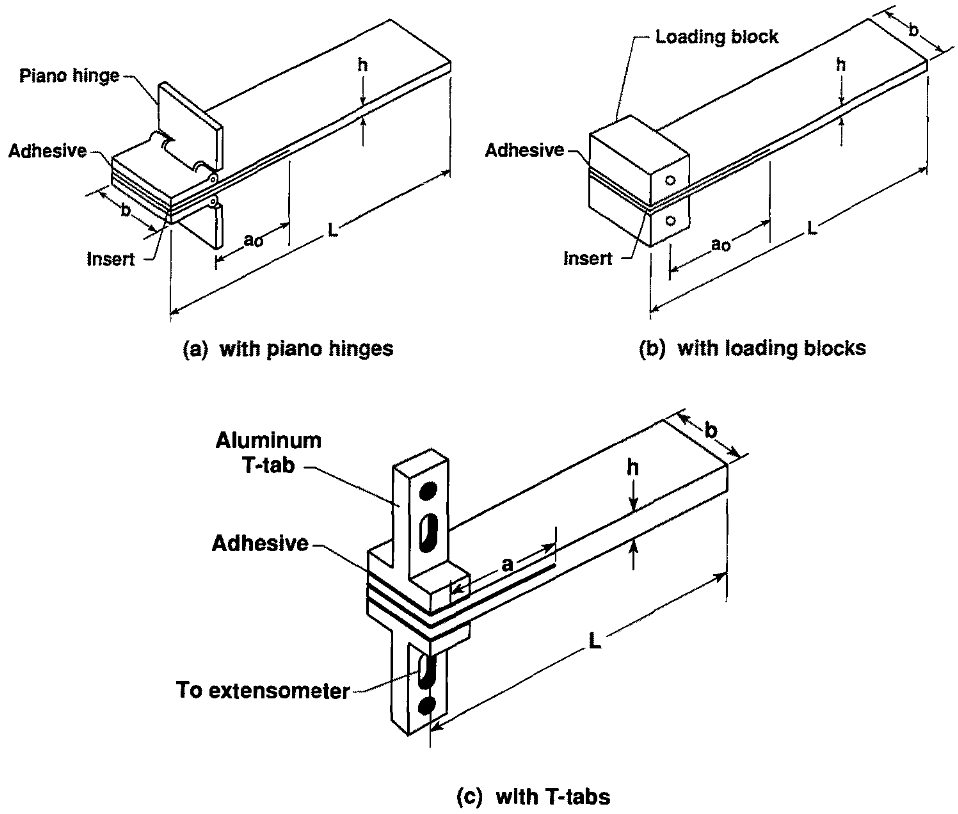

FIG. 1—*DCB specimen configurations.*

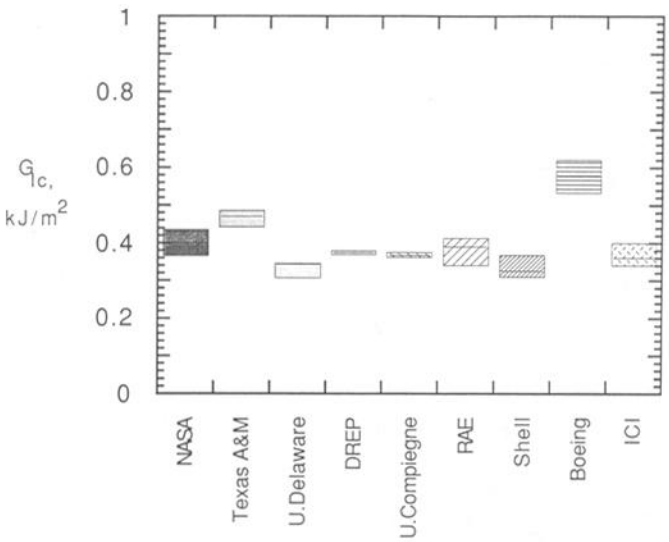

FIG. 2—*Box plot showing* G_{Ic} *round robin results for AS4/BP907 DCB tests using piano hinges.*

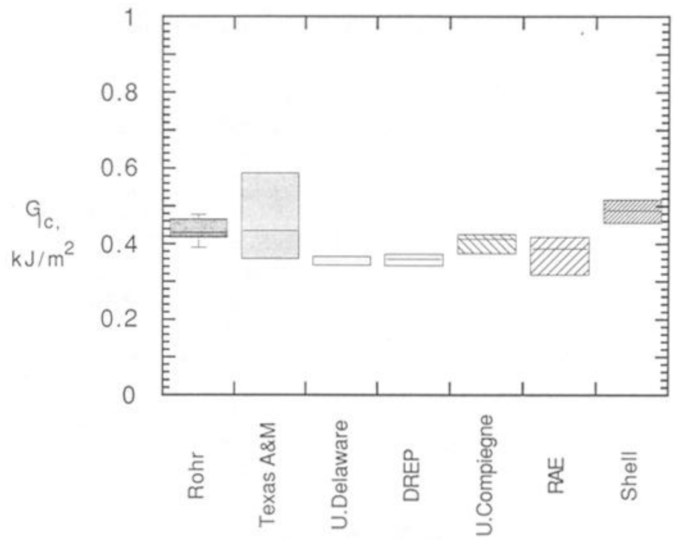

FIG. 3—*Box plot showing* G_{Ic} *round robin results for AS4/BP907 DCB tests using T-tabs.*

laminates for the AS4/PEEK tests, with a nominal ply thickness of 0.127 mm (0.005 in.). By the third round, guidelines for specimen thickness and correction factors for geometric nonlinearity were available. Hence, tests on 24-ply AS4/PEEK specimens were conducted in rounds 3 through 5.

The most sensitive parameter that was examined was the type and thickness of the insert used to start the delamination. Because of the fiber bridging that develops in the unidirectional DCB specimen after the delamination grows from the end of the insert, the value of G_{Ic} measured at the initiation of delamination from the end of the insert was considered the only measured value representative of the interlaminar fracture toughness of the material being tested (see Ref *1*). In the first round, 26-μm (1.0 mil = 0.001 in.) Kapton film inserts were used for the AS4/3501-6 and AS4/BP907 specimens. However, data could only be obtained for the AS4/BP907, because the Kapton film layed up in

TABLE 1—*Summary of ASTM round robin data.*

Round	Material	Load Intro	# Labs	Tests/Lab	Insert	Avg. Mean G_{Ic}, kJ/m^2	S_r	$(CV)_r$ %	S_R	$(CV)_R$ %
1	AS4/BP907	Hinges	9	3	25 μm Kapton	0.400*	0.028	7.0	0.077	19.3
	AS4/BP907	T-Tabs	7	3	25 μm Kapton	0.410*	0.041	10.0	0.052	12.7
2	AS4/3501-6	Hinges	3	3	13 μm Kapton	0.085*	0.015	17.6	0.014	16.5
	AS4/PEEK	Hinges	2	4	13 μm Kapton	1.340*	0.139	10.4	0.211	15.7

*Visual values using compliance calibration (Berry's Method).

the AS4/3501-6 specimens was not sprayed with a mold release agent before curing. The absence of release agent resulted in specimens that were intermittently bonded in the insert area and, hence, no useful data were obtained from these specimens. The AS4/PEEK specimens tested in the first round had 39-μm (1.5-mil) thick folded aluminum foil inserts yielding a total insert thickness of 78 μm (3.0 mil). These inserts proved to be too thick to measure a useful initiation value and, hence, the first round AS4/PEEK data were of limited value.

In the second round, four distinct Kapton insert types were employed for AS4/3501-6 and AS4/PEEK specimens, resulting in three insert thicknesses. Inserts were either (1) 13-μm (0.5-mil) single layers sprayed with a mold release agent; (2) 13-μm (0.5-mil) layers folded in two to achieve a 26-μm (1.0-mil) thickness, or 26-μm (1.0-mil) single layers sprayed with a mold release agent; or (3) 26-μm (1.0-mil) layers folded in two to achieve a 52-μm (2.0-mil) thickness. Data from this round indicated that the 13-μm (0.5-mil) sprayed insert consistently yielded the most reasonable and conservative value of G_{Ic} for all materials. However, the thinner inserts consistently yielded lower G_{Ic} values, without giving an indication that a minimum plateau had been obtained, as was observed in the literature for glass epoxy laminates [*7*].

In the third round, both 7-μm (0.25-mil) and 13-μm (0.5-mil) aluminum inserts were sprayed with a mold release agent and implanted in AS4/PEEK panels. The panels for the ASTM and JIS participants were X-rayed to examine the conditions of the inserts. Unfortunately, these radiographs indicated that many tears and folds were present in the aluminum inserts. Only specimens that appeared to be free of insert tears and folds in the radiographs were distributed to the ASTM and JIS participants, thereby limiting the number of specimens available from each panel. Unfortunately, even the specimens with inserts, which appeared straight and flat in the radiographs, exhibited uncharacteristic *R*-curves and yielded questionable initiation G_{Ic} values. Examination of the polished edge of an untested specimen indicated a tendency for the aluminum inserts to fold or crimp, resulting in the formation of resin pockets (see Ref *1*). The specimens sent to the EGF participants were not X-rayed before they were tested, but yielded similar results. These data were summarized separately.

In the fourth round, several labs generated static DCB results on AS4/PEEK specimens with 13-μm (0.5-mil) sprayed Kapton inserts as part of an ASTM fatigue round robin. Attempts to manufacture AS4/3501-6 graphite epoxy laminates with Kapton inserts were unsuccessful for the same reasons previously noted.

In the fifth round, tests were conducted on AS4/PEEK specimens with both 7.5-μm (0.25-mil) and 13-μm (0.5-mil) Upilex inserts. This fifth round of testing yielded sufficient data on AS4/PEEK specimens with thin Upilex inserts to determine the precision of the DCB standard test method.

Results for each round of testing were summarized first in the form of a "box plot." These box plots were used simply to show trends in central tendency for groups of variables. A box plot represents each plotted variable as a separate box with a dark line drawn inside showing the median value of the variable and the top and bottom of the box representing the limits of +25% and −25% of the variable population. Lines extending from the top and bottom of the box mark the maximum and minimum for each variable. Typically, a maximum of 20 variables can be plotted in a box plot. For consistency, however, a box plot was used to show trends in central tendency for test matrices with more than 20 variables. This often resulted in isolated data points being shown discretely on the plot if they fell outside of the box. Mean G_{Ic} values and standard deviations for individual labs were then compared using bar charts. Finally, results for each round of testing were summarized in tables.

Results from Round 1

Nine labs each received three specimens to test where the load was introduced using piano hinges. Seven labs each received three specimens to test where the load was introduced using T-tabs. A first draft test procedure was sent to each lab. The data were reduced using a compliance calibration technique commonly known as Berry's method [*8*].

Figures 2 and 3 show the visually observed initiation G_{Ic} values, measured from a 26-μm (1.0-mil) Kapton insert, for the AS4/BP907 DCB specimens. Figure 4 shows a comparison of mean G_{Ic} values for the six labs that performed tests with both configurations. Figure 5 shows the standard deviation in the data

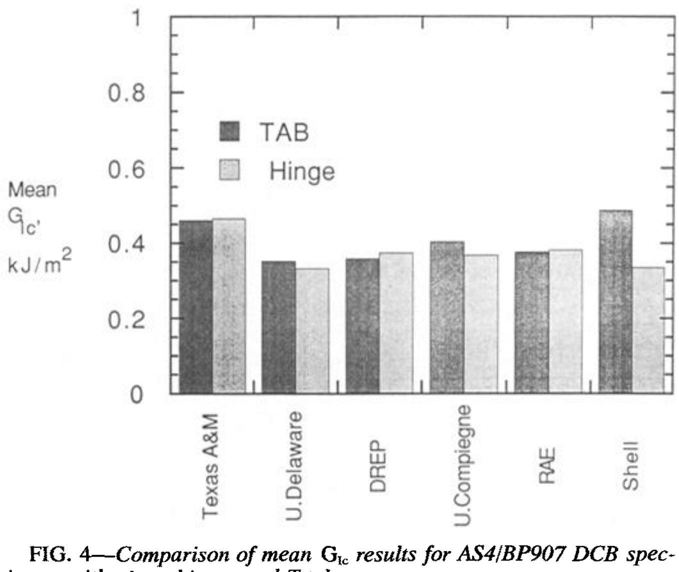

FIG. 4—*Comparison of mean* G_{Ic} *results for AS4/BP907 DCB specimens with piano hinges and T-tabs.*

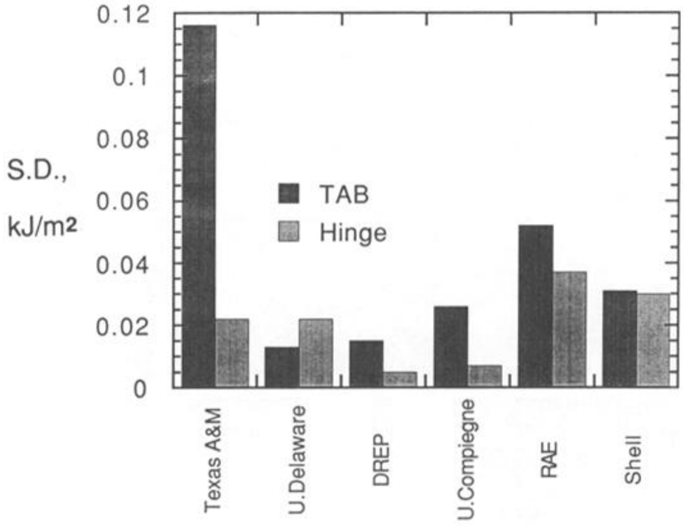

FIG. 5—*Comparison of standard deviation in* G_{Ic} *results for AS4/BP907 DCB specimens with piano hinges and T-tabs.*

obtained for these six labs. The data for all the labs that performed tests are summarized in Table 1. Similar mean G_{Ic} values were obtained for both configurations. Also summarized in Table 1 are the variability in mean G_{Ic} values for the piano hinge and T-tab configurations in the form of standard deviations within a given laboratory, S_r, (a measure of repeatability) and the standard deviations between laboratories, S_R, (a measure of reproducibility). These measures of repeatability and reproducibility are required to obtain an estimate of the precision of the test method as specified by ASTM E 691, Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method. The data are also summarized in Table 1 as coefficients of variation (CV) calculated by dividing the standard deviation by the mean G_{Ic} value. Coefficients of variation are tabulated corresponding to the repeatability, $(CV)_r$, and reproducibility, $(CV)_R$.

Results from Round 2

Three labs each received three specimens to test where the load was introduced using piano hinges. Figure 6 shows the G_{Ic} values measured by visually observing delamination onset at the edge of DCB specimens of AS4/3501-6 with 13-μm (0.5-mil) sprayed, 13-μm (0.5-mil) folded, 26-μm (1.0-mil) sprayed, and 26-μm (1.0-mil) folded Kapton inserts. The results indicate that the 13-μm (0.5-mil) sprayed inserts yield the lowest mean values; the 13-μm (0.5-mil) folded, 26-μm (1.0-mil) sprayed inserts, both of which result in a 26-μm (1.0-mil) insert thickness, yield higher mean values; and the 26-μm (1.0-mil) folded inserts, which result in a 52-μm (2.0-mil) insert thickness, yield the highest mean values and have the greatest scatter. Figures 7 and 8 show the mean G_{Ic} values and standard deviation, respectively, for each of the three labs that performed the tests. Figure 9 compares the G_{Ic} values measured from the 13-μm (0.5-mil) sprayed insert for the three labs. The mean G_{Ic} values measured from the 13-μm (0.5-mil) sprayed insert for the three labs, and the statistical measures of repeatability within a given laboratory, S_r, and the reproducibility between laboratories, S_R, are summarized in Table 1. Also summarized in Table 1 are the coefficients of variation corresponding to repeatability within a given

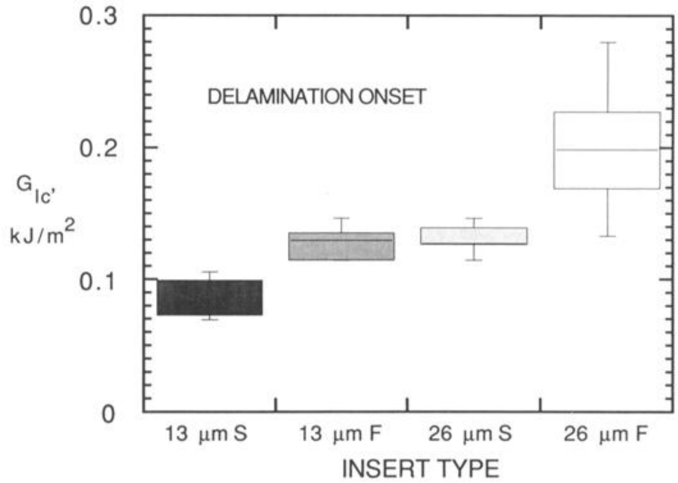

FIG. 6—*Box plot showing* G_{Ic} *round robin results for AS4/3501-6 DCB tests using different insert types.*

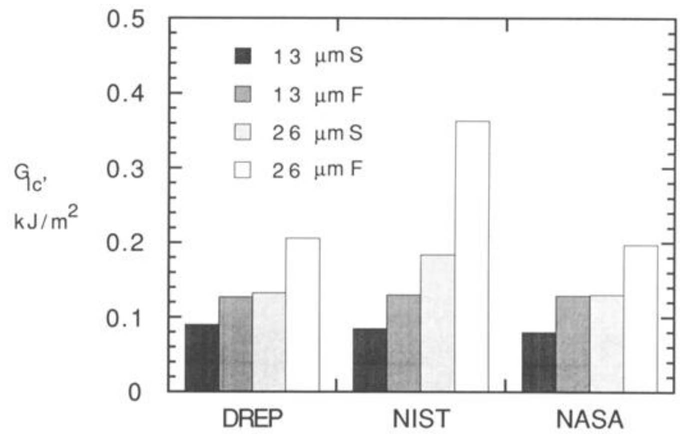

FIG. 7—*Comparison of mean* G_{Ic} *results for AS4/3501-6 DCB specimens with different insert types.*

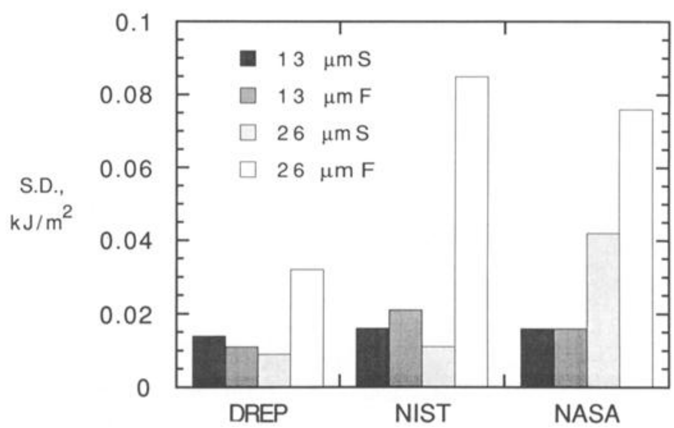

FIG. 8—*Comparison of standard deviation in* G_{Ic} *results for AS4/3501-6 DCB specimens with different insert types.*

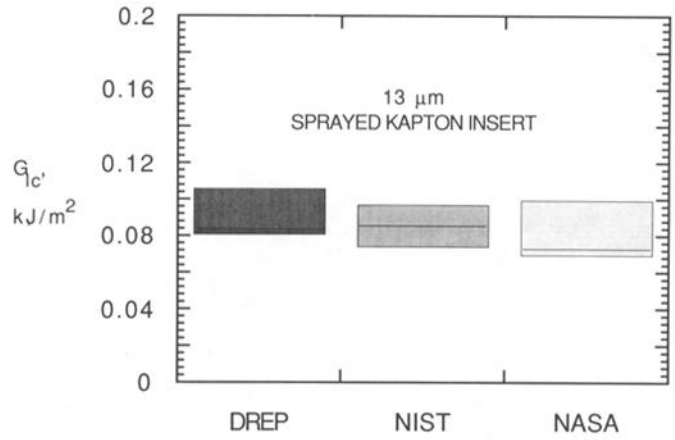

FIG. 9—*Box plot showing* G_{Ic} *round robin results for AS4/3501-6 DCB tests 13-μm sprayed Kapton inserts.*

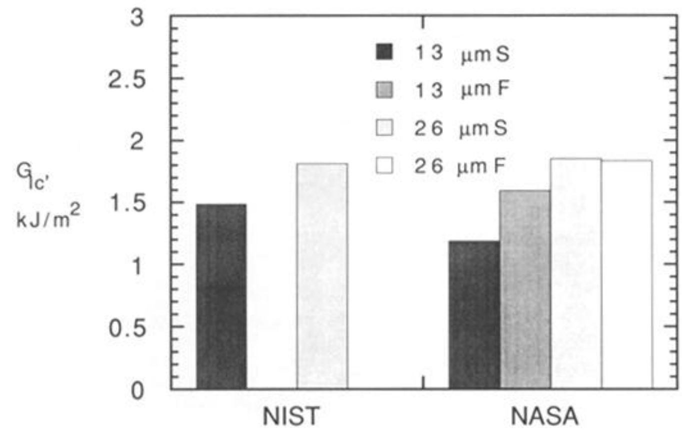

FIG. 11—*Comparison of mean* G_{Ic} *results for AS4/PEEK DCB specimens with different insert types.*

laboratory, $(CV)_r$, and the reproducibility between laboratories, $(CV)_R$.

Figure 10 shows the G_{Ic} values measured by visually observing delamination onset at the edge of DCB specimens of AS4/PEEK with 13-μm (0.5-mil) sprayed, 13-μm (0.5-mil) folded, 26-μm (1.0-mil) sprayed, and 26-μm (1.0-mil) folded Kapton inserts. Because of difficulties manufacturing these panels, there were only enough specimens for two labs, with only one lab testing all four insert types. Each lab tested four specimens per insert type. The data from the two labs that performed the tests are included in Fig. 10. The results indicate that the 13-μm (0.5-mil) sprayed inserts yield the lowest mean values; the 13-μm (0.5-mil) folded, 26-μm (1.0-mil) sprayed inserts, both of which result in a 26-μm (1.0-mil) insert thickness, yield slightly higher mean values; and the 26-μm (1.0-mil) folded inserts, which result in a 52-μm (2.0-mil) insert thickness, yield the highest mean values.

Figure 11 shows the mean G_{Ic} values for the four insert configurations for the two labs that performed the tests. The mean G_{Ic} values measured from the 13-μm (0.5-mil) sprayed insert for the two labs, and the standard deviations for repeatability and reproducibility are shown in Table 1. The coefficients of variation corresponding to repeatability, $(CV)_r$, and reproducibility, $(CV)_R$, are also shown in Table 1. However, a larger data set was needed to obtain an accurate estimate of the repeatability and reproducibility between laboratories.

Unlike the tests on AS4/3501-6 and AS4/BP907, the load deflection curves for the AS4/PEEK DCB tests became nonlinear before the delamination was visually observed to initiate from the insert on the edge of the specimen (Fig. 12). Hence, several different initiation measurements, as well as several different data reduction methods, were proposed for reducing data from DCB tests on AS4/PEEK in round 3. As a prelude to the third round, these initiation measurements and data reduction methods were used to plot the data generated on 13-μm (0.5-mil) sprayed Kapton insert tests from addition tests conducted by three labs, each testing four specimens, during round 2.

Figure 13 shows G_{Ic} values measured using the load at onset of nonlinearity (NL), the load at visual observation of delamination onset at the edge (VIS), and the load corresponding to a 5% offset in the initial compliance of the DCB specimen (5%).

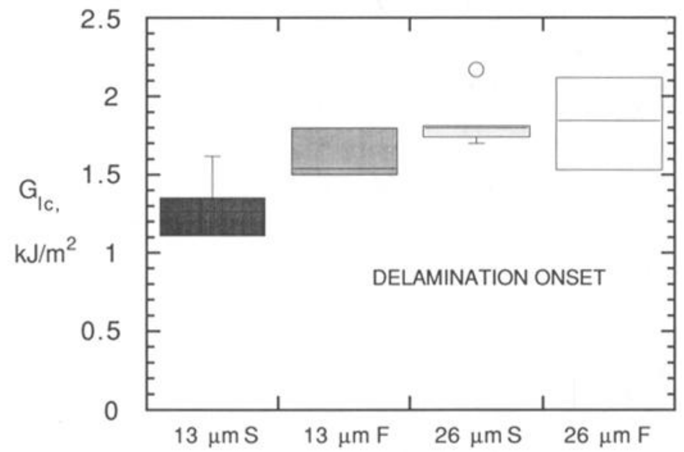

FIG. 10—*Box plot showing* G_{Ic} *round robin results for AS4/PEEK DCB tests using different insert types.*

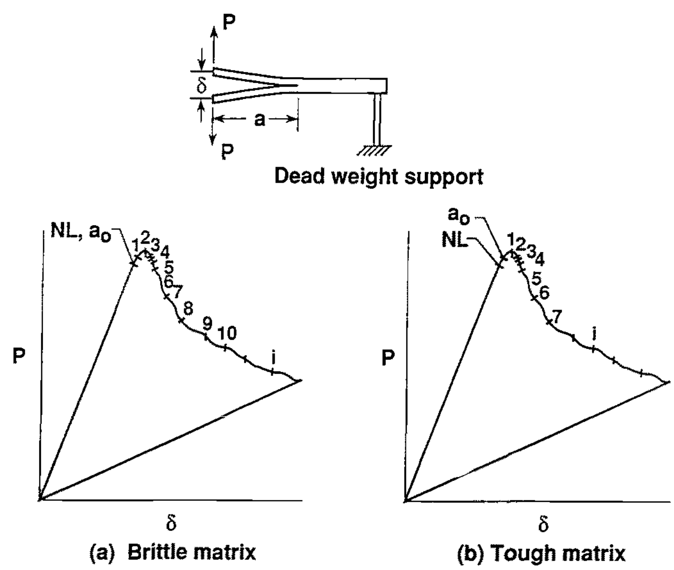

FIG. 12—*Load displacement trace from typical DCB tests.*

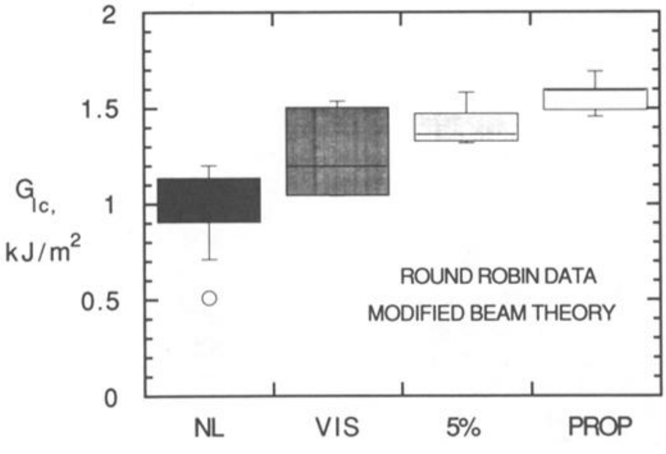

FIG. 13—*Box plot showing* G_{Ic} *round robin values for AS4/PEEK DCB tests with 13-μm sprayed Kapton inserts.*

These data were reduced using a modified beam theory (MBT) technique [*9*]. The MBT technique yielded slightly lower mean G_{Ic} values than Berry's method (BRY) for the same test data (see Ref *1*). Also shown in Fig. 13 are the plateau values of G_{Ic} (PLAT) corresponding to stabilized delamination growth in the presence of fiber bridging. The mean NL G_{Ic} value for the three labs that tested the 13-μm (0.5-mil) sprayed Kapton insert specimens is listed in Table 2. Also shown in Table 2 are the coefficients of variation for repeatability and reproducibility. However, a larger data set was needed to obtain an accurate estimate of the repeatability and reproducibility between laboratories.

Results from Round 3

International Round Robin (ASTM and JIS)

Tests performed by ASTM and JIS participants using specimens that were X-rayed and appeared to have no tears or folds in the aluminum inserts are summarized first. The 13-μm (0.5-mil) insert specimens were tested by 16 labs, whereas the 7-μm (0.25-mil) specimens were tested by five labs. Each lab received four specimens to test per insert thickness.

Figure 14 shows the mean NL G_{Ic} values for the 16 labs that tested the 13-μm aluminum insert specimens. Figure 15 shows the mean NL G_{Ic} values for the five labs that tested the 7-μm aluminum insert specimens. The data were reduced using three data reduction methods: (1) MBT, (2) BRY, and (3) a Modified

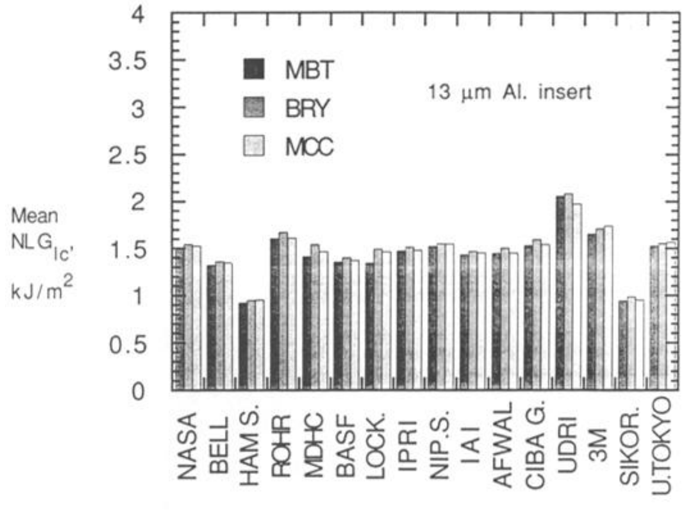

FIG. 14—*Comparison of mean NL* G_{Ic} *results for AS4/PEEK DCB specimens with 13-μm aluminum inserts calculated using three different data reduction methods.*

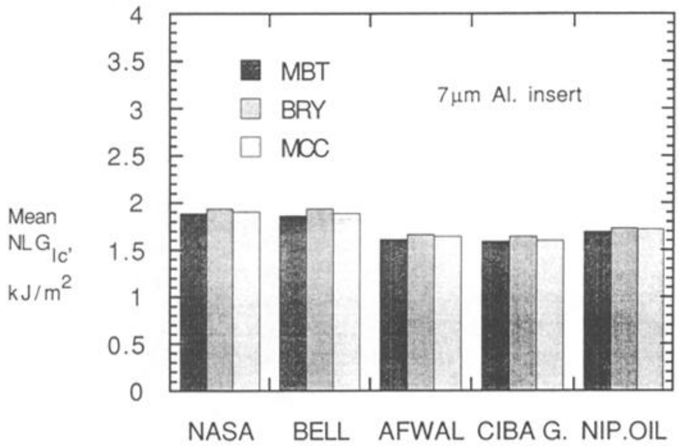

FIG. 15—*Comparison of mean NL* G_{Ic} *results for AS4/PEEK DCB specimens with 7-μm aluminum inserts calculated using three different data reduction methods.*

Compliance Calibration (MCC) method [*10*]. For both insert thicknesses, the variation between the three data reduction methods for any single lab was no greater than 3.1%. Hence, none of the three data reduction techniques was clearly superior to

TABLE 2—*Summary of AS4/PEEK round robin data.*

Round	Round Robin	# Labs	Tests/ Lab	Insert	Value	Avg. Mean G_{Ic}, kJ/m^2	S_r	$(CV)_r$ %	S_R	$(CV)_R$ %
2	ASTM	3	4	13 μm Kapton	NL	0.983*	0.132	13.4	0.178	18.1
3	ASTM/JIS	16	4	13 μm Al Foil	NL	1.439*	0.187	13.4	0.261	18.1
	ASTM/JIS	16	4	13 μm Al Foil	VIS	1.724*	0.232	13.5	0.194	11.3
	ASTM/JIS	16	4	13 μm Al Foil	5%	1.799*	0.213	11.8	0.146	8.1
	ASTM/JIS	5	4	7 μm Al Foil	NL	1.727*	0.226	13.0	0.140	8.1
	ASTM/JIS	5	4	7 μm Al Foil	VIS	1.929*	0.257	13.3	0.201	10.4
	ASTM/JIS	5	4	7 μm Al Foil	5%	2.059*	0.218	10.9	0.218	10.6

*All G_{Ic} values determined using Modified Beam Theory (MBT).

the others. However, because the MBT method yielded the most conservative values of G_{Ic} for 80% of the specimens tested, the remaining data in this report are summarized using the MBT method only.

13-μm Aluminum Insert Results—Figure 16 summarizes G_{Ic} values measured using the load at onset of nonlinearity (NL), the load at visual observation of delamination onset at the edge (VIS), and the load corresponding to a 5% offset in the initial compliance of the DCB specimen (5%) for the 16 labs that conducted tests on 13-μm (0.5-mil) aluminum insert specimens. Each lab received four specimens to test. Figure 17 shows the mean G_{Ic} values for the 16 labs that tested the 13-μm aluminum insert specimens. Figure 18 shows the standard deviation in the data reported by each of these 16 labs.

The mean NL, VIS, and 5% offset G_{Ic} values measured from the 13-μm (0.5-mil) aluminum insert for the 16 labs, and the standard deviations for repeatability and reproducibility, are listed in Table 2. Also summarized in Table 2 are the coefficients of variation for repeatability and reproducibility.

As noted in Table 2, the variability between laboratories was greater for the NL onset measurements than for the VIS or 5% offset measurements. However, the average mean NL G_{Ic} value was significantly lower than the VIS and 5% offset G_{Ic} values. Figure 19 shows the percentage difference between the NL and 5% G_{Ic} values. The average difference in G_{Ic} was 20.2%.

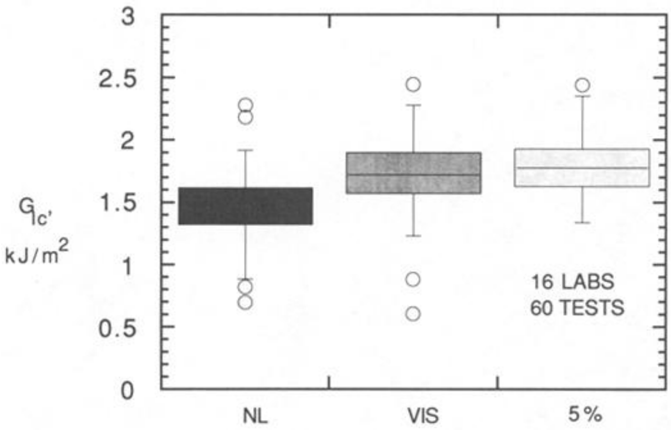

FIG. 16—*Box plot showing* G_{Ic} *round robin values for AS4/PEEK DCB tests with 13-μm aluminum inserts.*

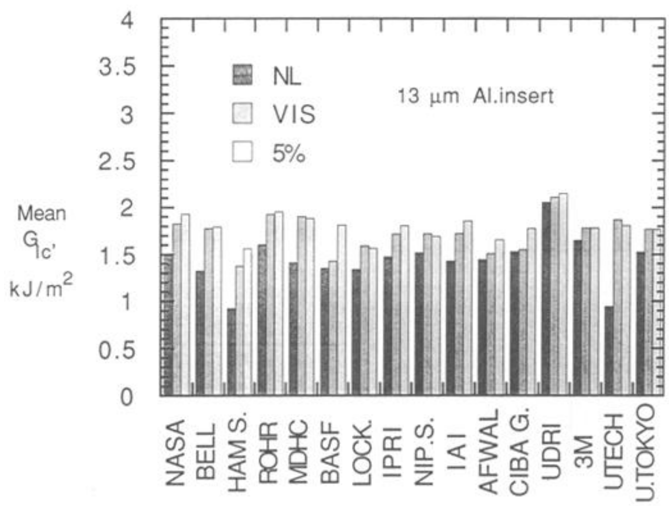

FIG. 17—*Comparison of mean* G_{Ic} *values for AS4/PEEK DCB specimens with 13-μm aluminum inserts.*

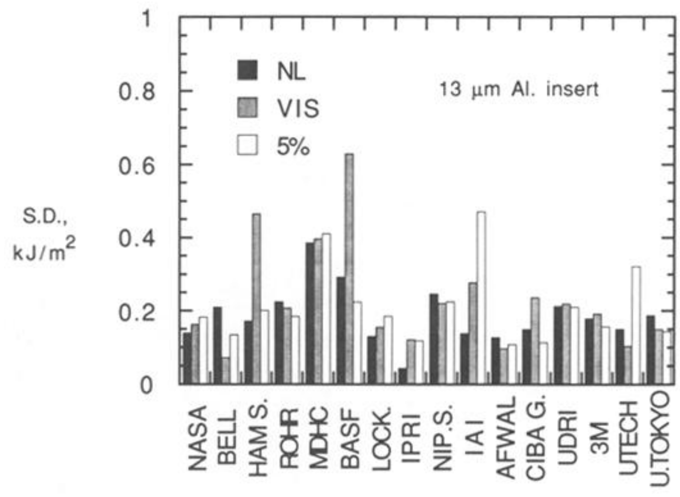

FIG. 18—*Comparison of standard deviation in* G_{Ic} *values for AS4/PEEK DCB specimens with 13-μm aluminum inserts.*

In 73% of the tests with the 13-μm (0.5-mil) aluminum inserts, propagation values of G_{Ic}, corresponding to delamination growth in the presence of fiber bridging, were lower than NL or VIS, or both, onset values. In 70% of the individual tests, the VIS G_{Ic} values were lower than the 5% offset values.

7-μm Aluminum Insert Results—Figure 20 summarizes G_{Ic} values measured using the load at onset of nonlinearity (NL), the load at visual observation of delamination onset at the edge (VIS), and the load corresponding to a 5% offset in the initial compliance of the DCB specimen (5%) for the five labs that conducted tests on specimens with the 7-μm (0.25-mil) aluminum inserts. Each lab tested four specimens. Figure 21 shows the mean G_{Ic} values for the five labs. Figure 22 shows the standard deviation in the data reported by each of these five labs.

The mean NL, VIS, and 5% offset G_{Ic} values measured from

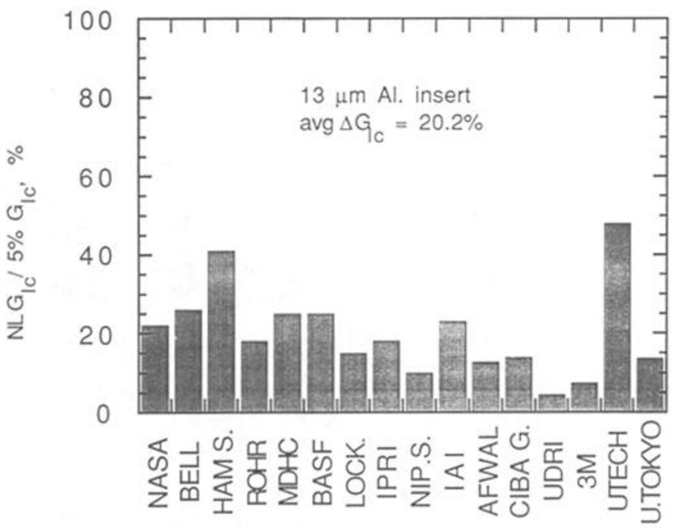

FIG. 19—*Percentage difference in NL and 5% offset* G_{Ic} *values for AS4/PEEK DCB specimens with 13-μm aluminum inserts.*

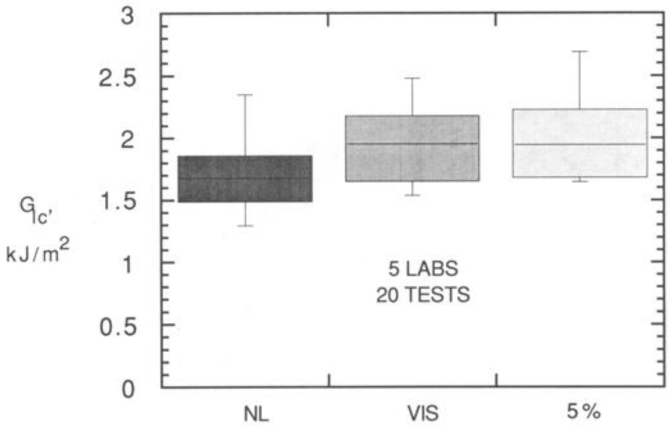

FIG. 20—*Box plot showing* G_{Ic} *round robin values for AS4/PEEK DCB tests with 7-μm aluminum inserts.*

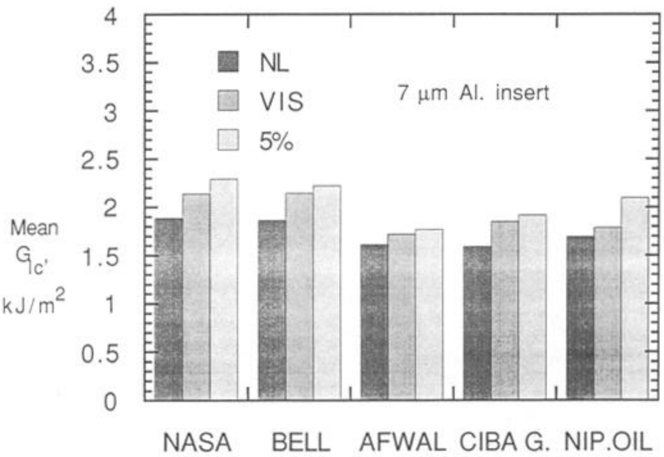

FIG. 21—*Comparison of mean* G_{Ic} *values for AS4/PEEK DCB specimens with 7-μm aluminum inserts.*

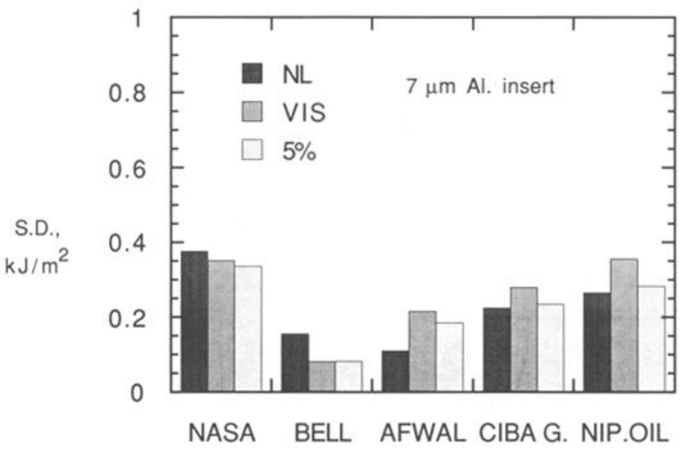

FIG. 22—*Comparison of standard deviation in* G_{Ic} *values for AS4/PEEK DCB specimens with 7-μm aluminum inserts.*

the 7-μm (0.25-mil) aluminum insert for the five labs, and the standard deviation corresponding to the repeatability and reproducibility, are listed in Table 2. Also summarized in Table 2 are the coefficients of variation for repeatability and reproducibility.

The reproducibility and repeatability were similar for all three onset measurements. However, as was noted for the 13-μm (1.0-mil) insert specimens, the NL G_{Ic} values were significantly lower than the VIS and 5% offset G_{Ic} values. In 75% of the tests with the 7-μm (0.25-mil) aluminum inserts, propagation values of G_{Ic}, corresponding to delamination growth in the presence of fiber bridging, were lower than NL or VIS, or both, onset values. The VIS G_{Ic} values were lower than the 5% offset values in 100% of the individual tests.

In nearly 75% of the tests with aluminum inserts, PLAT values of G_{Ic} corresponding to delamination growth in the presence of fiber bridging were lower than NL or VIS, or both, onset values. This resulted in an *R*-curve, a plot of G_{Ic} as a function of delamination length, that rose and then decreased below VIS or NL G_{Ic} values, or both (Fig. 23). In contrast, *R*-curves for specimens with Kapton inserts always achieved PLAT G_{Ic} values above the NL and VIS G_{Ic} values (Fig. 24). Microscopy studies performed at ICI on untested specimens indicated that localized yielding (crimping) may have occurred during cutting of the aluminum foil inserts (See Ref *1*). These crimps, which were not evident in the original panel radiographs, were responsible

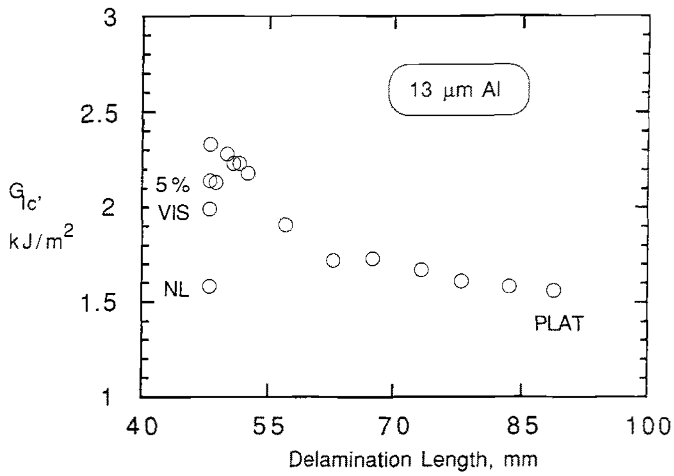

FIG. 23—R-*curve for AS4/PEEK DCB specimens with 13-μm aluminum inserts.*

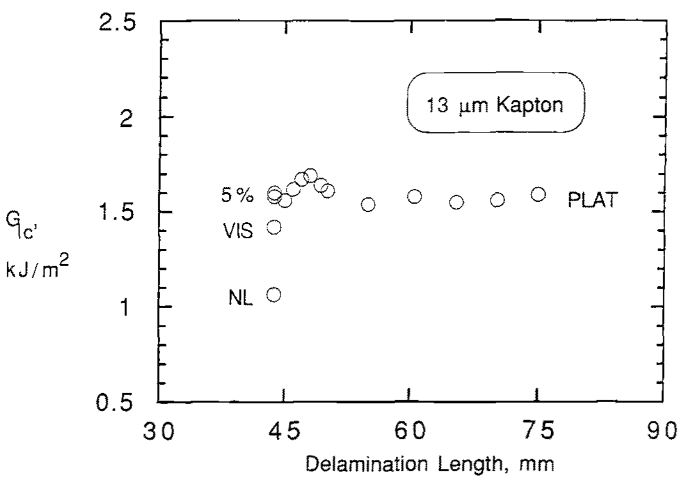

FIG. 24—R-*curve for AS4/PEEK DCB specimens with 13-μm Kapton inserts.*

TABLE 3—*Summary of AS4/PEEK round robin data.*

Round	Round Robin	# Labs	Tests/ Lab	Insert	Value	Avg. Mean G_{Ic}, kJ/m²	S_r	$(CV)_r$ %	S_R	$(CV)_R$ %
3	EGF	6	4	13 μm Al Foil	NL	1.574*	0.230	14.6	0.283	18.0
	EGF	6	4	13 μm Al Foil	VIS	1.953*	0.209	10.7	0.286	14.6
	EGF	6	4	13 μm Al Foil	5%	1.938*	0.202	10.4	0.220	11.4
	EGF	4	4	7 μm Al Foil	NL	1.575*	0.160	10.2	0.121	7.7
	EGF	4	4	7 μm Al Foil	VIS	1.883*	0.150	8.0	0.154	8.2
4	ASTM	10	3	13 μm Kapton	NL	1.303*	0.180	13.8	0.207	15.9
	ASTM	10	3	13 μm Kapton	VIS	1.549*	0.198	12.8	0.151	9.7
	ASTM	10	3	13 μm Kapton	5%	1.713*	0.171	10.0	0.139	8.1

*All G_{Ic} values determined using MBT.

for the formation of resin pockets at the end of the inserts resulting in elevated G_{Ic} values. The tendency of aluminum inserts to crimp when cut may be worse in the thinner 7-μm foils, which yield higher apparent G_{Ic} values than the 13-μm foils (Table 2).

International Round Robin (EGF)

The 13-μm (0.5-mil) insert specimens were tested by six labs, whereas the 7-μm (0.25-mil) insert specimens were tested by four labs. Each lab received four specimens to test. The data were reduced using the MBT method. These tests, performed by EGF participants using specimens that were not X-rayed to isolate specimens with tears or folds in the aluminum inserts, yielded results similar to those obtain by ASTM and JIS participants. Hence, only a summary of these results are presented in this report. A detailed description of the results may be found in Ref *1*.

The mean NL, VIS, and 5% offset G_{Ic} values measured from the 13-μm (0.5-mil) aluminum insert for the six labs, and the standard deviations for repeatability and reproducibility, are listed in Table 3. Also summarized in Table 3 are the coefficients of variation for repeatability and reproducibility. The variability between laboratories was greater for the NL onset measurements than for the VIS or 5% offset measurements. However, as noted for the ASTM/JIS results, the average mean NL G_{Ic} value was significantly lower than the VIS and 5% values.

The mean NL, VIS, and 5% offset G_{Ic} values measured from the 7-μm (0.25-mil) aluminum insert for the four labs, and the standard deviation corresponding to the repeatability and reproducibility, are listed in Table 3. Also summarized in Table 3 are the coefficients of variation for repeatability and reproducibility. The reproducibility and repeatability between laboratories were similar for the NL and VIS onset measurements. However, as noted for the ASTM/JIS results, the average mean NL G_{Ic} value was significantly lower than the VIS values.

Results from Round 5

For this round, 13-μm Kapton polyimide film inserts were sprayed with a mold release agent and were implanted before consolidation of the AS4/PEEK panels. Three static DCB tests were performed by each of ten labs as part of an ASTM DCB fatigue round robin. For all these tests, *R*-curves achieved PLAT G_{Ic} values above the NL and VIS G_{Ic} values (Fig. 24).

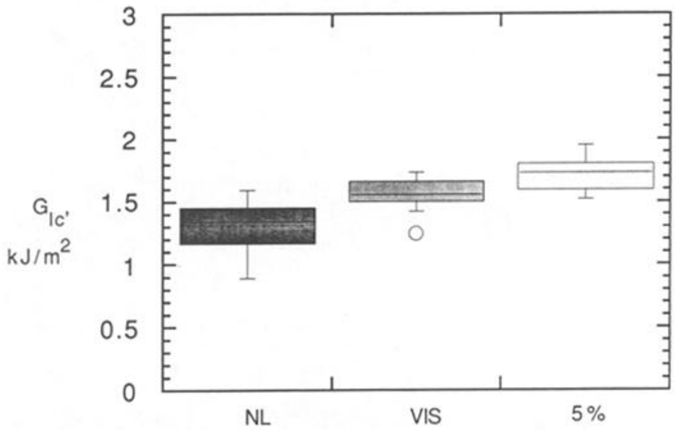

FIG. 25—*Box plot showing* G_{Ic} *fatigue round robin values for AS4/PEEK static DCB tests with 13-μm Kapton inserts.*

Figure 25 summarizes G_{Ic} values measured using the load at onset of nonlinearity (NL), the load at visual observation of delamination onset at the edge (VIS), and the load corresponding to a 5% offset in the initial compliance of the DCB specimen (5%) for the ten labs that each conducted tests on three specimens with the 13-μm (0.5-mil) Kapton inserts as part of the DCB fatigue round robin. Figure 26 shows the mean G_{Ic} values for the ten labs. Figure 27 shows the standard deviation in the data reported by each of the ten labs.

The mean NL, VIS, and 5% offset G_{Ic} values measured from the 13-μm (0.5-mil) Kapton insert for the ten labs, and the standard deviations for repeatability and reproducibility, are listed in Table 3. Also summarized in Table 3 are the coefficients of variation for repeatability and reproducibility. The variability between laboratories was greater for the NL onset measurements than for the VIS or 5% offset measurements. However, the average mean NL G_{Ic} value was significantly lower than the VIS and 5% values.

The reproducibility and repeatability of data from this round robin were similar to earlier round robins conducted with 13-μm inserts. However, the mean NL G_{Ic} values for this round robin were lower than the mean NL G_{Ic} values obtained with the aluminum inserts, but higher than those obtained from specimens with Kapton inserts in the original ASTM round robin. However, none of the two round robins conducted on specimens with Kapton inserts satisfied the requirements for a database to justify the precision statement for an ASTM standard (see

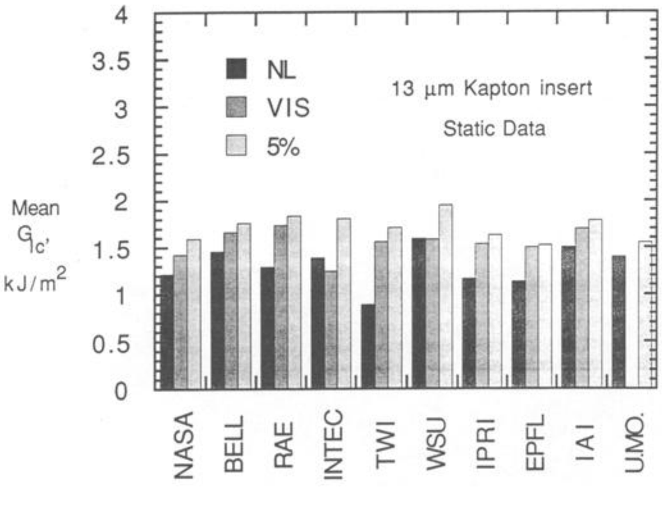

FIG. 26—*Comparison of mean* G_{Ic} *values for AS4/PEEK DCB specimens with 13-µm Kapton inserts.*

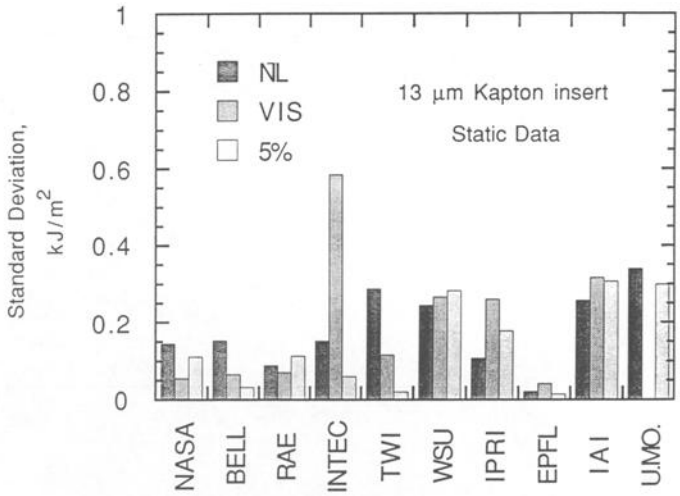

FIG. 27—*Comparison of standard deviation in* G_{Ic} *values for AS4/PEEK DCB specimens with 13-µm Kapton inserts.*

ASTM E 691). The required database includes a minimum of five tests conducted by at least six different laboratories. In order to generate the required database, and to quantify the sensitivity of G_{Ic} to insert thickness, a second international round robin was conducted.

Results from Round 5

For this round robin, both 7.5- and 13-µm Upilex polyimide film inserts were sprayed with a mold release agent and were implanted before consolidation of the AS4/PEEK panels. Five specimens of each thickness insert were tested by nine labs. Each lab conducted the tests according to a draft ASTM DCB standard. For all DCB tests with Upilex inserts, *R*-curves achieved PLAT G_{Ic} values above the NL and VIS G_{Ic} values similar to results for specimens with Kapton inserts (Fig. 24).

Figures 28 and 29 summarize G_{Ic} values measured using the load at onset of nonlinearity (NL), the load at visual observation of delamination onset at the edge (VIS), and the load corresponding to a 5% offset in the initial compliance of the DCB specimen (5%) for the nine labs that each conducted tests on five specimens with the 13-µm and 7.5-µm Upilex inserts. Figures 30 and 31 show the mean G_{Ic} values for the nine labs. Figures 32 and 33 show the standard deviation in the data reported by each of the nine labs.

The mean NL, VIS, and 5% offset G_{Ic} values measured from the 13-µm and 7.5-µm Upilex inserts for the nine labs, and the standard deviations for repeatability and reproducibility, are listed in Table 4. Also summarized in Table 4 are the coefficients of variation for repeatability and reproducibility. The variability between laboratories was greater for the NL onset measurements than for the VIS or 5% offset measurements. However, the average mean NL G_{Ic} values was significantly lower than the VIS and 5% values.

The reproducibility and repeatability of data from this round was as good as, and in many cases better than, the earlier rounds. Mean NL G_{Ic} values for this round robin were lower than obtained from all the previous round robins except for the original ASTM round robin with Kapton inserts. The average NL G_{Ic} values for the 7.5-µm Upilex insert specimens were 6.3% lower

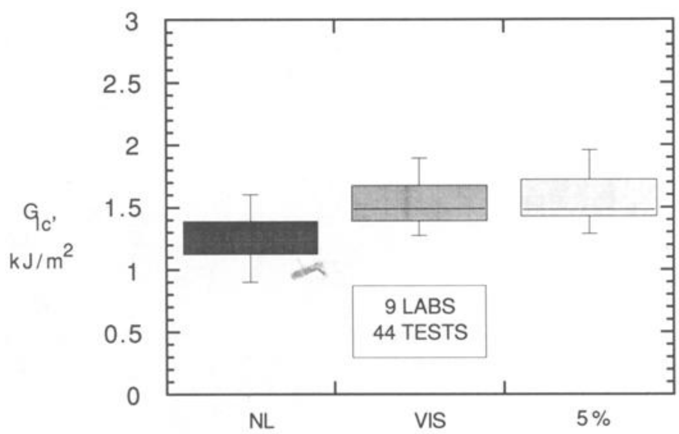

FIG. 28—*Box plot showing* G_{Ic} *round robin values for AS4/PEEK DCB tests with 13-µm Upilex inserts.*

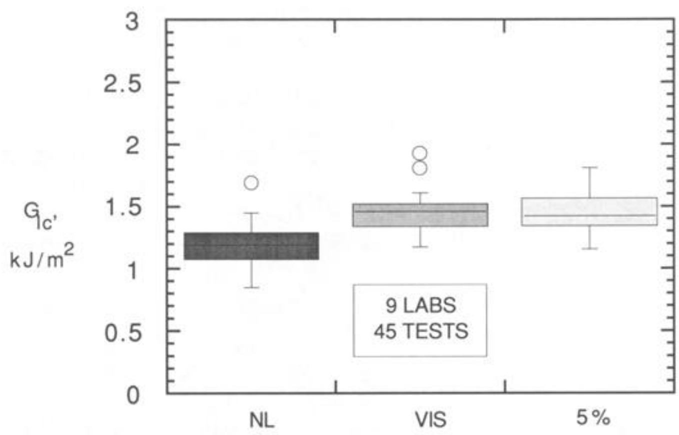

FIG. 29—*Box plot showing* G_{Ic} *round robin values for AS4/PEEK DCB tests with 7.5-µm Upilex inserts.*

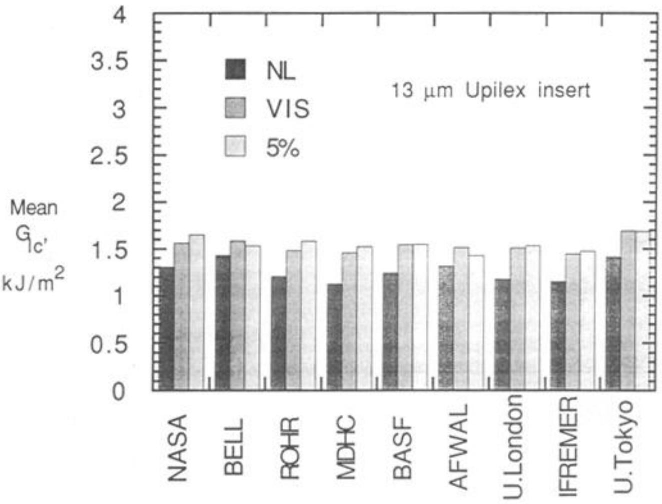

FIG. 30—*Comparison of mean* G_{Ic} *values for AS4/PEEK DCB specimens with 13-μm Upilex inserts.*

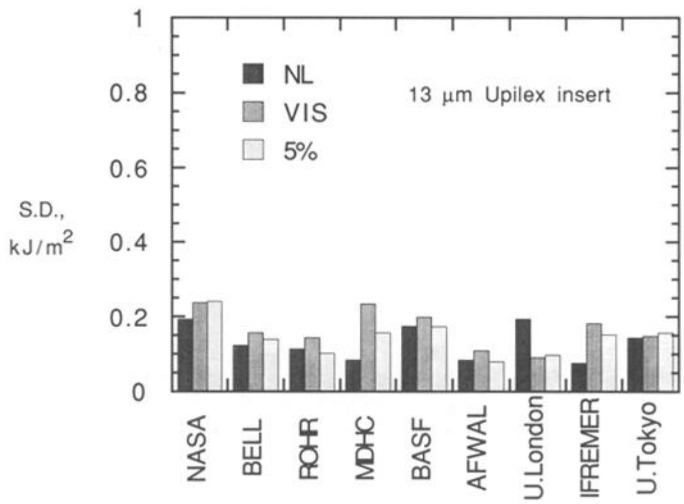

FIG. 32—*Comparison of standard deviation in* G_{Ic} *values for AS4/PEEK DCB specimens with 13-μm Upilex inserts.*

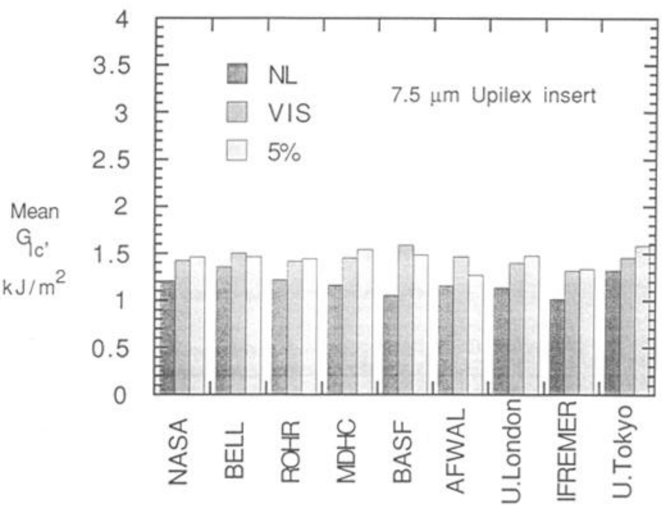

FIG. 31—*Comparison of mean* G_{Ic} *values for AS4/PEEK DCB specimens with 7.5-μm Upilex inserts.*

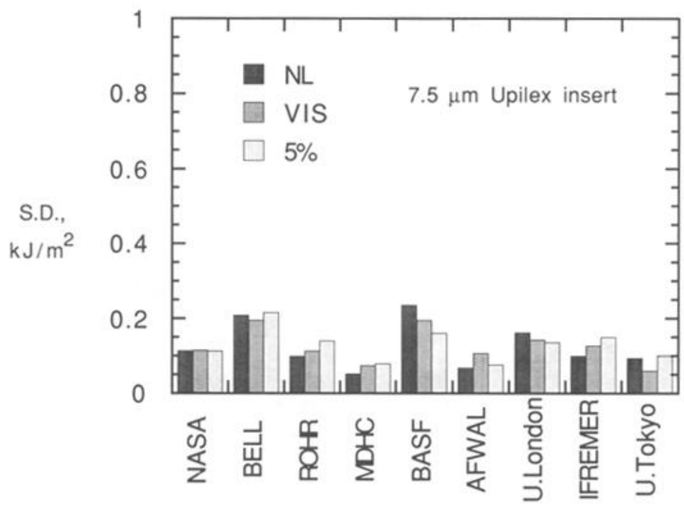

FIG. 33—*Comparison of standard deviation in* G_{Ic} *values for AS4/PEEK DCB specimens with 7.5-μm Upilex inserts.*

than the average NL G_{Ic} values for the 13-μm Upilex insert specimens.

Summary

As a result of round 5, the draft DCB standard was updated and submitted for ballot within ASTM subcommittee D30.06 on Interlaminar Properties in June of 1992 and then, following resolution of negative votes, was resubmitted for concurrent subcommittee and D30 main committee ballot in December of 1992. Guidelines were included in the standard for choosing piano hinges, blocks, or t-tabs for load introduction. The standard includes generation of the complete *R*-curve for each test and reporting of G_{Ic} values measured using the load at onset of nonlinearity in the load versus displacement plot (NL), the load at visual observation of delamination onset at the edge (VIS), and the load corresponding to a 5% offset in the initial compliance of the DCB specimen (5%). However, the standard makes several recommendations.

First, because of the difficulty initiating delaminations in brittle epoxy matrix composites from polyimide (Kapton) films sprayed with a mold release agent, PTFE (Teflon®) film inserts were recommended for these materials. Polyimide films are recommended only for materials with high cure (or consolidation) temperatures.

Second, because specimens with insert thicknesses greater than 13 μm yield unrealistically high G_{Ic} values, and because the difference in average NL G_{Ic} values for 7.5- and 13-μm Upilex inserts was relatively small (6.3%), an insert thickness requirement of 13 μm or less was adopted for the ASTM DCB standard. The 7.0 to 7.5-μm inserts were optional because they represent minimum polyimide film thicknesses that are presently commercially available. Furthermore, these ultra-thin films are typically more difficult to obtain, and are considerably more difficult to handle than the 13-μm films. The polyimide films were recommended over the aluminum films for use as inserts in the DCB test because of the problems with crimping, tears, and folds in aluminum inserts noted in the first international round robin.

TABLE 4—*Summary of AS4/PEEK round robin data.*

Round	Round Robin	# Labs	Tests/ Lab	Insert	Value	Avg. Mean G_{Ic}, kJ/m^2	S_r	$(CV)_r$ %	S_R	$(CV)_R$ %
5	ASTM/JIS/EGF	9	5	13 μm Upilex	NL	1.262*	0.132	10.5	0.110	8.7
	ASTM/JIS/EGF	9	5	13 μm Upilex	VIS	1.532*	0.167	10.9	0.075	4.9
	ASTM/JIS/EGF	9	5	13 μm Upilex	5%	1.549*	0.144	9.3	0.080	5.1
	ASTM/JIS/EGF	9	5	7.5 μm Upilex	NL	1.182*	0.126	10.7	0.111	9.4
	ASTM/JIS/EGF	9	5	7.5 μm Upilex	VIS	1.447*	0.126	8.7	0.075	5.2
	ASTM/JIS/EGF	9	5	7.5 μm Upilex	5%	1.451*	0.130	9.0	0.096	6.6

*All G_{Ic} values determined using MBT.

Third, three data reduction methods for calculating G_{Ic} values were included. These consisted of a Modified Beam Theory (MBT), a Compliance Calibration method (CC), and a Modified Compliance Calibration method (MCC). Because G_{Ic} values determined by the three different data reduction methods differed by no more than 3.1%, none of the three was clearly superior to the others. However, because of the MBT method yielded the most conservative values of G_{Ic} for 80% of the specimens tested, this method was recommended as the preferred data reduction technique. The area method was not recommended because it did not yield an initiation value of G_{Ic} or a delamination resistance curve.

Fourth, the NL G_{Ic} value was recommended as the preferred measure of Mode I interlaminar fracture toughness for generating delamination failure criteria in durability and damage tolerance analyses of laminated composite structures. However, all three initiation values may be used for the other purposes cited in the scope, such as identifying the effects of fiber surface treatment, local variations in fiber volume fraction, and processing and environmental variables on G_{Ic} and comparing quantitatively the relative values of G_{Ic} for composite materials with different constituents.

The preference for NL G_{Ic} values for durability and damage tolerance analyses is based on physical observations, made using video based *in-situ* dye penetrant enhanced X-radiography, that the delaminations initiate in the DCB specimen at the end of the insert, in the interior of the specimen width, when the load deflection curve becomes nonlinear [*11–14*]. The difference in NL and VIS G_{Ic} values is negligible for brittle epoxy matrix composites, but the difference is significant for tough thermoplastic matrix composites. As shown in Figure 34, mean VIS and mean 5% offset G_{Ic} values were typically 18 to 22% higher than mean NL G_{Ic} values even though VIS and 5% measurements were more repeatable (Fig. 35). Hence, The NL G_{Ic} values are conservative values corresponding to the first onset of delamination.

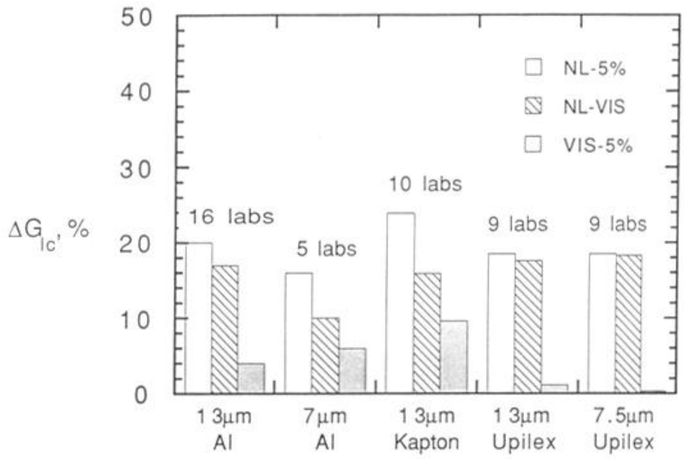

FIG. 34—*Percentage variation in mean* G_{Ic} *initiation values for AS4/PEEK laminates with different insert types.*

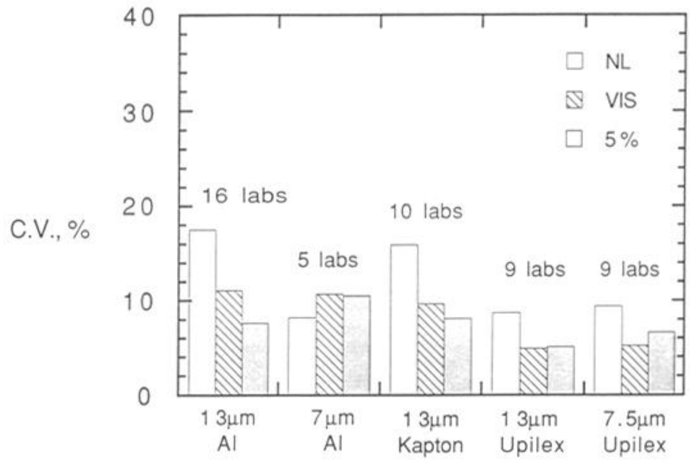

FIG. 35—*Coefficient of variation in* G_{Ic} *initiation values for AS4/PEEK laminates with different insert types.*

APPENDIX

List of Round Robin Participants

Round 1

1. NASA Langley Research Center
2. Texas A&M University
3. Defense Research Establishment Pacific (DREP) Canada
4. University of Compiegne, France
5. Royal Aerospace Establishment (RAE) England
6. Shell Development Company
7. Boeing Commercial Airplane Co.
8. Imperial Chemicals Industries (ICI) England
9. Rohr Industries, Inc.
10. University of Delaware

Round 2

1. NASA Langley Research Center
2. Texas A&M University
3. Defense Research Establishment Pacific (DREP) Canada
4. National Institute for Standards Technology (NIST)

Round 3

ASTM/JIS

1. NASA Langley Research Center
2. Bell Helicopter Co.
3. Hamilton Standard (HAM S.)
4. University of Dayton Research Institute (UDRI)
5. McDonnell Douglas Helicopter Co. (MDHC)
6. BASF, Charlotte, NC
7. Lockheed Aeronautical Systems (LOCK.)
8. Industrial Products Research Institute (IPRI) Japan
9. Israel Aircraft Industries, Ltd. (IAI)
10. Air Force Wright Aeronautical Laboratories (AFWAL)
11. Ciba Geigy Corp. (CIBA G.), Anaheim, CA
12. Nippon Steel Co. (NIP.S.) Japan
13. 3M Corporation
14. Sikorsky Aircraft Co. (SIKOR.)
15. University of Tokyo, Japan
16. Nippon Oil Co. (NIP.OIL) Japan

EGF

1. Imperial College, England
2. University of Portugal
3. FFA, Sweden
4. The Welding Institute, England
5. Ecole Polytechnic Federale de Lausanne (EPFL), Switzerland
6. Imperial Chemicals Industries (ICI), England
7. University of Cranfield, England
8. IFREMER, France
9. University of Tokyo, Japan

Round 4

1. NASA Langley Research Center
2. Bell Helicopter Co.
3. Integrated Technologies (INTEC)
4. Royal Aerospace Establishment (RAE), England
5. The Welding Institute (TWI), England
6. Wichita State University (WSU)
7. Ecole Polytechnic Federale de Lausanne (EPFL), Switzerland
8. Industrial Products Research Institute (IPRI), Japan
9. University of Missouri (U.Mo.)
10. Israel Aircraft Industries, Ltd. (IAI)

Round 5

1. NASA Langley Research Center
2. Bell Helicopter Co.
3. Rohr Industries, Inc.
4. McDonnell Douglas Helicopter Co. (MDHC)
5. BASF, Charlotte, NC
6. Air Force Wright Aeronautical Laboratories (AFWAL)
7. Imperial College (U.London), England

References

[*1*] O'Brien, T. K. and Martin, R. H., "Results of ASTM Round Robin Testing for Mode I Interlaminar Fracture Toughness of Composite Materials," *NASA TM 104222*, August 1992.

[2] Wilkins, D. J., Eisenmann, J. R., Camin, R. A., et al., "Characterizing Delamination Growth in Graphite Epoxy," *Damage in Composite Materials: Basic Mechanisms, Accumulation, Tolerance, and Characterization, ASTM STP 775*, K. L. Reifsnider, Ed., American Society for Testing and Materials, Philadelphia, 1982, pp. 168–183.

[*3*] Bascom, W. D., Bitner, R. J., Moulton, R. J., and Siebert, A. R., "The Interlaminar Fracture of Organic-Matrix Woven Reinforced Composites," *Composites*, Vol. 11, No. 9, January 1980, pp. 9–18.

[*4*] de Charentenay, F. X., Harry, J. M., Prel, Y. J., and Benzeggagh, M. L., "Characterizing the Effect of Delamination Defect by Mode I Delamination Test," *The Effect of Defects in Composite Materials, ASTM STP 836*, D. J. Wilkins, Ed., American Society for Testing and Materials, Philadelphia, 1984, pp. 84–103.

[*5*] Russell, A. J., "Factors Affecting the Opening Mode Delamination of Graphite Epoxy Laminates," *DREP Materials Report 82-Q*, Defense Research Establishment Pacific, Victoria, B. C., Canada, 1982.

[*6*] Johnson, W. S. and Mangalgari, P. D., "Investigation of Fiber Bridging in Double Cantilever Beam Specimens," *Journal of Composites Technology and Research*, Vol. 9, Spring 1987, pp. 10–13.

[*7*] Martin, R. H., "Effect of Initial Delamination on G_{Ic} and G_{Ith} Values from Glass Epoxy Double Cantilever Beam Tests," *Proceedings, 3rd American Society for Composites (ASC) Conference*, Seattle, WA, Technomic Publishers, September 1988, pp. 688–700.

[*8*] Berry, J. P., "Determination of Fracture Energies by the Cleavage Technique," *Journal of Applied Physics*, Vol. 34, No. 1, January 1963, pp. 62–68.

[*9*] Hashemi, S., Kinloch, A. J., and Williams, J. G., "The Analysis of Interlaminar Fracture in Uniaxial Fibre-polymer Composites," *Proceedings of the Royal Society of London*, Vol. A427, No. 1827, 1990, pp. 173–199.

[*10*] Kageyama, K. and Hojo, M., "Proposed Methods for Interlaminar Fracture Toughness Tests of Composite Laminates," *Proceedings of the 5th U.S./Japan Conference on Composite Materials*, Tokyo, June 1990, pp. 227–234.

[*11*] de Kalbermatten, T., Jaggi, R., and Flueler, P., "Interlaminares Bruchverhalten von CFK-Epoxy/PEEK-Laminaten in Mode I an DCB-Proben (Interlaminar Fracture of CFK-Epoxy/PEEK Laminates in Mode I and DCB-Test)," *EMPA Report No. 116'598/ZEP*, Swiss Federal Laboratories for Materials Testing and Research, Dubendorf, Switzerland, September 1990.

[*12*] Brunner, A. J., "Real Time X-Ray Radiography and Simultaneous Acoustic Emission Monitoring During Mode I Crack Propagation in Double Cantilever Beam (DCB) Specimens from Carbon-Fiber Reinforced PEEK, *EMPA Report No. 116'598/9 ZfP*, Swiss Federal Laboratories for Material Testing and Research, Dubendorf, Switzerland, December 1992.

[*13*] Flueler, P., "Crack Propagation in Fiber Reinforced Composite Materials Analysed with Insitu Microfocal X-ray Radiography and Simultaneous Acoustic Emission Monitoring," *Proceedings of the ECCM Conference on Composites Testing and Standardization*, Amsterdam, 1992.

[*14*] de Kalbermatten, T., Jaggi, R., Flueler, P., and Kausch, H. H., *Journal of Materials Science Letters*, Vol. 11, 1992, pp. 543–546.