IN-24
130520
p-122

NASA TECHNICAL MEMORANDUM 104222
AVSCOM TECHNICAL REPORT 92-B-013

RESULTS OF ASTM ROUND ROBIN TESTING FOR MODE I INTERLAMINAR FRACTURE TOUGHNESS OF COMPOSITE MATERIALS

T. K. O'Brien and R. H. Martin

NOVEMBER 1992

National Aeronautics and Space Administration
Langley Research Center
Hampton, Virginia 23665-5225

US ARMY
AVIATION
SYSTEMS COMMAND
AVIATION R&T ACTIVITY

RESULTS OF ASTM ROUND ROBIN TESTING FOR MODE I INTERLAMINAR FRACTURE TOUGHNESS OF COMPOSITE MATERIALS

T. Kevin O'Brien
U.S. Army Aerostructures Directorate
NASA Langley Research Center
Hampton, Virginia

Roderick H. Martin
Analytical Services & Materials, Inc.
Hampton, Virginia

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 D30 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.

KEY WORDS

Composite Materials, Double Cantilever Beam, Interlaminar Fracture Toughness, Delamination, Standard Test Method.

INTRODUCTION

The data contained herein were generated by voluntary participants using the Double Cantilever Beam (DCB) tests (fig.1). The DCB test consists of a unidirectional fiber reinforced laminate, manufactured with a thin insert implanted at the midplane 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 marterial suppliers. A list of participants is included in Appendix 1. A chronology of the activity is documented in Appendix 2 in the form of excerpts from ASTM meeting minutes (less figures) from 1986 to the present.

Early discussions (prior to 1985) resulted in limiting the DCB test to zero degree unidirectional laminates to prevent the initial delamination from branching to interfaces away from the midplane [1]. The width-tapered DCB configuration [2] was abandoned because of the added complexity of machining this configuration and the tendency for zero degree unidirectional width tapered laminates to split at the juncture between the narrow and tapered regions. Furthermore, the phenomenon of fiber bridging between the two zero degree plies on either side of the delamination [3-5] 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 obtain 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 thin 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. 1a) or loading blocks (fig. 1b). A variation on the loading block configuration designated "T-tabs" was also used (fig. 1c). 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. 1a) and end loading blocks (fig.1b) were used in rounds 3-5.

Specimens in the first two rounds were 25mm (1 inch) wide. In the third round, however, 20 mm wide specimens were tested, but a limited number of 0.5, 1.0, and 1.5 inch wide specimens were also tested. Because no significant width effect was discovered (see appendix 2.6), 20-25 mm wide specimens were tested in the 4th and 5th rounds.

In the first and second rounds, specimens consisted of 24 ply laminates for the AS4/3501-6 and AS4/BP907 tests, and of 36 ply laminates for the AS4/PEEK tests, with a nominal thickness of 0.28 mm (0.005 inches). 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 3-5.

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 appendix 2.5-2.9). In the first round, 1.0 mil 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 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 1.5 mil thick folded aluminum foil inserts yielding a total insert thickness of 3.0 mil. These inserts proved to be too thick to measure a useful initiation value, and hence, the first round AS4/PEEK data was 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) 0.5 mil single layers sprayed with a mold release agent, (2) 0.5 mil layers folded in two to achieve a 1.0 mil thickness or 1.0 mil single layers sprayed with a mold release agent, or (3) 1.0 mil layers folded in two to achieve a 2.0 mil thickness. Data from this round indicated that the 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 [6] .

In the third round, both 7 micron (0.25 mil) and 13 micron (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, that 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 appendix 2.6-2.8). 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 micron (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 was unsuccessful for the same reasons noted previously.

In the fifth round, tests were conducted on AS4/PEEK specimens with both 7.5 micron (0.25 mil) and 13 micron (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" using the Kaleidagraph software package for the Macintosh computer. 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 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 both graphically in the form of bar charts and in Tables.

RESULTS FROM ROUND I

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 single draft test procedure was sent to each lab. The data were reduced using a compliance calibration technique commonly known as Berry's method [7].

Figures 2 and 3 show the visually observed initiation G_{Ic} values, measured from a 1.0 mil Kapton insert, for the AS4/BP907 DCB specimens. There was significant variability in the results reported from the various labs. Figures 4 and 5 show that the compliance calibration exponent, n, was very consistent for both configurations, indicating that variation in G_{Ic} values resulted primarily from variations in measured delamination onset loads and delamination lengths. Figure 6 shows a comparison of mean G_{Ic} values for the six labs that performed tests with both configurations. Similar mean G_{Ic} values were obtained for both configurations by 5 of the 6 labs. Figure 7 shows the standard deviation in the data obtained for these six labs. Four of the six labs had significantly higher standard deviations for the T-tab configurations than for the piano hinge configuration. Figures 8 and 9 show the variability in mean G_{Ic} values for all the labs that tested the piano hinge and T-tab configurations, respectively. Also shown in these figures are the 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 standard E691. 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) corresponding to the repeatability and reproducibility.

RESULTS FROM ROUND II

Three labs each received three specimens to test where the load was introduced using piano hinges. Figure 10 shows the G_{Ic} values measured by visually observing delamination onset at the edge of DCB specimens of AS4/3501-6 with 0.5 mil sprayed, 0.5 mil folded, 1.0 mil sprayed, and 1.0 mil folded Kapton inserts. The results indicate that the 0.5 mil sprayed inserts yield the lowest mean values, the 0.5 mil folded, 1.0 mil sprayed inserts, both of which result in a 1.0 mil insert thickness, yield higher mean values, and the 1.0 mil folded inserts, which result in a 2.0 mil insert thickness, yield the highest mean values and have the greatest scatter. Figures 11 and 12 show the mean G_{Ic} values and standard deviation, respectively, for each of the three labs that performed the tests. Figure 13 compares the G_{Ic} values measured from the 0.5 mil sprayed insert for the 3 labs. Figure 14 compares the mean G_{Ic} values measured from the 13 micron (0.5 mil) sprayed insert for the 3 labs, and the statistical measures of repeatability within a given laboratory, S_r, and the reproducibility between laboratories, S_R. These data are summarized in table 1 along with the coefficients of variation corresponding to repeatability within a given laboratory, $(CV)_r$, and the reproducibility between laboratories, $(CV)_R$.

Figure 15 shows the G_{Ic} values measured by visually observing delamination onset at the edge of DCB specimens of AS4/PEEK with 0.5 mil sprayed, 0.5 mil folded, 1.0 mil sprayed, and 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 figure 15. The results indicate that the 0.5 mil sprayed inserts yield the lowest mean values, the 0.5 mil folded, 1.0 mil sprayed inserts, both of which result in a 1.0 mil insert thickness, yield slightly higher mean values, and the 1.0 mil folded inserts, which result in a 2.0 mil insert thickness, yield the highest mean values.

Figure 16 shows the mean G_{Ic} values, for the four insert configurations, for the two labs that performed the tests. Figure 17 compares the mean G_{Ic} values measured from the 0.5 mil (13 micron) sprayed insert for the two labs, and the standard deviations for repeatability within a given laboratory and reproducibility between laboratories. These data are summarized in table 1 along with the coefficients of variation corresponding to repeatability within a given laboratory, $(CV)_r$, and the reproducibility between laboratories, $(CV)_R$. 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.18). 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 III. As a prelude to the third round, these initiation measurements and data reduction methods were used to plot the data generated on 0.5 mil sprayed Kapton insert tests from additional tests conducted by three labs, each testing four specimens, during round II.

Figure 19 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%). These data were reduced using the compliance calibration technique, known as Berry's method, that had been used previously in round I. These same values were plotted in fig.20, where the data were reduced using a modified beam theory (MBT) technique [8]. The MBT technique yielded slightly lower mean G_{Ic} values than Berry's method for the same test data. Therefore, all subsequent figures were plotted using the MBT method. Also shown in figures 19-20 are the plateau values of G_{Ic} (PLAT) corresponding to stabilized delamination growth in the presence of fiber bridging. Although the 5% offset and plateau values have less scatter than the NL and VIS values, they are significantly higher, and may correspond to delamination growth in the presence of fiber bridging as opposed to delamination onset. Figure 21 shows the mean G_{Ic} values for the 3 labs that tested the 13 micron (0.5 mil) sprayed Kapton insert specimens. Figure 22 compares the mean G_{Ic} values, and standard deviations associated with repeatability within a given lab, S_r, and reproducibility between labs S_R, measured from the three labs for the 13 micron (0.5 mil) sprayed insert specimens. These data are summarized in table 2 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories. However, a larger data set was needed to obtain an accurate estimate of the repeatability and reproducibility between laboratories.

RESULTS FROM ROUND III

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 micron (0.5 mil) insert specimens were tested by 16 labs, whereas the 7 micron (0.25 mil) specimens were tested by 5 labs. Each lab received 4 specimens to test per insert thickness.

Figures 23 and 24 show the mean NL and VIS G_{Ic} values, respectively, for the 16 labs that tested the 13 micron aluminum insert specimens. The data was reduced using three data reduction methods: (1) the Modified Beam Theory (MBT), (2) Berry's method (BRY), and (3) a Modified Compliance Calibration (MCC) method [9]. Figure 25 shows the mean NL G_{Ic} values for the 5 labs that tested the 7 micron aluminum insert specimens. For both insert thicknesses, the variation between the three data reduction methods for any single lab was no greater than 3.1%. However, because the MBT method yielded lower G_{Ic} values than the two compliance calibration methods for 80% of the tests that were conducted, the remaining data in this report is summarized using the MBT method only.

One additional feature of the MBT data reduction technique is the ability to measure the flexural modulus, E_f, for any delamination length. Ideally, E_f should not vary with delamination length. However, figures 26 and 27 show the variation that was observed for the 13 micron (0.5 mil) aluminum insert and 7 micron (0.25 mil) aluminum insert specimens. The average variation was 10.7% and 8.0%, respectively. The initial modulus measured before delamination onset was the maximum flexural modulus recorded for 50% of the 13 micron insert tests and 60% of the 7 micron insert tests.

13 micron aluminum insert results

Figure 28 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 ran tests on 13 micron (0.5 mil) aluminum insert specimens. Each lab received 4 specimens to test. Figure 29 shows the mean G_{Ic} values for the 16 labs that tested the 13 micron aluminum insert specimens. Figure 30 shows the standard deviation in the data reported by each of these 16 labs.

Figure 31 shows the mean NL G_{Ic} values measured from the 13 micron (0.5 mil) aluminum insert for the 16 labs, and the standard deviations for repeatability within a given laboratory and reproducibility between laboratories. Figure 32 shows the mean VIS G_{Ic} values measured from the 13 micron (0.5 mil) aluminum insert for the 16 labs, and the standard deviations for repeatability and reproducibility. Figure 33 shows the mean 5% offset G_{Ic} values measured from the 13 micron (0.5 mil) aluminum insert for the 16 labs, and the repeatability and reproducibility. The data shown in these three figures are summarized in table 2 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories.

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% values. Figures 34 and 35 show the percentage difference in the NL and VIS G_{Ic} values, and between the NL and 5% G_{Ic} values, respectively. The average difference in G_{Ic} was 16.4% and 20.2%, respectively, indicating that significant nonlinearity occurred before delamination onset was observed at the edge.

In 73% of the tests with the 13 micron (0.5 mil) aluminum inserts, propagation values of G_{Ic}, corresponding to delamination growth in the presence of fiber bridging, were lower than NL and/or VIS onset values. The visual observation usually preceded the 5%

offset estimates. In 70% of the individual tests, the VIS G_{Ic} values were lower than the 5% offset values.

7 micron aluminum insert results

Figure 36 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 5 labs that conducted tests on specimens with the 7 micron (0.25 mil) aluminum inserts. Each lab tested 4 specimens. Figure 37 shows the mean G_{Ic} values for the 5 labs. Figure 38 shows the standard deviation in the data reported by each of these 5 labs.

Figure 39 shows the mean NL G_{Ic} values measured from the 7 micron (0.25 mil) aluminum insert for the 5 labs, and the standard deviation corresponding to the repeatability and reproducibility between laboratories. Figure 40 compares the mean VIS G_{Ic} values measured from the 7 micron (0.25 mil) aluminum insert for the 5 labs, and the repeatability and reproducibility between laboratories. Figure 41 compares the mean 5% offset G_{Ic} values measured from the 7 micron (0.25 mil) aluminum insert for the 5 labs, and the repeatability and reproducibility between laboratories. The data shown in these three figures are summarized in table 2 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories.

The reproducibility and repeatability between laboratories was similar for all three onset measurements. However, the NL G_{Ic} values were significantly lower than the VIS and 5% values. Figures 42 and 43 show the percentage difference in the NL and VIS G_{Ic} values, and between the NL and 5% G_{Ic} values, respectively. The average difference in G_{Ic} was 10.2% and 15.9%, respectively, indicating that significant nonlinearity occurred before delamination onset was observed at the edge.

In 75% of the tests with the 7 micron (0.25 mil) aluminum inserts, propagation values of G_{Ic}, corresponding to delamination growth in the presence of fiber bridging, were lower than NL and/or VIS 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 and/or VIS 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 and/or NL G_{Ic} values

(Fig. 44). In contrast, R-curves for specimens with Kapton inserts always achieved PLAT G_{Ic} values above the NL and VIS G_{Ic} values (Fig. 45). Microscopy studies performed at ICI on untested specimens indicated that localized yielding (crimping) may have occurred during cutting of the aluminum foil inserts (see appendix 2.8). These crimps, which were not evident in the original panel radiographs, were responsible 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 micron foils, which yield higher apparent G_{Ic} values than the 13 micron foils (Table 2).

International Round Robin (EGF)

Tests performed by EGF participants using specimens that were not X-rayed to isolate specimens with tears or folds in the aluminum inserts are summarized next. The 13 micron (0.5 mil) insert specimens were tested by 6 labs, whereas the 7 micron insert specimens were tested by 4 labs. Each lab received 4 specimens to test. The data was reduced using the Modified Beam Theory (MBT) method.

13 micron aluminum insert results

Figure 46 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 6 labs that ran tests on 13 micron (0.5 mil) aluminum insert specimens. Figure 47 shows the mean G_{Ic} values for the 6 labs that tested the 13 micron aluminum insert specimens. One lab (ICI) reported only VIS G_{Ic} values, and another lab (U. of Portugal) did not report 5% offset G_{Ic} values. Figure 48 shows the standard deviation in the data reported by each of these 6 labs.

Figure 49 shows the mean NL G_{Ic} values measured from the 13 micron (0.5 mil) aluminum insert for 5 of the 6 labs that reported these values, and the standard deviation corresponding to the repeatability and reproducibility between laboratories. Figure 50 shows the mean VIS G_{Ic} values measured from the 13 micron (0.5 mil) aluminum insert for the 6 labs, and the repeatability and reproducibility between laboratories. Figure 51 shows the mean 5% offset G_{Ic} values measured from the 13 micron (0.5 mil) aluminum insert for 4 of the 6 labs that reported these values, and the repeatability and reproducibility between laboratories. The data shown in these three figures are summarized in Table 3 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories.

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.

7 micron aluminum insert results

Figure 52 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 4 labs that conducted tests on specimens with the 7 micron (0.25 mil) aluminum inserts. Figure 53 shows the mean G_{Ic} values for the 4 labs that tested the 7 micron insert specimens. Three labs did not report 5% offset G_{Ic} values. One lab (ICI) reported VIS G_{Ic} values for only one specimen. Figure 54 shows the standard deviation in the data reported by each of the 3 labs that tested more than one specimen.

Figure 55 shows the mean NL G_{Ic} values measured from the 7 micron (0.25 mil) aluminum insert for 3 of the 4 labs that reported these values, and the standard deviation corresponding to the repeatability and reproducibility between laboratories. Figure 56 compares the mean VIS G_{Ic} values measured from the 7 micron (0.25 mil) aluminum insert for the 4 labs, and the repeatability and reproducibility between laboratories. Because only one of the 4 labs reported 5% offset G_{Ic} values measured from the 7 micron (0.25 mil) aluminum insert, no mean values and repeatability and reproducibility were reported. The data shown in these two figures are summarized in Table 3 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories.

The reproducibility and repeatability between laboratories was 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 IV

For this round, 13 micron 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 10 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. 45).

Figure 57 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 10 labs that each conducted tests on 3 specimens with the 13 micron (0.5 mil) Kapton inserts as part of the DCB fatigue round robin. Figure 58 shows the mean G_{Ic} values for the 10 labs. Figure 59 shows the standard deviation in the data reported by each of the 10 labs.

Figure 60 shows the mean NL G_{Ic} values measured from the 13 micron (0.5 mil) Kapton insert for the 10 labs, and the standard deviations for repeatability within a given laboratory and reproducibility between laboratories. Figure 61 shows the mean VIS G_{Ic} values measured from the 13 micron (0.5 mil) Kapton insert for the 10 labs, and the repeatability and reproducibility between laboratories. Figure 62 shows the mean 5% offset G_{Ic} values measured from the 13 micron (0.5 mil) Kapton insert for the 16 labs, and the repeatability and reproducibility. The data shown in these three figures are summarized in table 3 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories.

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 was similar to earlier rounds conducted with 13 micron 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 data base to justify the precision statement for an ASTM standard (see ASTM standard E691). The required data base includes a minimum of 5 tests conducted by at least 6 different laboratories. In order to generate the required data base, and to quantify the sensitivity of G_{Ic} to insert thickness, a second international round robin was conducted.

RESULTS FROM ROUND V

For this round robin, both 7.5 and 13 micron 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 9 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. 45).

Figures 63 and 64 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 9 labs that each conducted tests on 5 specimens with the 13 micron (0.5 mil) and 7.5 micron (0.25 mil) Upilex inserts. Figures 65 and 66 show the mean G_{Ic} values for the 9 labs. Figures 67 and 68 show the standard deviation in the data reported by each of the 9 labs.

Figures 69 and 70 show the mean NL G_{Ic} values measured from the 13 micron (0.5 mil) and 7.5 micron (0.25 mil) Upilex inserts for the 9 labs, and the standard deviations for repeatability within a given laboratory and the reproducibility between laboratories. Figures 71 and 72 show the mean VIS G_{Ic} values measured from the 13 micron (0.5 mil) and 7.5 micron (0.25 mil) Upilex inserts for the 9 labs, and the repeatability and reproducibility between laboratories. Figures 73 and 74 show the mean 5% offset G_{Ic} values measured from the 13 micron (0.5 mil) and 7.5 micron (0.25 mil) Upilex inserts for the 9 labs, and the repeatability and reproducibility. The data shown in these six figures are summarized in Table 4 along with the coefficients of variation for repeatability within a given laboratory and reproducibility between laboratories.

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 round robins. Mean NL G_{Ic} values for this round 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 micron Upilex insert specimens were 6.3% lower than the average NL G_{Ic} values for the 13 micron Upilex insert specimens.

SUMMARY

As a result of round V, the draft DCB standard was updated and submitted for balloting within ASTM committee D30 in June 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 microns yield unrealistically high G_{Ic} values, and because the difference in average NL G_{Ic} values for 7.5 and 13 micron Upilex inserts was relatively small (6.3%), an insert thickness requirement of 13 microns or less was adopted for the ASTM DCB standard. The 7.0-7.5 micron 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 micron 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.

Third, the NL G_{Ic} value was recommended as the preferred measure of mode I interlaminar fracture toughness. This recommendation is based on physical observations, made using video based in-situ dye penetrant enhanced X-radiography, that the delaminations initiate at the end of the insert, in the interior of the specimen width, when the load deflection curve becomes nonlinear [10-13]. 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 75, mean VIS and mean 5% offset G_{Ic} values were typically 18-22% higher than mean NL G_{Ic} values even though VIS and 5% offset measurements were more repeatable (Fig.76). Hence, The NL G_{Ic} values are conservative values corresponding to the first onset of delamination.

REFERENCES

1. Wilkins, D.J., Eisenmann, J.R., Camin, R.A., Margolis, W.S., and Benson, R.A., "Characterizing Delamination Growth in Graphite Epoxy," Damage in Composite Materials, ASTM STP 775, 1982, pp. 168-183.

2. 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, Jan., 1980, pp. 9-18.

3. 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.

4. Russell, A.J., "Factors Affecting the Opening Mode Delamination of Graphite Epoxy Laminates," DREP Materials Report 82-Q, Defense Research Establishment Pacific, Victoria, BC, Canada, 1982.

5. Johnson, W.S. and Mangalgari, P.D., "Investigation of Fiber Bridging in Double Cantilever Beam Specimens," Journal of Composite Technology and Research, Vol.9, Spring 1987, pp. 10-13.

6. Martin, R.H., "Effect of Initial Delamination on G_{Ic} and G_{Ith} Values from Glass Epoxy Double Cantilever Beam Tests," Proc. 3rd American Society for Composites (ASC) Conference, Seattle, WA., Sept.,1988, Technomic Publishers, pp. 688-700.

7. Berry, J.P., "Determination of Fracture Energies by the Cleavage Technique," Journal of Applied Physics, Vol.34, No.1, January 1963, pp.62-68.

8. 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.

9. 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.

10. de Kalbermatten, T., Jaggi, R., and Flueler, P., "Interlaminares Bruchverhalten von CFK-Epoxy/PEEK-Laminaten in Mode I an DCB-Proben," EMPA Report No. 116'598/ZEP, Swiss Federal Laboratories for Materials Testing and Research, Dubendorf, Switzerland, Sept. 1990.

11. A.J. Brunner, et.al., "Rissausbreitung in DCB-Proben aus kohlenstoff-faserverstarkten PEEK-Laminaten unter Belastungsart I (Mode I) untersucht mit Echtzeit-Radiographie und simultanen Schallemissions-Messungen," EMPA Report No. 116'598/5 ZfP, Swiss Federal Laboratories for Materials Testing and Research, Dubendorf, Switzerland, July, 1991.

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

13. de Kalbermatten, T., Jaggi, R., Flueler, P., and Kausch, H.H., J. Materials Science Letters, Vol.11, 1992, pp. 543-546

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	$(CV)_R$ %
I	AS4/BP907	HINGES	9	3	25 μm Kapton	0.400*	0.028	7.0	0.077	19.3
I	AS4/BP907	T-TABS	7	3	25 μm Kapton	0.410*	0.041	10.0	0.052	12.7
II	AS4/3501-6	HINGES	3	3	13 μm Kapton	0.085*	0.015	17.6	0.014	16.5
II	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)

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	$(CV)_R$ %
II	ASTM	3	4	13 µm Kapton	NL	0.983	0.132	13.4	0.178	18.1
III	ASTM/JIS	16	4	13 µm Al. Foil	NL	1.439	0.187	13.4	0.261	18.1
III	ASTM/JIS	16	4	13 µm Al. Foil	VIS	1.724	0.232	13.5	0.194	11.3
III	ASTM/JIS	16	4	13 µm Al. Foil	5%	1.799	0.213	11.8	0.146	8.1
III	ASTM/JIS	5	4	7 µm Al. Foil	NL	1.727	0.226	13.0	0.140	8.1
III	ASTM/JIS	5	4	7 µm Al. Foil	VIS	1.929	0.257	13.3	0.201	10.4
III	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

TABLE 3 - 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	$(CV)_R$ %
III	EGF	6	4	13 μm Al. Foil	NL	1.574	0.230	14.6	0.283	18.0
III	EGF	6	4	13 μm Al. Foil	VIS	1.953	0.209	10.7	0.286	14.6
III	EGF	6	4	13 μm Al. Foil	5%	1.938	0.202	10.4	0.220	11.4
III	EGF	4	4	7 μm Al. Foil	NL	1.575	0.160	10.2	0.121	7.7
III	EGF	4	4	7 μm Al. Foil	VIS	1.883	0.150	8.0	0.154	8.2
IV	ASTM	10	3	13 μm Kapton	NL	1.303	0.180	13.8	0.207	15.9
IV	ASTM	10	3	13 μm Kapton	VIS	1.549	0.198	12.8	0.151	9.7
IV	ASTM	10	3	13 μm Kapton	5%	1.713	0.171	10.0	0.139	8.1

* ALL G_{IC} VALUES DETERMINED USING MODIFIED BEAM THEORY

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	$(CV)_R$ %
V	ASTM/JIS/ EGF	9	5	13 µm Upilex	NL	1.262	0.132	10.5	0.110	8.7
V	ASTM/JIS/ EGF	9	5	13 µm Upilex	VIS	1.532	0.167	10.9	0.075	4.9
V	ASTM/JIS/ EGF	9	5	13 µm Upilex	5%	1.549	0.144	9.3	0.080	5.1
V	ASTM/JIS/ EGF	9	5	7.5 µm Upilex	NL	1.182	0.126	10.7	0.111	9.4
V	ASTM/JIS/ EGF	9	5	7.5 µm Upilex	VIS	1.447	0.126	8.7	0.075	5.2
V	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 MODIFIED BEAM THEORY

APPENDIX 1 - List of Round Robin Participants

ROUND I

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 II

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 III (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, N.C.
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

ROUND III (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

ROUND IV

1. NASA Langley Research Center
2. Bell Helicopter Co.
3. (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 V

1. NASA Langley Research Center
2. Bell Helicopter Co.
3. Rohr Industries, Inc.
4. McDonnell Douglas Helicopter Co. (MDHC)
5. BASF, Charlotte, N.C.
6. Air Force Wright Aeronautical Laboratories (AFWAL)
7. Imperial College (U.London) England
8. IFREMER, France
9. University of Tokyo, Japan

APPENDIX 2

MINUTES

ASTM D30.02.02 Task group on Interlaminar Fracture

1986-1990

ASTM D30.06 on Interlaminar Properties

1991-1992

APPENDIX NO.	MEETING DATE	LOCATION
2.1	April, 1986	Charleston, SC
2.2	April, 1987	Cincinnati, OH
2.3	October, 1987	Bal Harbour, FL
2.4	April, 1988	Reno, NV
2.5	November, 1989	Orlando, FL
2.6	April, 1990	San Francisco, CA
2.7	November, 1990	San Antonio, TX
2.8	May, 1991	Indianapolis, IN
2.9	October, 1991	San Diego, CA
2.10	May, 1992	Pittsburgh, PA

ASTM MEETING MINUTES
D30.02.02 TASK GROUP ON INTERLAMINAR
FRACTURE TOUGHNESS

APRIL 30,1986
SHERATON HOTEL, CHARLESTON, S.C.

The meeting was called to order at 5:15 p.m. by chairperson T.K.O'Brien.

The chairman reviewed the status of the round robin. The final list of participants is included as enclosure #1. Participants are receiving the first test specimens of AS4/BP907. The AS4/BP907 panels were manufactured at NASA Langley from prepreg supplied by Cyanamid. The majority of the specimens were cut and distributed before the meeting. The remaining specimens are being cut from the panels and will be distributed by the end of June. Richard Hall of Hercules reported that the AS4/3501-6 panels are currently being manufactured, and that he plans to distribute specimens by the end of May. Christopher Price of ICI reported that the AS4/PEEK panels should be manufactured by the end of June, at which time they will be sent to NASA Langley to be cut into specimens, to check the crystallinity percentage, and to be distributed.

Kevin O'Brien noted that Ran Kim of the University of Dayton had completed testing of the ENF and EDT specimens that were sent to AFWAL. Jim Whitney of AFWAL mentioned that although they could detect the onset of edge delamination in the $(35/-35/0/90)_s$ EDT specimens without inserts, they had difficulty detecting the onset of delamination in the specimens with the mid-plane inserts. Kevin O'Brien will forward some EDT specimens with mid-plane inserts to Ron Zabora at Boeing to see if his through-thickness displacement gage can more accurately detect delamination onset in these laminates.

The manufacturing procedures for each test specify that a specific specimen from each panel shoud be digested to determine volume fraction according to ASTM standard D3171. Because some of these specimens had already been distributed for testing, Kevin O'Brien suggested that the volume fraction measurment be performed on the specimens after testing. Walter Bradley suggested that the portion of the specimen to be digested should be cut from the tested specimens before they are split into two pieces for fractographic examination. The membership agreed to these changes, and modifications to the manufacturing and test procedures shall be forwarded to the participants. In addition, an alternate procedure for determining volume fraction ,used by Norm Johnston at NASA Langley, was suggested by Kevin O'Brien and is shown in enclosure #2. Richard Hall from Hercules noted that there are several techniques for determining volume fraction that are superior to digestion, and the proposed technique was one of the best. Ian Kowalski from Union Carbide noted that a ball-tipped micrometer should be used to get an accurate thickness measurement independent of surface texture. The proposed technique will be used by Hercules, ICI, and NASA Langley to measure volume fraction and compare to the values determined by digestion.

Walter Bradley suggested that selected specimens should be examined for local volume fraction variations in the vicinity of the delamination tip. He agreed to come up with a random sampling plan to investigate this potential variation.

Steve Johnson's presentation on "Investigation of Fiber Bridging in Double Cantilever Beam Specimens" originally scheduled for the task group meeting was presented during the conference on "Composite Materials: Testing and Design" to fill in for a canceled paper. Steve found that G_{Ic} measured at the end of the insert in the DCB test represented a characteristic in situ toughness of the matrix material in the composite. Fiber bridging artificially raised the G_{Ic} values measured further down the beam. In addition, he found that a thin adhesive bondline of matrix material between metal adherends in a DCB configuration yielded toughness values equal to the composite DCB values without fiber bridging. This work is published in NASA TM 87716, and can be obtained from Steve at MS 188E, NASA Langley Research Center, Hampton, Virginia, 23665.

ASTM MEETING MINUTES
D30.02.02 TASK GROUP ON INTERLAMINAR
FRACTURE TOUGHNESS

APRIL 27, 1987
OMNI NETHERLANDS HOTEL
CINCINNATI OHIO

The meeting was called to order at 5:15 p.m. by chairperson T.K.O'Brien.

The chairman reviewed the status of the round robin. Participants have received all of the test specimens of AS4/BP907 and AS4/PEEK. The AS4/3501-6 panels will either be manufactured by Hercules this summer, or will be manufactured at NASA Langley and cut into specimens this fall. The chairman noted that participants may not have these specimens in hand until the end of 1987, and he requested that they should proceed with testing of the other specimens immediately. The chairman also reported the results of the lamina property measurements that were conducted by three laboratories. These data are included as enclosure one. Each data point recorded is the mean value of five tests that were conducted by each laboratory. The average of these mean values should be used for data reduction for the various interlaminar fracture tests.

The chairman reminded participants of several points that were agreed upon at last year's meeting in Charleston, South Carolina. Walter Bradley of Texas A&M requested that each participant send him a 1/2" by 1" wide piece of each test specimen (carefully labeled to indicate material, panel and specimen number, and fracture toughness measurement) cut from the specimen just ahead of where the crack stopped in the test. This piece will be examined to detemine localized variations in fiber volume fraction. The rest of the tested specimens should be sent to John Masters, chairman of the task group on fractography. In addition, all of the load-displacement records for the various tests should be submitted, along with the tabulated data, to the task group chairman and the working group chairman for that particular test. This will insure that future data reduction techniques may be applied to the raw data generated during the round robin. Furthermore, the chairman requested that the participants contact the working group chairman concerning any questions or difficulties they encounter when conducting the various tests. This type of feedback is critical to our efforts to update the test procedures as we learn from the round robin testing.

ASTM MEETING MINUTES
D30.02.02 TASK GROUP ON INTERLAMINAR
FRACTURE TOUGHNESS

OCTOBER 19, 1987
SHERATON HOTEL
BAL HARBOUR, FLORIDA

The meeting was called to order at 5:00 pm by chairperson T.K. O'Brien.

The chairman reviewed the status of the round robin. The testing of AS4/BP907 and AS4/PEEK laminates is approximately one third completed (see enclosure #1). The chairman reported that Hercules plans to fabricate and distribute the AS4/3501 laminates to participants by the end of November. The chairman emphasized that it was important that all participants complete their tests before the next meeting in April. This meeting will preceed the conference on Composite Materials: Testing and Design. This will be the last meeting where the conference theme is related to interlaminar fracture of continuous reinforced polymer matrix composites until the fall 1989 meeting, 1&1/2 years later. If enough data is in from the round robin before the meeting in Reno, we will be able to start the process of drafting ASTM standards for interlaminar fracture toughness.

ASTM MEETING MINUTES
D30.02.02 TASK GROUP ON
INTERLAMINAR FRACTURE TOUGHNESS

APRIL 26, 1988
NUGGET HOTEL
RENO, NEVADA

The chairman called the meeting to order at 8:00 am.

The chairman reviewed the status of the round robin and the objectives and agenda for the meeting (see enclosure #1). For the AS4/BP907 and AS4/PEEK materials, the ENF and EDT tests are nearly completed, whereas the DCB specimens are just over 1/2 finished and the CLS specimens were only 1/3 completed. Difficulties were observed with the AS4/3501-6 specimens that were fabricated and distributed by Hercules. Initial EDT panels were laid up incorrectly. New panels were made promptly, and were immediately distributed to participants. Unfortunately, these panels, and the panels for the other tests, had inserts that were not fully debonded from the graphite composite. This problem arose because Hercules was not made aware of the need to spray the Kapton insert with a release agent before laying up and curing of the panels. These bonded inserts caused difficulty in precracking the ENF and CLS specimens, and made precracking necessary for the DCB specimens. Furthermore, the bonded laminates yielded identical moduli before and after delamination for the midplane EDT specimens. Anyone who has not run their AS4/3501-6 specimens should contact their working group chairman before doing so (see enclosure #2).

Herzl Chai from the National Bureau of Standards in Gaithersburg, Maryland, reviewed the results to date for the DCB test (see enclosure #3). Participants reported similar results for the T-tab and piano hinge configurations. However, the piano hinge configuration was preferred because it does not induce bending at the point of load application. Good quality piano hinges were easily obtained by all participants. Hence, the piano hinge configuration will be recommended as the standard configuration. The load-deflection plots were linear up to the onset of delamination growth for all materials. Furthermore, the exponent, n, of the power law relationship between specimen compliance and delamination length was around 2.75 for all configurations and materials. The AS4/BP907 tests exhibited significant resistance to delamination growth as characterized by an R-curve when G_{Ic} was plotted against delamination length. This resistance was due to the significant fiber bridging that was observed during the tests. The participants agreed that because of the fiber bridging phenomenon the only meaningful value obtained from the DCB test was the initiation value obtained from the insert. Some fiber bridging was observed in the AS4/PEEK specimens as well, however, plots of G_{Ic} versus delamination length were relatively flat. Unfortunately, the effect of the fiber bridging in the AS4/PEEK specimens was not clear because these specimens had relatively thick (3 mils) folded aluminum inserts. Hence, the initial value of G_{Ic} measured from the insert was artificially high. Rod Martin, NRC - NASA Langley, showed the results of a study he is conducting on the influence of insert thickness on G_{Ic} measured using the DCB test on Glass Epoxy laminates (see enclosure #4). He obtained similar G_{Ic}

values for measurements from inserts of thicknesses ranging from 0.5 mil to 3 mils, but he obtained larger values of G_{Ic} using inserts of 5 mils. Kevin O'Brien will request that ICI make more panels of the AS4/PEEK with a variety of Kapton insert sizes to identify if there is a consistant initiation value of G_{Ic} for this material. Furthermore, Richard Hall of Hercules offered to make more AS4/3501-6 DCB panels using thin Teflon (1 mil) inserts to overcome the bonding problem experienced with the Kapton film. Both the new AS4/PEEK and AS4/3501-6 specimens will be tested by a limited number of participants using only the piano hinge configuration. Ramesh Shah's suggestion that the panels be made with inserts that extend from the edge for easy location will be followed.

ORIGINAL PAGE IS
OF POOR QUALITY

ASTM MEETING MINUTES
D30.02.02 TASK GROUP ON
INTERLAMINAR FRACTURE TOUGHNESS

NOVEMBER 6, 1989
HILTON - WALT DISNEY WORLD
ORLANDO, FLORIDA

Task group chairman, Kevin O'Brien, called the meeting to order at 5:15 p.m.

The chairman reviewed the agenda for the current meeting and the objectives and status of the round robin (enclosure #1). The AS4/BP907 tests were completed and were reported in the minutes of the last meeting in Reno in April of 1988 (see minutes of that meeting). However, as reported in those minutes, fabrication problems with the AS4/3501-6 and AS4/PEEK resulted in no useful data being generated. Since then, new AS4/3501-6 panels, having inserts with different thicknesses that were either folded or sprayed with a mold release agent, have been fabricated at Hercules. These panels were cut into DCB and ENF specimens and were sent to five laboratories for testing. However, only a few labs have completed the testing to date. Also since the last meeting in Reno, new AS4/PEEK panels with Kapton inserts of different thicknesses, that were either folded or sprayed with a mold release agent, were fabricated by ICI. These panels were sent to NASA Langley to be cut into DCB and ENF specimens. Unfortunately, when these panels were cut into specimens several problems were found with the inserts, which resulted in only enough good DCB and ENF specimens, for all the insert types, for two labs. Five labs were sent DCB specimens with 0.5 mil sprayed inserts. As of the current task group meeting date, only two labs had completed testing the DCB specimens, and only one had completed testing the ENF AS4/PEEK specimens. Hence, there was very little new round robin data to report.

Herzl Chai from the National Institute for Standards (NIST) reviewed the results to date for the DCB test. He had difficulty with his piano hinges debonding in his AS4/PEEK tests such that he only obtained two good test results. He used a room temperature cure adhesive to bond his piano hinges to the DCB specimen. Rod Martin of Analytical Services and Materials (AS&M) at NASA Langley also bonded his hinges to the AS4/PEEK DCB specimens with a room temperature cure adhesive. However, Rod found this bond was strengthened by the post cure that occurred when subjecting the DCB specimens to the drying procedure presecibed in the ASTM test procedure (enclosure #2). Hence, Rod was able to obtain good data for all his DCB tests. Rod's results were presented by Herzl, and are shown in enclosure #3. Rod found that only the 0.5 mil (12.5 μm) sprayed kapton insert specimens exhibited stable initiation from the insert for both the AS4/3501-6 and the AS4/PEEK. These specimens also yielded the most conservative results for G_{Ic} measured from the insert. Herzl Chai commented that the few good tests he was able to run yielded similar results. The remaining discussion centered around the significance of the R-curve measured after onset, and how the load at initiation from the insert should be measured. Kevin O'Brien expressed the consensus opinion from the last task group meeting in Reno (see minutes of that meeting) that because of the fiber bridging mechanism, the only meaningful value obtained from the DCB test was the initiation value from the insert. Herzl Chai showed some data from his own work that showed that the shape of the R-curve, and the so-called plateau propagation value, were dependent on the DCB specimen geometry (enclosure #4). Tony Kinloch and Gordon Williams, representing the European Group on Fracture (EGF), expressed their concern about omitting the R-curve altogether. They were especially concerned because the possible methods for measuring the initiation from the insert, one of which is an offset method requiring generation of at least the first part of the R-curve, had not been clearly specified in the ASTM round robin procedure. Rod Martin's data that was presented was generated by visually observing the onset of the delamination from the insert at one edge of the DCB specimen using a 60x magnification microscope. For the brittle AS4/3501-6 composites, the deviation from linearity in the load-displacement curve agreed with the load obtained by visual observation. However, for the more ductile AS4/PEEK composites, significant deviation from linearity was observed in the load-displacement plot before visual observation of delamination onset. Tony Kinloch expressed his concern about the repeatability of such visual measurements, a point which could not be resolved at the task group meeting due to the limited data that was reported. A plan for resolving these issues through an international round robin was achieved at a meeting the following Wednesday morning of the ASTM D30.02.02 task group, the EGF representatives, and representatives of the Japanese Industrial Standards (JIS) group (see enclosed minutes of that meeting). Ron Zabora noted that none of the materials in the DCB round robin had an interleaf, and that any standard that evolved from the round robin should exclude interleaf composites with two-phase matrices.

ASTM MEETING MINUTES
D30.02.02 TASK GROUP ON INTERLAMINAR FRACTURE

APRIL 26, 1990
HYATT REGENCY
SAN FRANCISCO, CALIFORNIA

The meeting was called to order at 8:00 am by chairman T.K. O'Brien

The chairman reviewed the history and current status of the round robin test program (see enclosure #1). AS4/3501-6 and AS4/PEEK data were received from 3 of the 5 labs conducting the DCB and ENF tests in the second round of the round robin. For the DCB, tests were conducted on specimens with three different Kapton insert thicknesses: (1) 0.5 mil - in the form of a single sheet that was sprayed with a mold release agent, (2) 1.0 mil - in two forms, a single 1.0 mil sheet that was sprayed with a mold release agent, and an 0.5 mil sheet that was folded but not sprayed, and (3) 2.0 mil - in the form of a 1.0 mil sheet that was folded but not sprayed. For both materials, the initiation values of G_{Ic} measured from the insert were lowest for the thinnest 0.5 mil insert. Furthermore, unstable jumps occured at the intiation of the delamination from the thicker inserts. For the ENF, tests were conducted on specimens with 0.5 mil and 1.0 mil Kapton inserts that were single sheets sprayed with a mold release agent. Tests were also conducted on specimens that had been precracked in tension or shear. Different behavior was observed for the two materials tested. For the AS4/3501-6, which has a brittle epoxy matrix, G_{IIc} values generated at initiation from the 0.5 mil insert were lower than values measured from the 1.0 mil insert, but were greater than values measured using either a tension or shear precrack. However, for the AS4/PEEK, which has a tough thermoplastic matrix, G_{IIc} values generated at initiation from the 0.5 mil insert were similar to values measured from the 1.0 mil insert, but were less than values measured using a shear precrack. The scatter between laboratories was greatest for the shear precracked specimens. These results were consistant with the preliminary results from the second round that were reported in the minutes from the last meeting in November, 1989, in Orlando, that led to the organization of an international round robin with the European Group on Fracture (EGF) and the Japanese Industrial

Standards (JIS) group. This activity is the third round of testing for the ASTM participants.

The chairman reviewed the status of the international round robin (see enclosure #2). ASTM, EGF, and JIS representatives set up a timetable for conducting a joint round robin on AS4/PEEK in 1990 on the DCB, ENF, and MMB (Mixed Mode Bending) tests (see minutes of November, 1989, Orlando meeting). Participants were solicited and chosen from each organization. There are 17 ASTM, 13 EGF, and 3 JIS participants that plan to conduct the DCB test, and there are 17 ASTM, 15 EGF, and 3 JIS participants that plan to conduct the ENF test. Also, 7 ASTM, 1 JIS, and 3 EGF participants have expressed an interest in conducting the MMB test. The EGF protocols for the DCB and ENF test have been reviewed and modified by the ASTM and JIS representatives. James Reeder, of NASA Langley, is drafting a protocol for the MMB test. AS4/PEEK panels with either 13 micron (0.5 mil) or 7 micron (0.25 mil) aluminum foil inserts were manufactured at ICI in England and were sent to EPFL, Switzerland, for cutting and distribution to the EGF participants, and to NASA Langley for cutting and distribution to the ASTM and JIS participants. There were only half as many 7 micron insert panels as 13 micron insert panels. The panels received at NASA Langley were X-rayed and found to contain several tears in the aluminum inserts and a few folds at the ends of the inserts. The panels were cut into specimens and X-rayed again to isolate specimens with good quality inserts. There was a 50% rejection rate for the 13 micron insert specimens, and a 70% rejection rate for the 7 micron insert specimens. This left only enough specimens with good quality inserts to conduct the DCB round robin. The DCB protocol and test specimens have been sent to the ASTM and JIS participants. ASTM and EGF representatives are currently negotiating with ICI to obtain more panels for the ENF and MMB round robins.

The chairman reviewed some of the AS4/PEEK DCB results from the second round of the round robin using the data reduction techniques that are outlined in the protocol for the third-round international round robin (enclosure #3). In the DCB protocol, three initiation values of G_{Ic} will be recorded using either (1) the load at first deviation from linearity (NL), (2) the load at which the delamination is observed visually on either edge (VIS), and (3) the load corresponding to a 5% offset from the initial linear compliance. In addition, a propagation value (PROP) will be recorded corresponding to the plateau in the R-curve generated as a result of fiber bridging.

Furthermore, results will reduced using both Berry's method, as done previously in the ASTM round robins, and a modified beam theory method currently used by EGF and JIS. The 0.5 mil sprayed Kapton insert AS4/PEEK data, generated from three ASTM laboratories during the second round, were reduced and plotted (see enclosure #3). Values measured using the load at onset of nonlinearity were lower than values measured using the load when the delamination was observed visually at the edges. For the brittle AS4/3501-6 specimens, these two values were identical. Hence, the cause of the nonlinearity in the load deflection trace, before visual confirmation of delamination onset at the edges, must be identified for the AS4/PEEK. Previous work by EGF participants indicated that the delamination formed on the interior before it was visible on the specimen edges. Hence, for the international round robin, several labs will be asked to terminate loading between these two points and section specimens to identify when the delamination forms. For all four loads, the modified beam theory data reduction method yielded slightly lower G_{Ic} values than Berry's method.

MINUTES OF ASTM TASK GROUP D30.02.02 ON INTERLAMINAR FRACTURE TOUGHNESS

HILTON PALACIO DEL RIO HOTEL
SAN ANTONIO, TEXAS

NOVEMBER 13, 1990

The meeting was opened at 2:30 pm by the chairman, Kevin O'Brien.

The chairman began by reviewing the purpose for the international round robin conducted for the Double Cantilever Beam (DCB) test by ASTM, the European Group on Fracture (EGF), and the Japanese Industrial Standards (JIS) group. This round robin was initiated in response to concerns raised by the EGF representatives at the November 1989 task group meeting in Orlando. Specifically, because the load-displacement curve becomes nonlinear before delamination onset is visually observed at the edge of the DCB specimens for a toughened matrix composite such as AS4/PEEK, it was not clear how to measure a valid initiation value for these materials. Therefore, it was agreed in Orlando to conduct a joint round robin where initiation would be measured in three ways: (1) by recording the first deviation from linearity in the load-displacement plot (NL), (2) by visually observing the onset of delamination at the edge of the DCB specimen (VIS), and (3) by plotting a 5% offset line from the original linear load-displacement curve and recording its intersection with the nonlinear portion of the curve (5%). All of these "initiation" values were plotted along with subsequent "propagation" values to produce a delamination resistance curve (R-curve) for each specimen tested. The data was reduced using three different data reduction methods: (1) a modified beam theory (MBT), (2) a compliance calibration method commonly referred to as Berry's method (BRY), and a modified compliance calibration method (MCC).
As a prelude to this round robin, data from DCB specimens with 13 micron Kapton film inserts, previously tested in the last ASTM round robin, were reduced as per the planned international round robin. Mean values and standard deviations were recorded for each data reduction scheme. This information was later compared to the results from the international round robin.

AS4/PEEK panels were manufactured by ICI in Wilton, England, for the DCB round robin. Panels were manufactured with either 13 or 7 micron aluminum foil inserts. Four panels with 13 micron inserts and 2 panels with 7 micron inserts were sent to NASA Langley to cut into specimens and to distribute to ASTM and JIS participants. A similar set of panels was sent to EPFL in Lausanne, Switzerland, to cut into specimens and distribute to EGF participants. Unfortunately, X-ray photographs taken at NASA Langley showed many tears present in the aluminum foil inserts. The specimens cut from panels at NASA were screened prior to distributing to participants. This resulted in rejection of 50% of the 13 micron insert specimens and 70% of the 7 micron insert specimens. The ASTM and Japanese participants were not sent any specimens with tears. The EGF participants, however, received specimens that were not screened beforehand, and it was left up to the individual laboratories to check their specimens. All participants ran the tests by following a common protocol.

Kevin O'Brien then reviewed the data for the ASTM and Japanese participants. There was very little difference in G_{Ic} values measured using the three different data reduction methods outlined in the round robin protocol. However, the Modified Beam Theory (MBT) data reduction method yielded the most conservative results for 85% of the ASTM and JIS tests that were performed. Hence, this method will be recommended as the preferred data reduction method in the draft standard. Kevin also summarized results in the form of bar charts showing the mean and standard deviations used to determine repeatability and reproducibility. The repeatability parameter, S_r, is the average of the standard deviations for each laboratory, and the reproducibility parameter, S_R, is the standard deviation from the mean G_{Ic} values measured for all the laboratories. Several significant observations were noted. First, the NL, VIS, and 5% offset measurements all had very similar repeatability and reproducibility. Hence, contrary to intuition, it was no more difficult to measure the first point of nonlinearity in the load-deflection curves, or the first visual observation of delamination onset from the edges, than it was to determine the intercept of a line drawn at a 5% offset compliance with the load deflection curve. Furthermore, in 85% of the specimens tested by ASTM and JIS participants, the visual observation of delamination on the edge occurred before the 5% offset point. Hence, the validity of a 5% offset G_{Ic} value as an initiation value is questionable. Other trends in the data that caused concern were the observations that the 7 micron insert values were greater than the 13 micron insert results, and that both yielded values 50% greater than values measured in the previous ASTM round robin conducted on specimens with 13 micron Kapton Inserts. Furthermore, in 75% of the tests, the R-curves decreased during the first 5mm of growth, and eventually showed propagation values for G_{Ic} that were less than the G_{Ic} values obtained from the first non-linearity in the load-deflection trace (NL) or from the visual measurement of delamination at the edge (VIS). These decreases in the R-curves suggested that all the initiation values may be questionable for the tests made on these panels.

Several explanations for these unusual R-curves were postulated, but none have yet been verified. One explanation would attribute the behavior to residual stresses that develop in the resin pocket as a result of the mismatch in thermal coefficients of expansion between the resin and the aluminum foil. Another explanation would attribute the behavior to a variation in specimen thickness along the length of the beam. Yet another explanation would attribute the behavior to a change in the crystalline structure of the PEEK matrix in the vicinity of the aluminum foil. At a joint meeting between ASTM and EGF representatives in Switzerland in September (see minutes enclosed), Peter Davies noted that similar R-curves were obtained in recent tests at EPFL on specimens taken from panels of IM6/PEEK moulded at temperatures of 400 degrees Celsius and above. Specimens moulded at lower temperatures had the more typical increasing R-curve.

As a result of these observations, several actions are underway. First, ICI is reviewing their manufacturing records to determine the exact forming temperature that was used to consolidate all of the AS4/PEEK panels made for the international round robin and the previous ASTM round robins. In addition, microscopy studies are being undertaken by ICI to establish the effect of the insert material on crystallinity in the PEEK matrix. In addition, the Swiss Federal Laboratory near Zurich will be conducting DCB tests on specimens remaining from all the previous round robins, including specimens with aluminum and Polyimide film inserts of different thicknesses, using an in-situ dye-penetrant-enhanced video. X-Radiography technique. They have used this technique previously during DCB tests of IM6/PEEK composites to demonstrate that the first nonlinear point in the load-deflection curve corresponds to the onset of delamination from the insert in the interior of the laminate width.

These results were presented at the September meeting in Switzerland. Confirmation of this interior initiation for the round robin data base will help justify the use of the first point of nonlinearity as the most appropriate initiation value.

A second batch of material was made by ICI to generate more specimens for testing because of the lost specimens due to foil tearing. Rod Martin conducted tests at NASA Langley on specimens with different widths made from this second batch of material. No width effect of significance was observed. However, when these data were compared with round robin data from the first batch, and with data from the previous ASTM round robin generated using Kapton inserts, some interesting trends were evident. All of the tests using aluminum inserts showed a much greater difference between the NL and VIS values than were observed with the Kapton insert specimens. In order to verify this trend, and help determine the cause of the unexpected insert material dependence on initiation values and decreasing R-curves, it was decided to have more labs run DCB tests on specimens made with ICI's "Upilex" film inserts, as is currently planned for the ENF and MMB round robins. Upilex is a polyimide film similar to Kapton. These tests, along with similar AS4/3501-6 DCB tests using 7 and 13 micron Upilex film inserts, should also provide the data base needed to identify any insert thickness dependence in the initiation values, and to support the required precision and bias statement in the DCB standard.

The draft ASTM Standard for the DCB test will be forwarded to the round robin participants and task group members for review prior to submitting it for balloting. It is hoped that the new tests on specimens with Upilex film inserts will be completed in sufficient time for the results to be incorporated in the draft standard, and supporting documents, before balloting.

MINUTES OF THE ASTM D30.06
SUBCOMMITTEE ON INTERLAMINAR PROPERTIES
MAY 8, 1991
INDIANAPOLIS, INDIANA

The meeting was called to order at 8:00 am by chairman Kevin O'Brien. The attendance list is given in attachment 1. The chairman first reviewed the agenda for the meeting (attachment 2). The meeting began with a discussion of the new D30 committee structure. The Executive Committee currently lists 42 voting and 17 non-voting members of D30.06. This list includes those who responded to Dale Wilson's letter to all D30 members requesting interest in the new subcommittees. Those people not on the current list who wish to participate in D30.06 should contact Kathy Schaaf, ASTM D30 Staff Manager. Kevin O'Brien will notify all members of the old D30.02.02 task group who do not appear on the new D30.06 membership list. It was proposed by the chairman that Rod Martin and Gretchen Murri, both at NASA Langley Research Center, serve as vice-chairman and secretary, respectively, to the new D30.06 subcommittee. A motion was made to accept these officers by Peter Shyprykevich and was seconded by Steve Hooper. The motion was voted on and approved. This was followed by a review of the proposed charter of the subcommittee and the proposed standards-development process.

The current status of the DCB round-robin activity was discussed by the chairman (attachment 3). A proposed DCB testing standard has been drafted and was submitted for comment with the D30.02.02 Task Group minutes from the November 1990 meeting in San Antonio. Kevin O'Brien asked that anyone having comments should refer them to specific sections in the draft standard and return them to him as soon as possible. The revised draft standard will be used as the testing protocol for the next DCB round robin. The issues that still must be resolved for the DCB test are: the dependence of the initiation value of G_{Ic} on the insert thickness for graphite composites; and, for toughened matrix graphite composites, the technique to use for determining the initiation value of G_{Ic} and the dependence of G_{Ic} on insert material. Results of a recent study by Gretchen Murri and Rod Martin showed that for a glass/epoxy material, G_{Ic} values measured using the DCB specimen showed a minimum level for specimens that contained inserts that were 75μm (3.0 mil) thick or thinner. Results for a few labs (3) from the current round-robin indicate that for AS4/3501-6 and AS4/PEEK, G_{Ic} values continue to decrease with decreasing insert thickness for inserts thicknesses ranging from 12.5 to 50.0μm (0.5 to 2.0 mil). Results were also shown from the current round-robin tests comparing G_{Ic} values for AS4/PEEK material, calculated using four different techniques. The load and displacement used in calculating G_{Ic} can be chosen at the point of deviation from linearity of the load-displacement curve (NL), the point of visual observation of delamination growth (VIS), the intersection of the load-displacement curve with a line corresponding to a 5% decrease in the initial compliance of the specimen (5%), or at the plateau value (PLAT) of the R-curve. Therefore, a final round of testing with a brittle and a tough matrix material will be conducted by selected labs to resolve these issues. Hercules has stated that they will not be able to make the AS4/3501-6 panels, however they will supply the prepreg. NASA Langley will manufacture the AS4/3501-6 panels and

ICI will manufacture AS4/PEEK panels. Panels of both materials will be made using inserts of 7 and 13μm Upilex film supplied by ICI. The panels will be cut at NASA Langley and distributed to the participating labs. Ron Zabora of Boeing Commercial Aircraft pointed out that the results of these insert effect studies may not apply to interlayer materials. Kevin O'Brien noted that the draft standard is currently limited to graphite composites with single phase matrices. Further round robin testing on interleaf systems would be needed to expand this scope. There was also some discussion concerning the variability of the G_{Ic} results for the AS4/PEEK specimens with different insert materials (aluminum and Kapton) used in the round robin testing. Kevin O'Brien noted that average NL G_{Ic} values were 10% lower for the 13μm Kapton inserts versus the 13μm aluminum inserts specimens. Roy Moore of ICI determined that the manufacturing procedure had been identical for all the AS4/PEEK panels used in the previous round-robin, so manufacturing differences do not explain the different test results. Microscopy studies performed by ICI, in Wilton, U.K., showed no difference in the crystallinity at the end of the two inserts, but did show a small fold at the end of the aluminum insert specimens. Kevin also pointed out that the summary of round-robin results for specimens with 13μm inserts showed that the coefficient of variation was greatest for the NL measurements and lowest for the 5% offset measurements, but the 5% offset mean values were 20% higher than the NL mean values. Kevin expressed his opinion that because the EMPA in-situ X-ray studies had shown that the delamination initiates at the end of the insert in the interior of the specimen width at the NL point in the load versus opening-displacement plot (see San Antonio meeting minutes), that the NL value should be used as the initiation G_{Ic} measurement, even though the scatter is slightly higher than for the 5% offset measurements. There was further discussion on whether the mold-release spraying process used with Kapton and Upilex inserts should be included in the test procedure to ensure that it is done properly.

The effect of anticlastic bending on DCB specimens was discussed briefly. An analysis developed by Barry Davidson of Syracuse University was used to show that for both graphite/epoxy and glass/epoxy unidirectional specimens, anticlastic bending reduces G only slightly compared to the uncorrected value.

MINUTES OF THE D30.06 SUB-COMMITTEE ON INTERLAMINAR PROPERTIES

Wednesday, October 16th 1991
8:00-10:00
San Diego, California

The meeting was called to order by the Chairman Kevin O'Brien at 8:00am. There were 15 people in attendance. Approval of the minutes of the previous D30.06 meeting in Indianapolis was proposed by Rod Martin and seconded by Mark Spearing. The Chairman then gave an introduction detailing the current D30.06 membership at 67 members, 44 voting and 23 non-voting. He also gave an agenda for the meeting (enclosure #1).

A review of the current status of the DCB round robin and standard was then given by Kevin O'Brien (enclosure #2). Kevin stated that AS4/PEEK panels had been made with both 7 and 13 micron Upilex film inserts. Upilex film was used because it was available in a 7 micron thickness whereas 7 micron Kapton was difficult to obtain. These panels were to be used to determine the effect of insert thickness on initiation values of G_{Ic}. Sufficient specimens for 9 labs were made and the results are due December 1st, these labs are given in enclosure #2. So far only one lab has returned the results. In addition, DCB specimens have been sent to EMPA to use their real time X-ray equipment to identify the location of delamination initiation. They have found that the delamination initiates in the interior. As the delamination grows it extends further in the interior than at the edges. Using this X-ray technique EMPA found it difficult to measure the delamination length. This was because the specimen moved and also because the dye penetrant did not penetrate into the newly formed delamination. Also, AS4/3501-6 specimens were made with the Upilex film but unfortunately the inserts stuck even though different release agents and methods of application were attempted. It is not anticipated to re-make these panels because all the data collected on the AS4/PEEK material should be sufficient for the standard. The draft standard has now undergone its final revision and will shortly be submitted for sub-committee ballot.

Minutes of the ASTM D30.06 Subcommittee on Interlaminar Properties
Wednesday, May 6, 1992
1:00 - 3:00 p.m.
Pittsburgh, PA

The meeting was called to order at 1:00 p. m. by Chairman Kevin O'Brien. There were 23 people in attendance (enclosure 1). Kevin began the meeting by reviewing the agenda (enclosure 2). Those ASTM members who wish to be added to Subcommittee D30.06 were asked to contact either Kathy Schaff at ASTM headquarters, or Subcommittee secretary Gretchen Murri.

The first topic was a review of the status of the static DCB Round Robin and Standard (enclosure 3). For specimens with either 13 or 7.5 μm thick Upilex inserts, consistent G_{Ic} values were obtained by the participants using the three different data reduction techniques - nonlinear (NL), visual (VIS) and 5% offset (5%). The 13 μm aluminum inserts that were used in some specimens were found to have crimps that resulted in unreliable data. It was recommended that only polymer films (Kapton, Upilex, etc.) be used for insert materials in future specimens. A comparison of G_{Ic} values for the various insert materials and thicknesses used showed that the lowest values were obtained from specimens with the Upilex inserts. Since there was very little difference between results from the 13 μm and 7.5 μm inserts, it was recommended that 13 μm film be used since it is easier to get and easier to use than the 7.5 μm material. The reproducibility of results between the participating labs, and the variation in G_{Ic} for the different insert materials and thicknesses were discussed. It was recommended that the nonlinear-visual (NL-VIS) technique be used. A summary of the Round Robin data from all the participating groups was presented. The Round Robin results from the tests on AS4/PEEK specimens with Upilex inserts provided sufficient results for a precision and bias statement. A draft standard of the DCB test is now ready for subcommittee ballot. Recommended procedures are given in the chart in enclosure 3. Results of the balloting will be reviewed at the ECCM Conference in Amsterdam in September 1992 and at the ASTM Conference in Miami in November 1992.

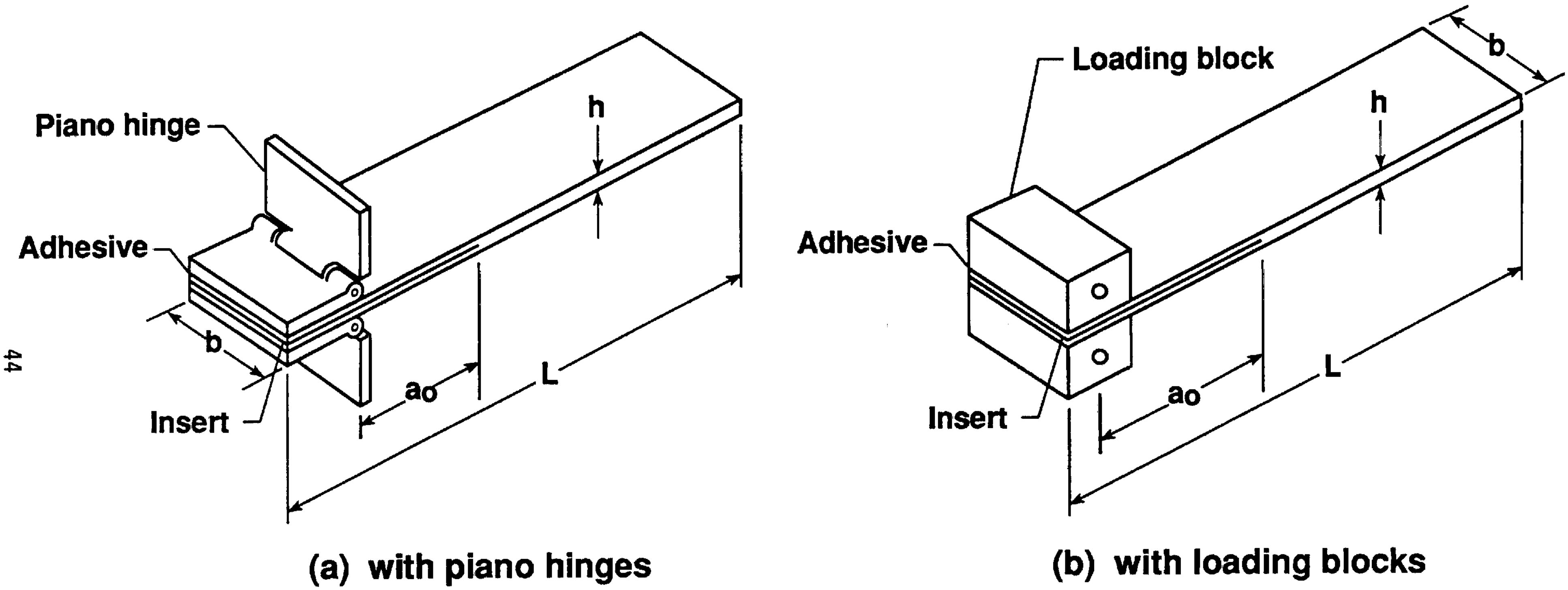


Figure 1. Double cantilever beam specimen

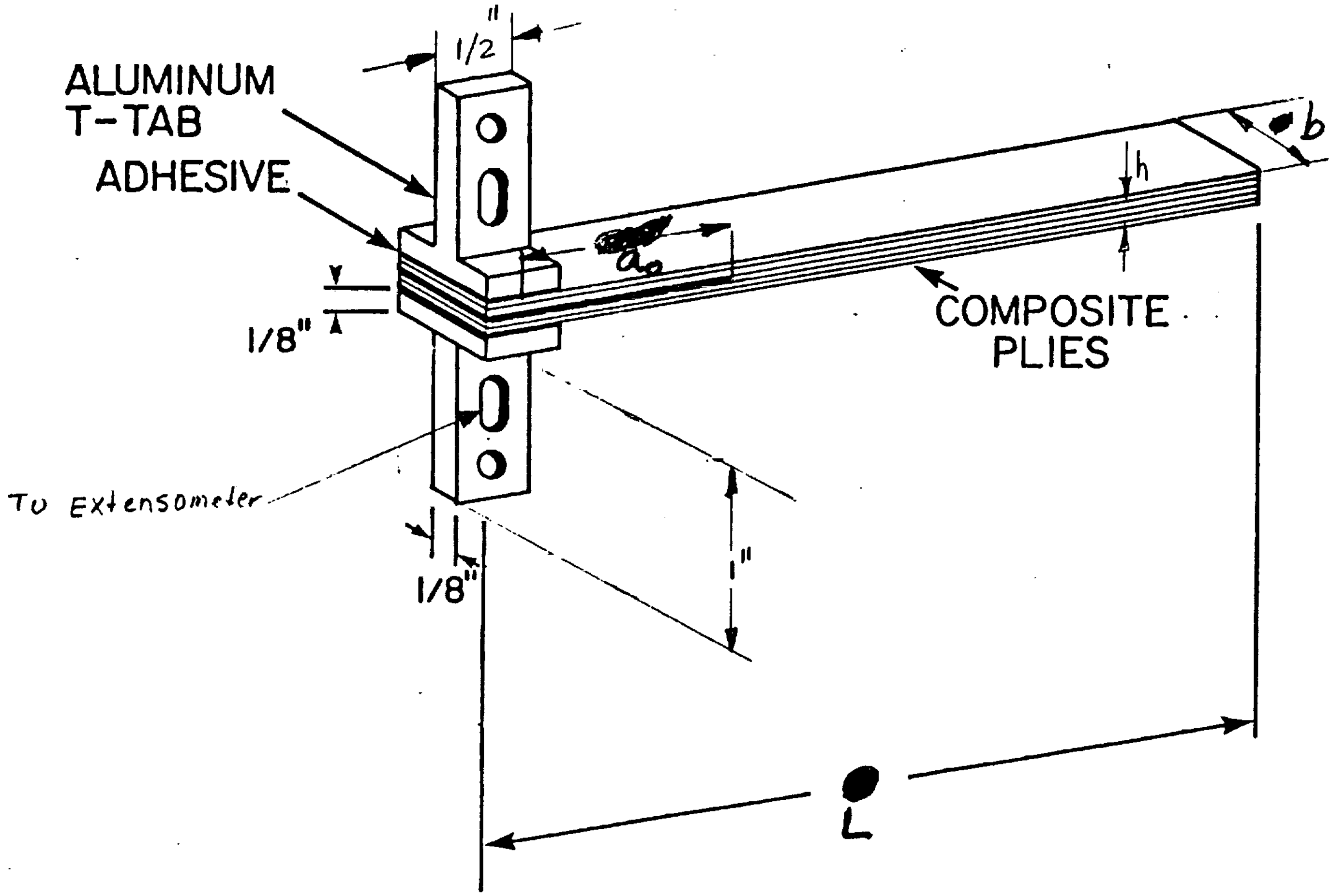
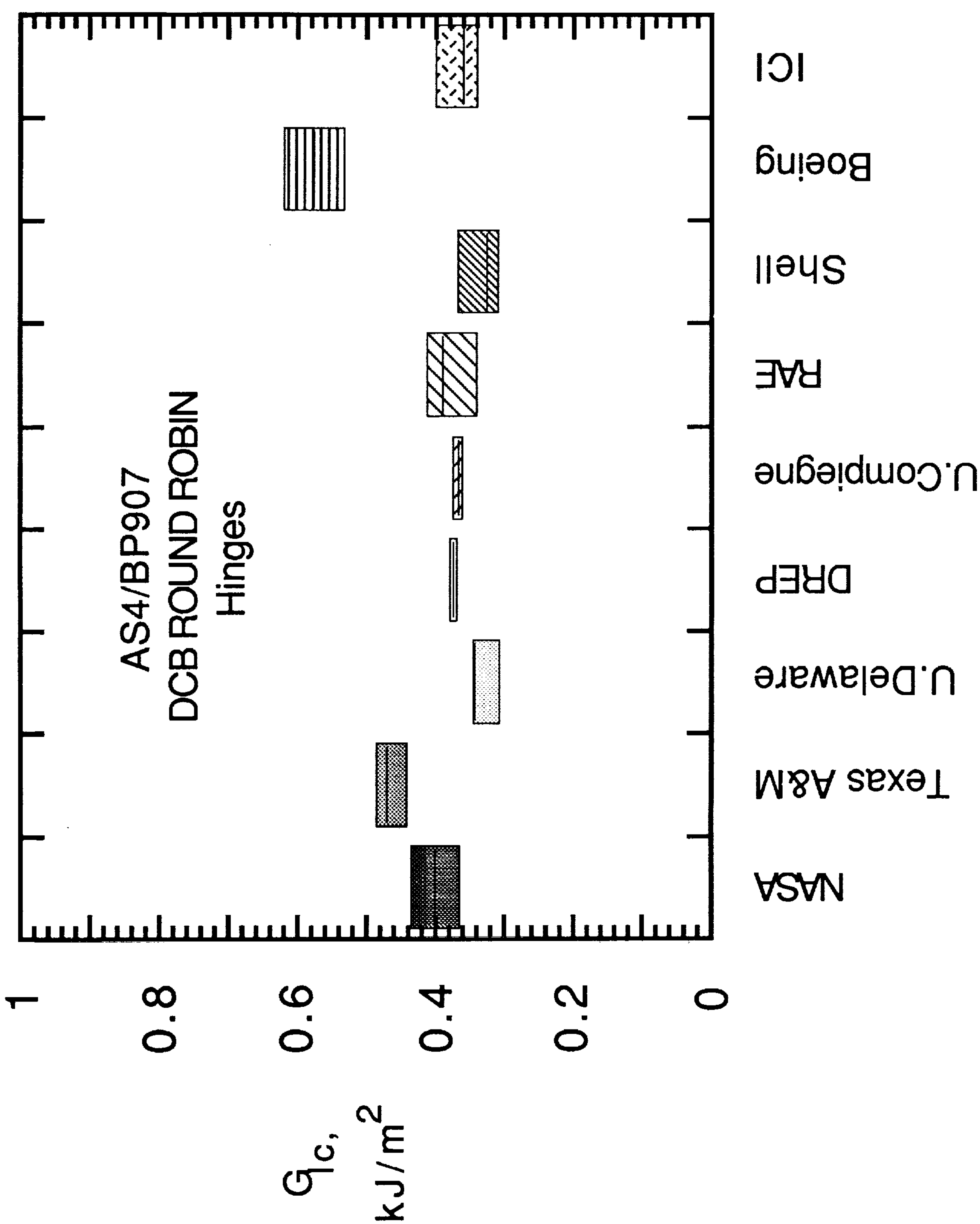
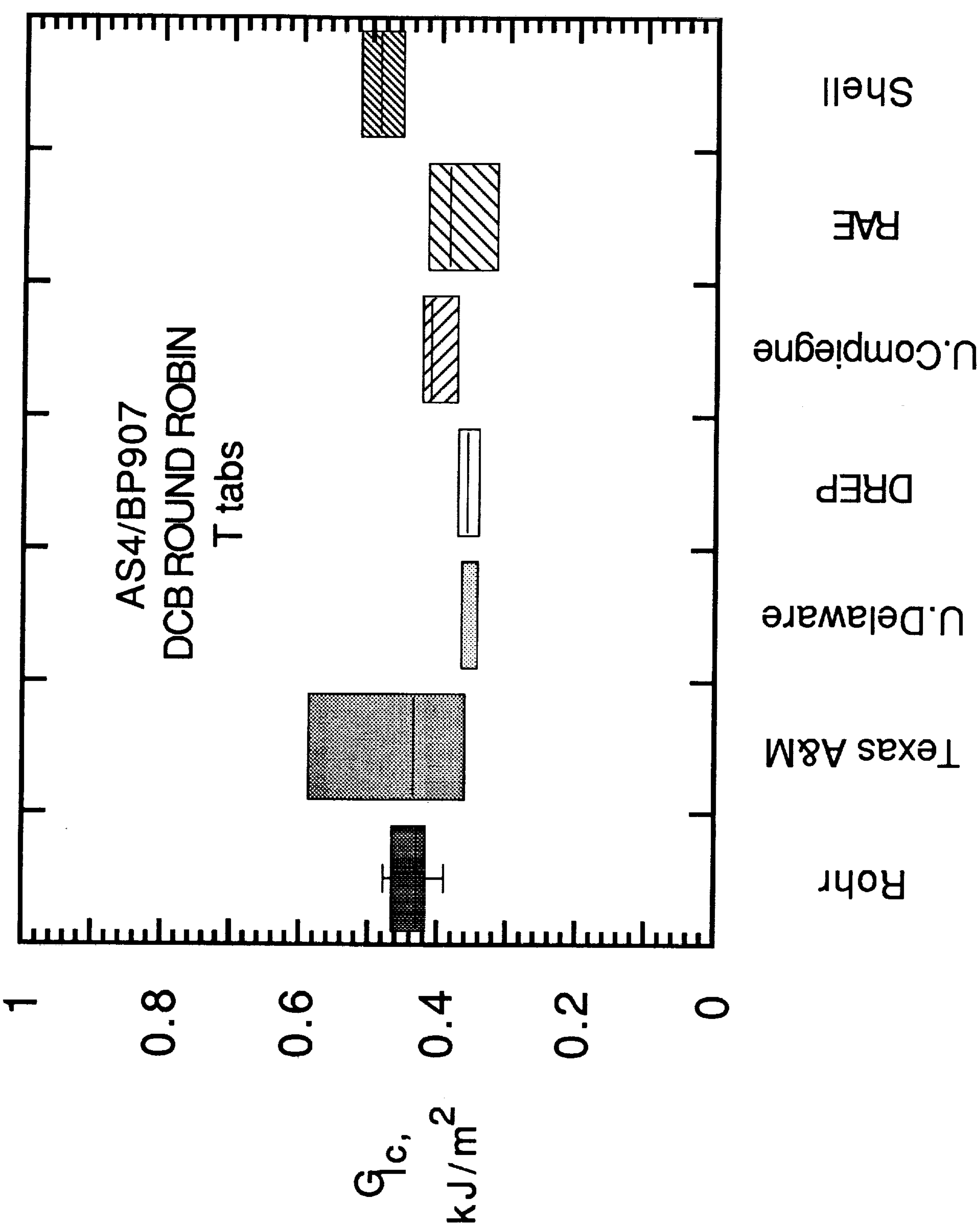
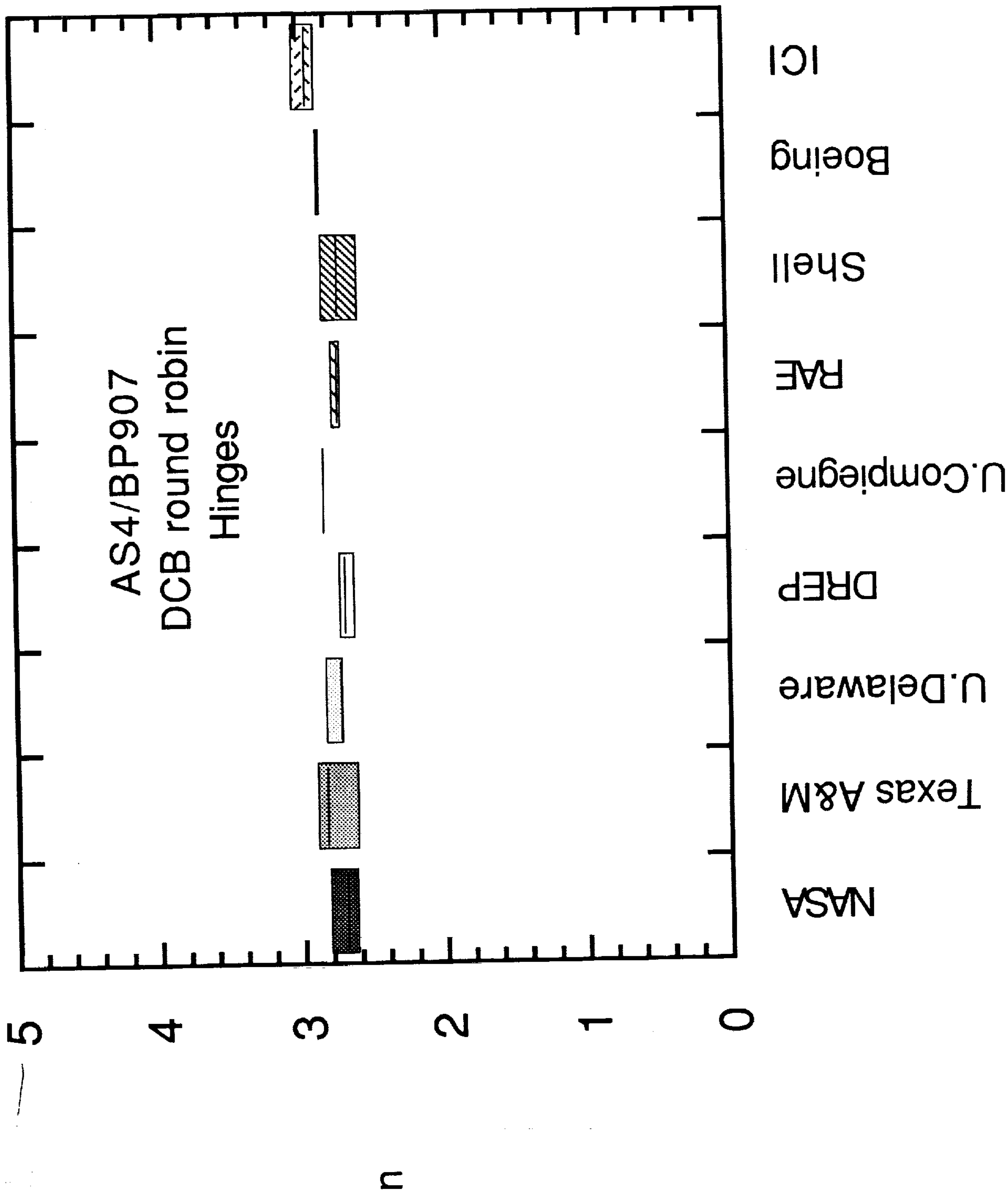
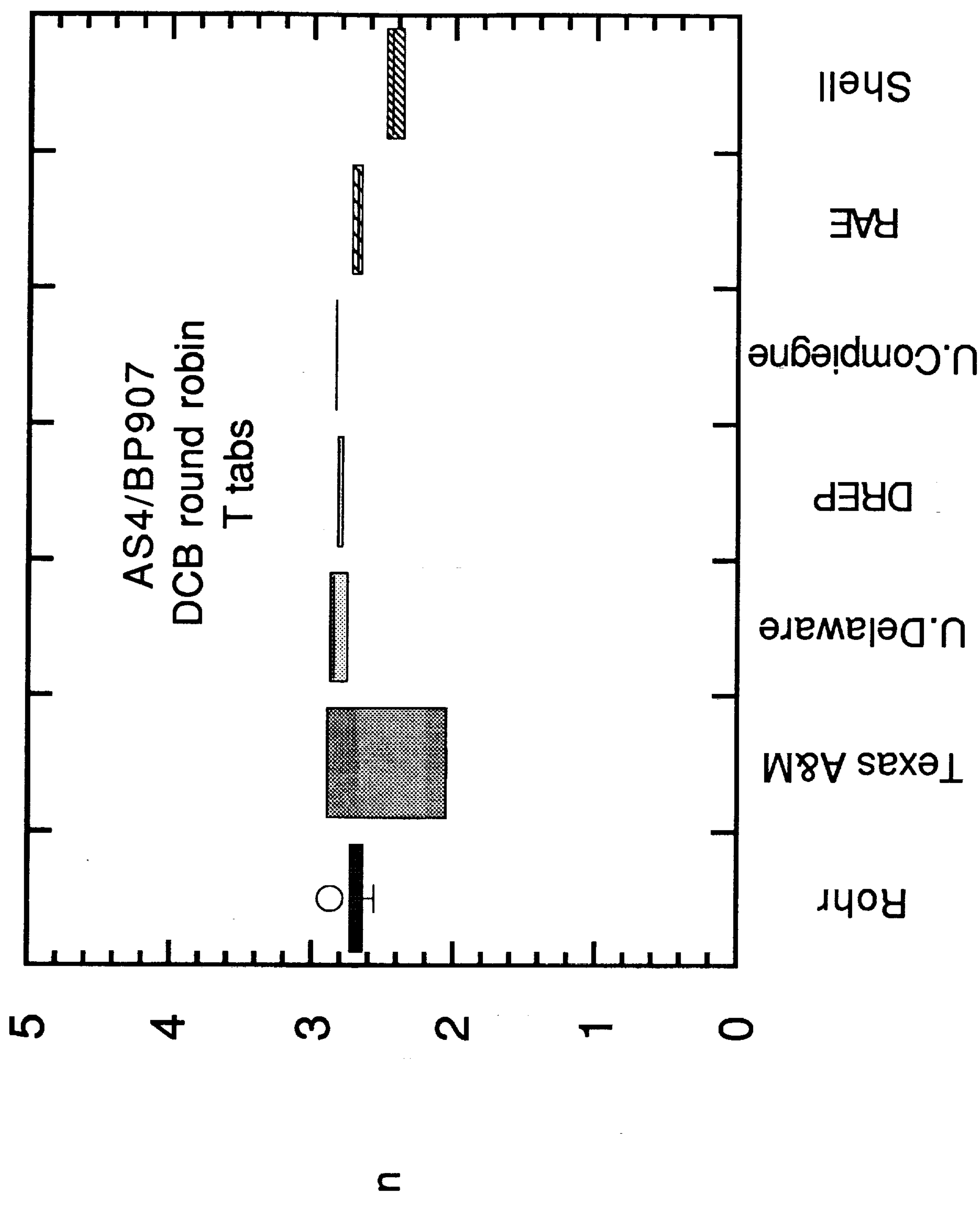


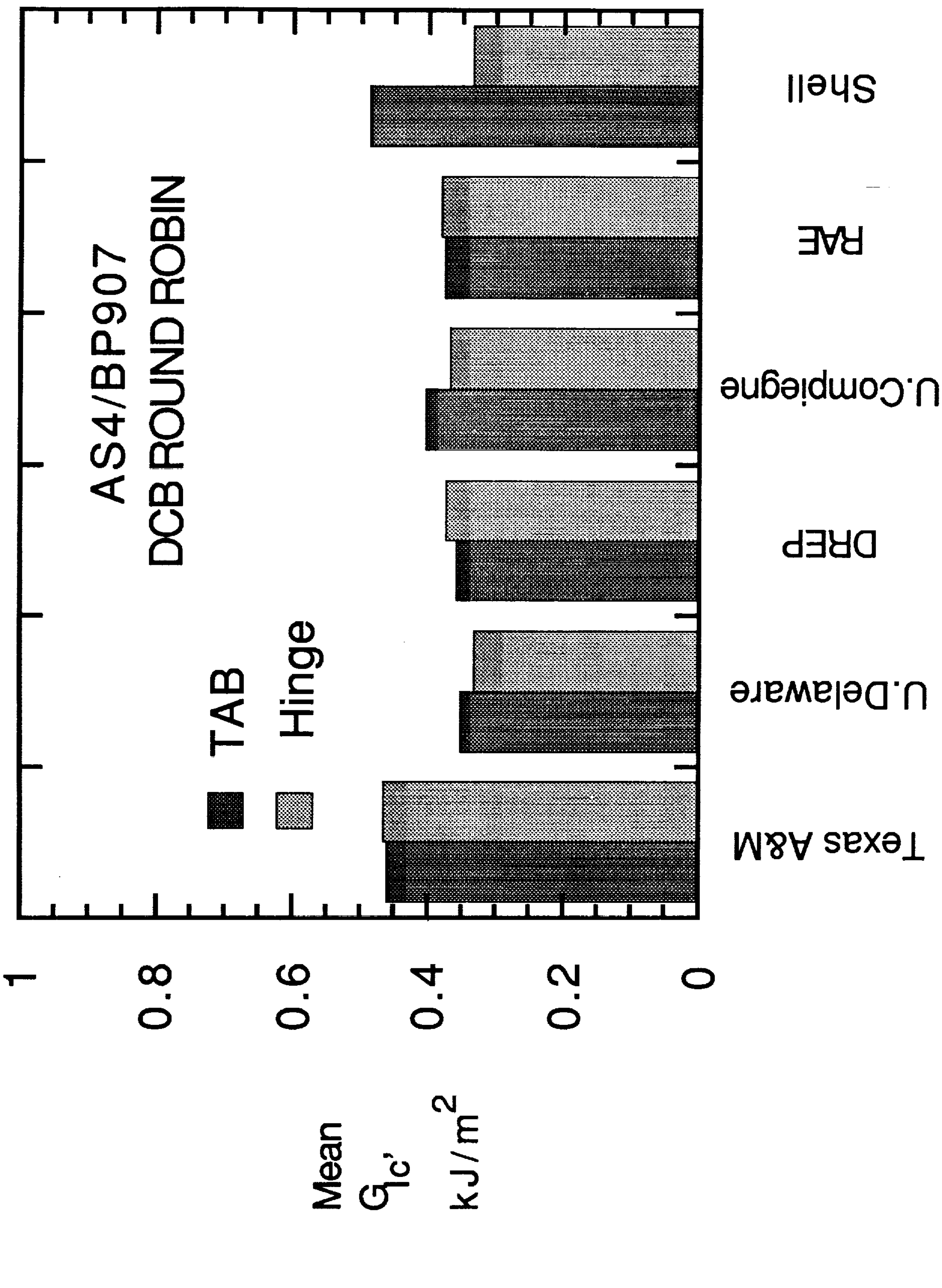
Figure 1c. - "T" Type Tab DCB Specimen


AS4/BP907
DCB ROUND ROBIN
Hinges
NASA
Texas A&M
U.Delaware
DREP
U.Compiegne
RAE
Shell
Boeing
ICI
0
0.2
0.4
0.6
0.8
1
G_{Ic},
kJ/m^2

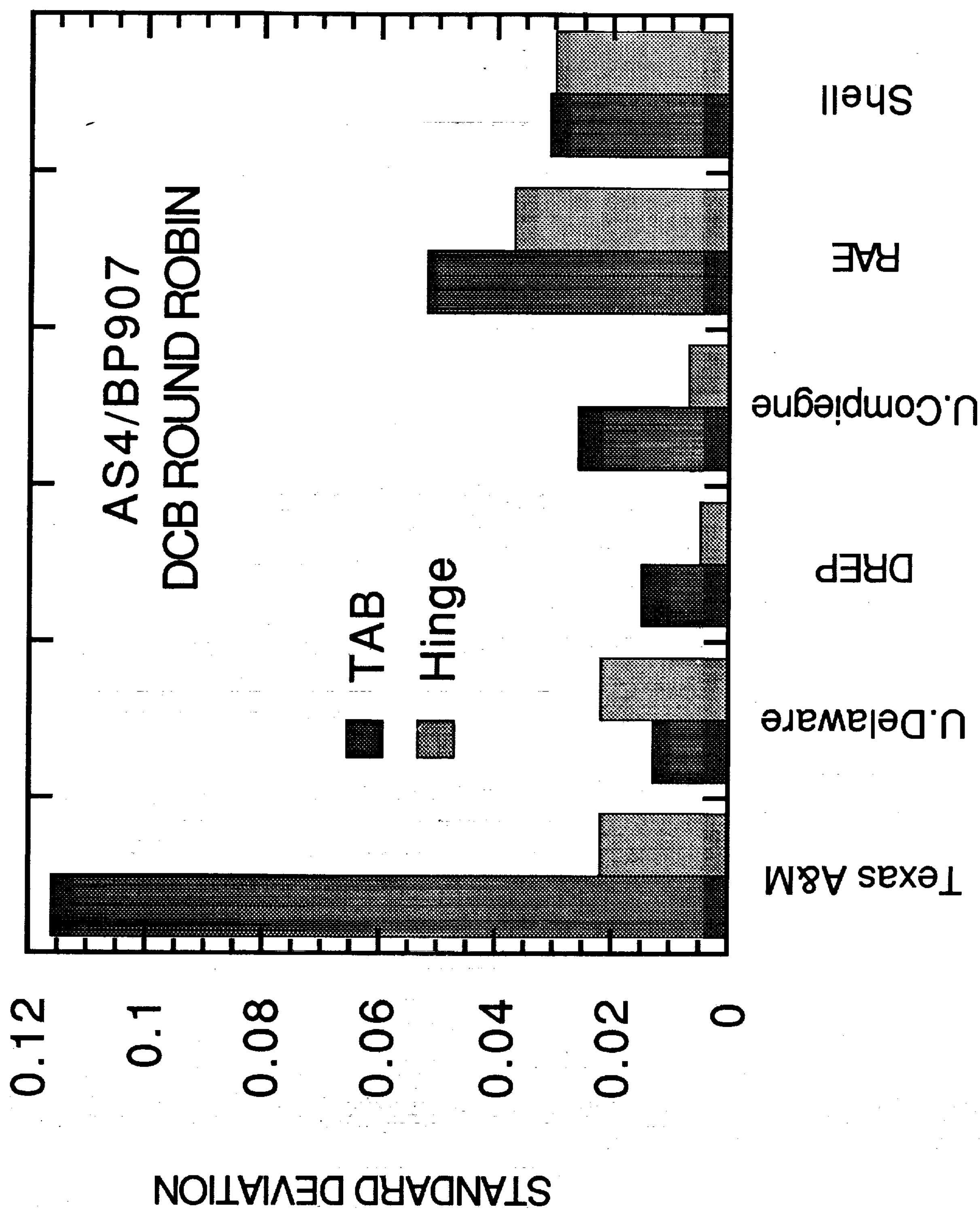

AS4/BP907
DCB ROUND ROBIN
T tabs
G_Ic, kJ/m^2
0
0.2
0.4
0.6
0.8
1
Rohr
Texas A&M
U.Delaware
DREP
U.Compiegne
RAE
Shell

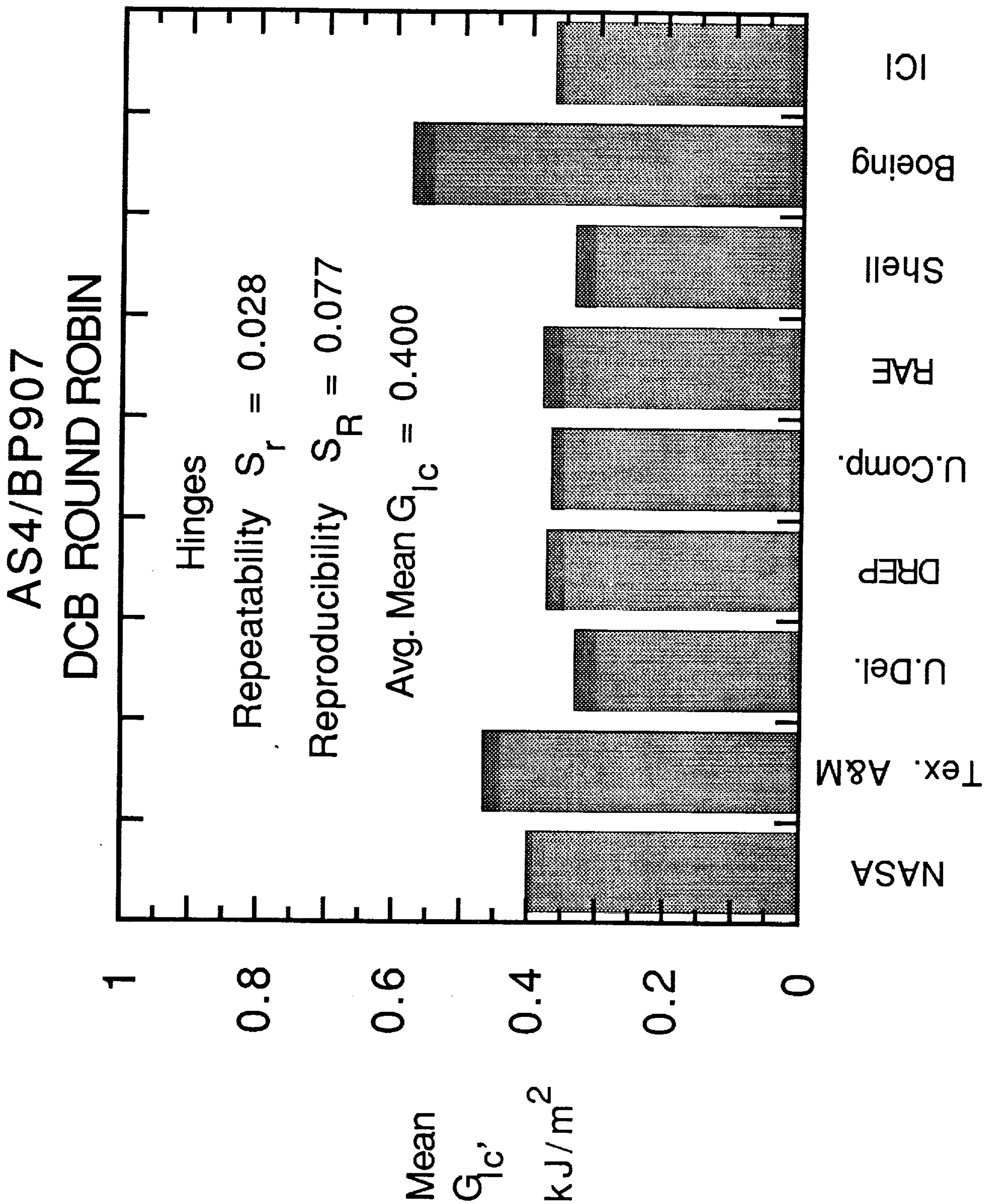

AS4/BP907
DCB round robin
Hinges
NASA
Texas A&M
U.Delaware
DREP
U.Compiegne
RAE
Shell
Boeing
ICI
0
1
2
3
4
5
n

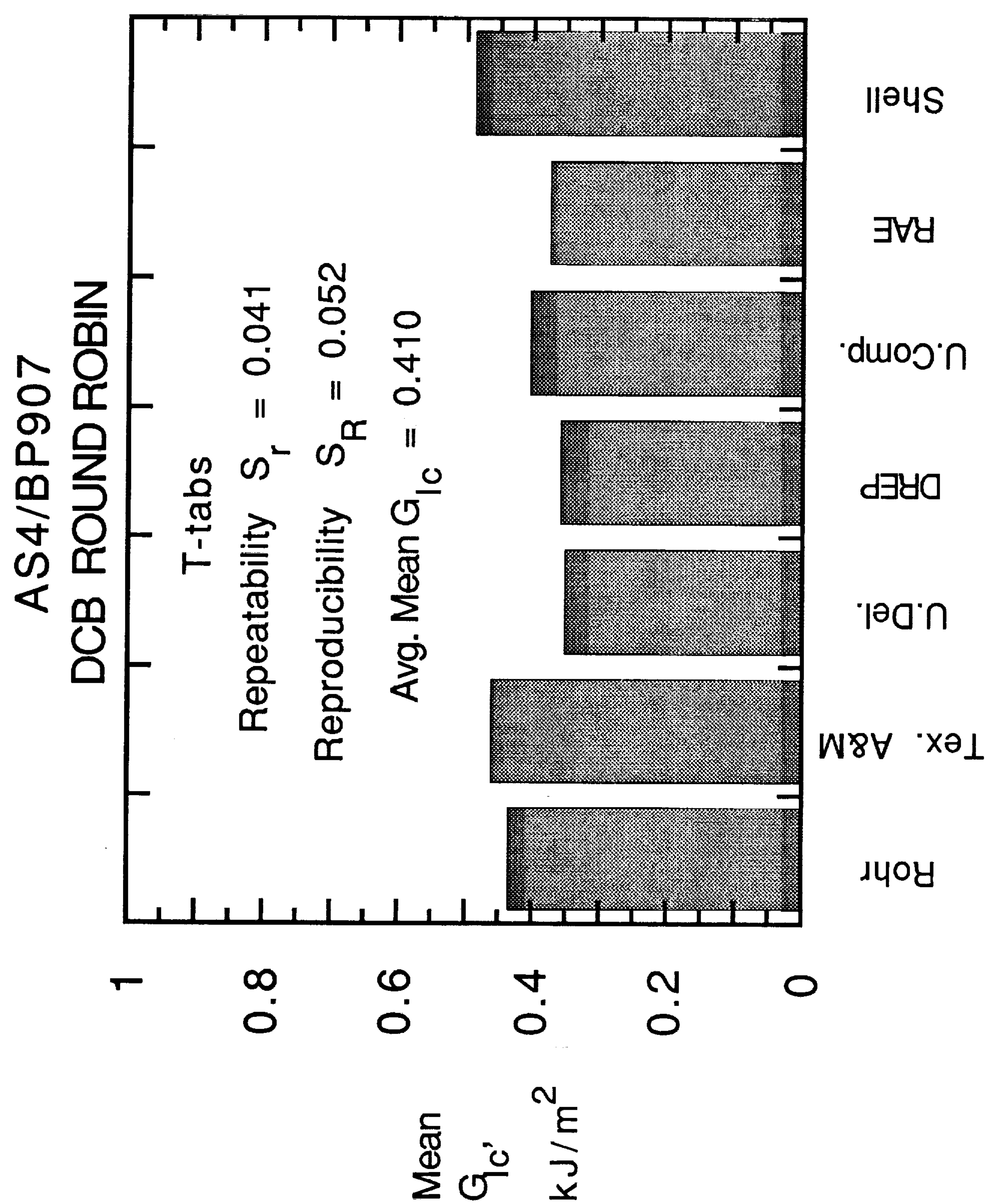

AS4/BP907
DCB round robin
T tabs
n
0
1
2
3
4
5
Rohr
Texas A&M
U.Delaware
DREP
U.Compiegne
RAE
Shell



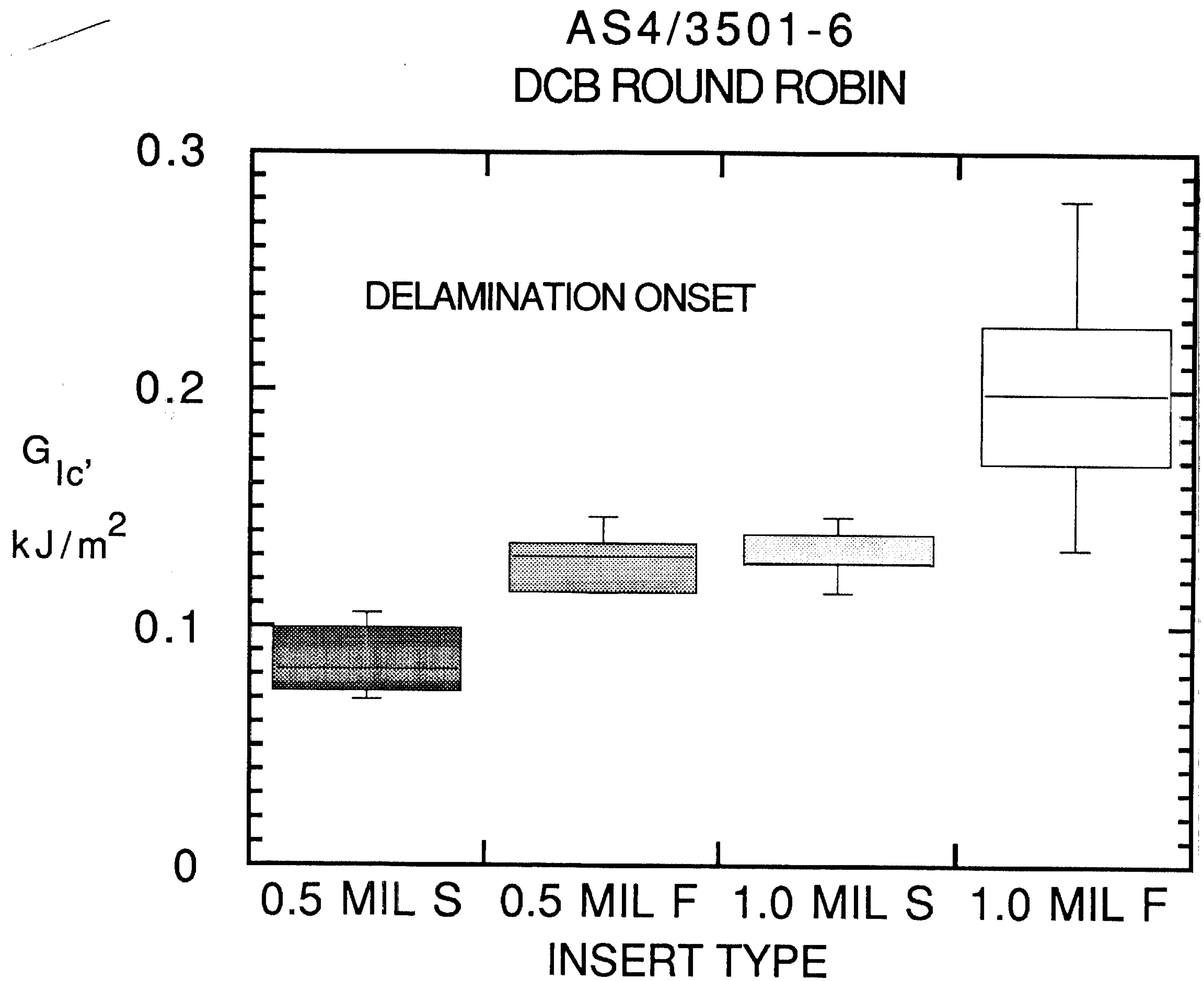
AS4/BP907
DCB ROUND ROBIN
TAB
Hinge
Mean
G_{Ic}'
kJ/m^2
0
0.2
0.4
0.6
0.8
1
Texas A&M
U.Delaware
DREP
U.Compiegne
RAE
Shell

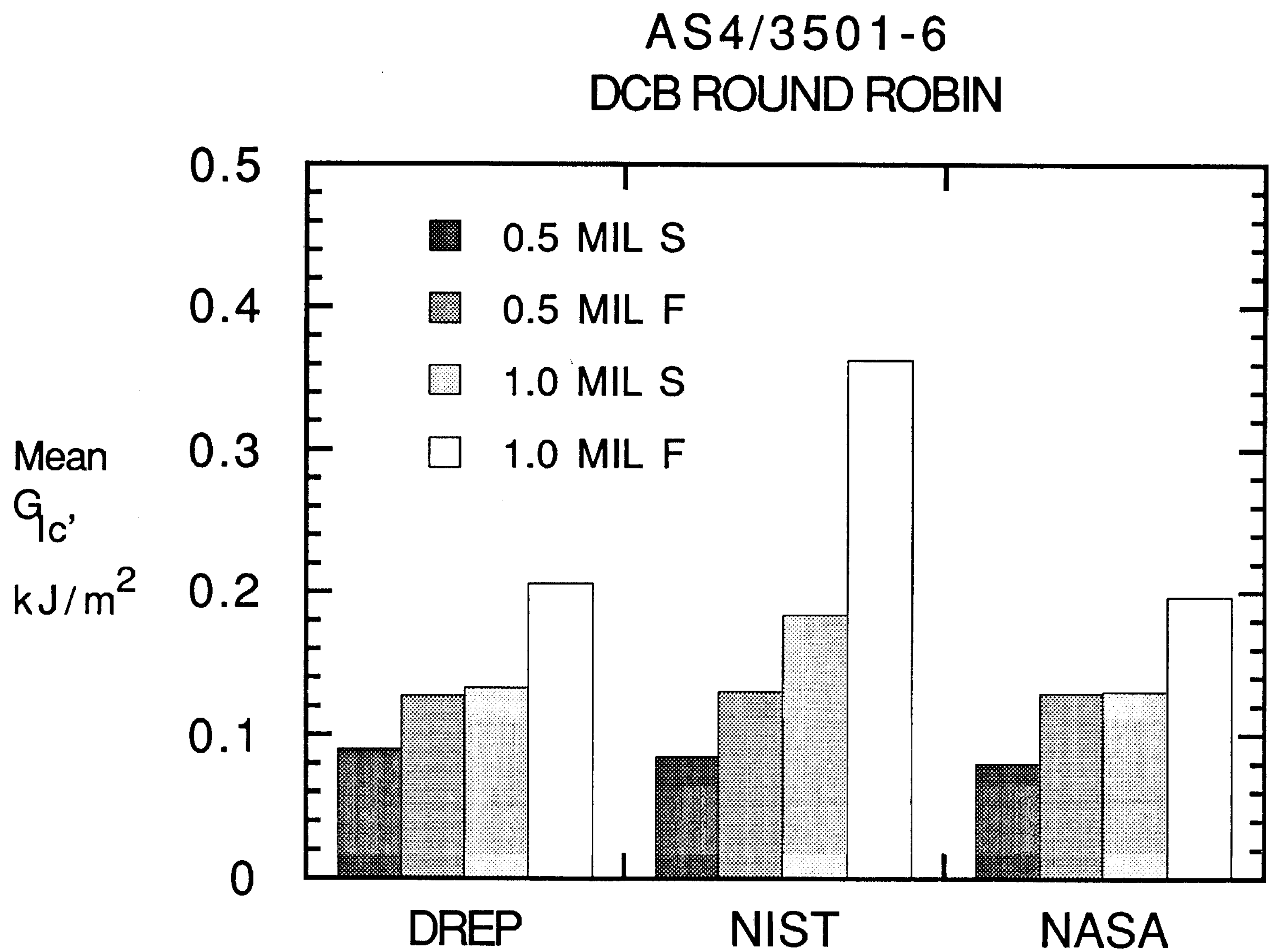

AS4/BP907
DCB ROUND ROBIN
TAB
Hinge
STANDARD DEVIATION
0
0.02
0.04
0.06
0.08
0.1
0.12
Texas A&M
U.Delaware
DREP
U.Compiegne
RAE
Shell

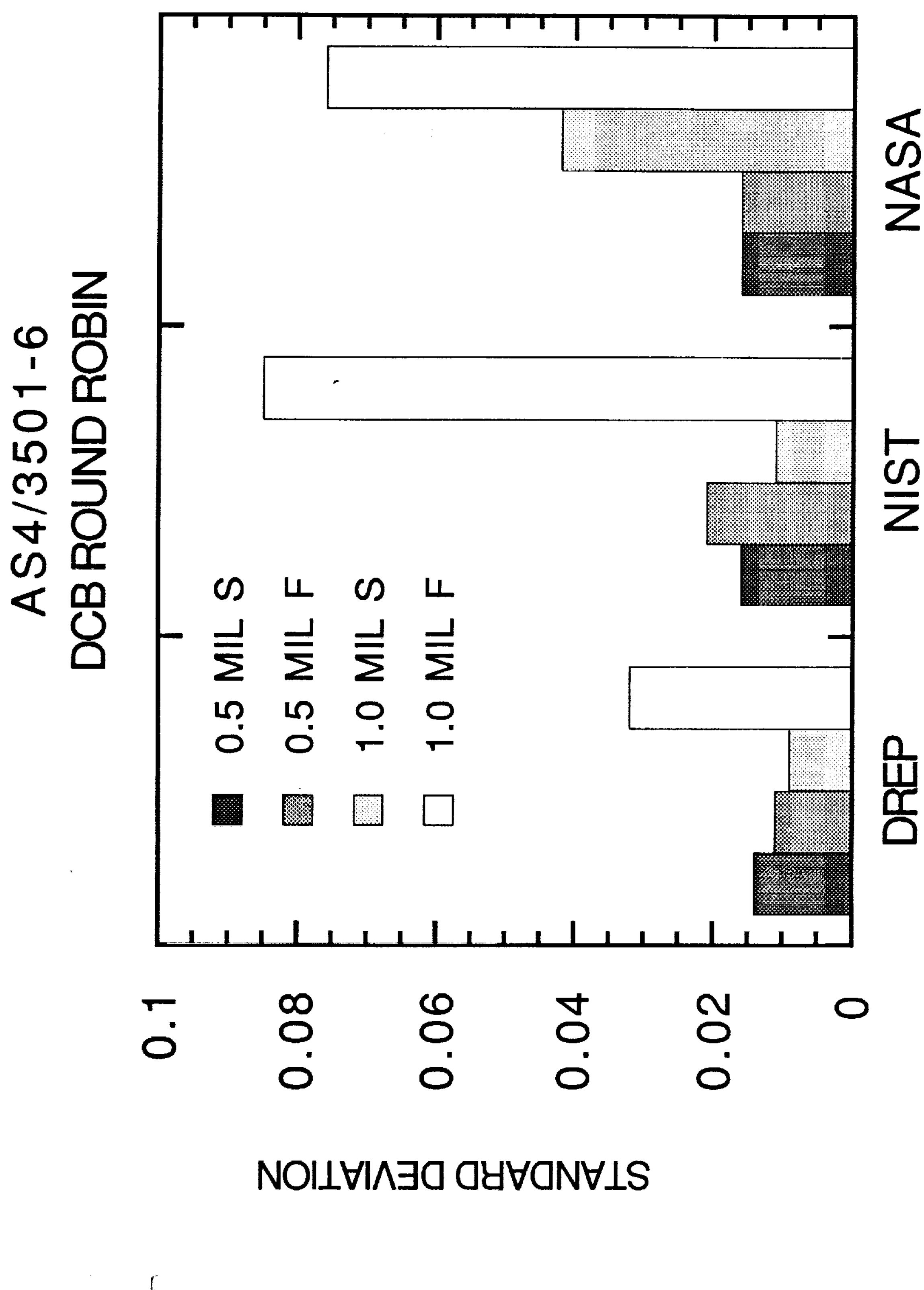

AS4/BP907
DCB ROUND ROBIN
Hinges
Repeatability S_r = 0.028
Reproducibility S_R = 0.077
Avg. Mean G_{Ic} = 0.400
Mean G_{Ic}, kJ/m^2
0
0.2
0.4
0.6
0.8
1
NASA
Tex. A&M
U.Del.
DREP
U.Comp.
RAE
Shell
Boeing
ICI

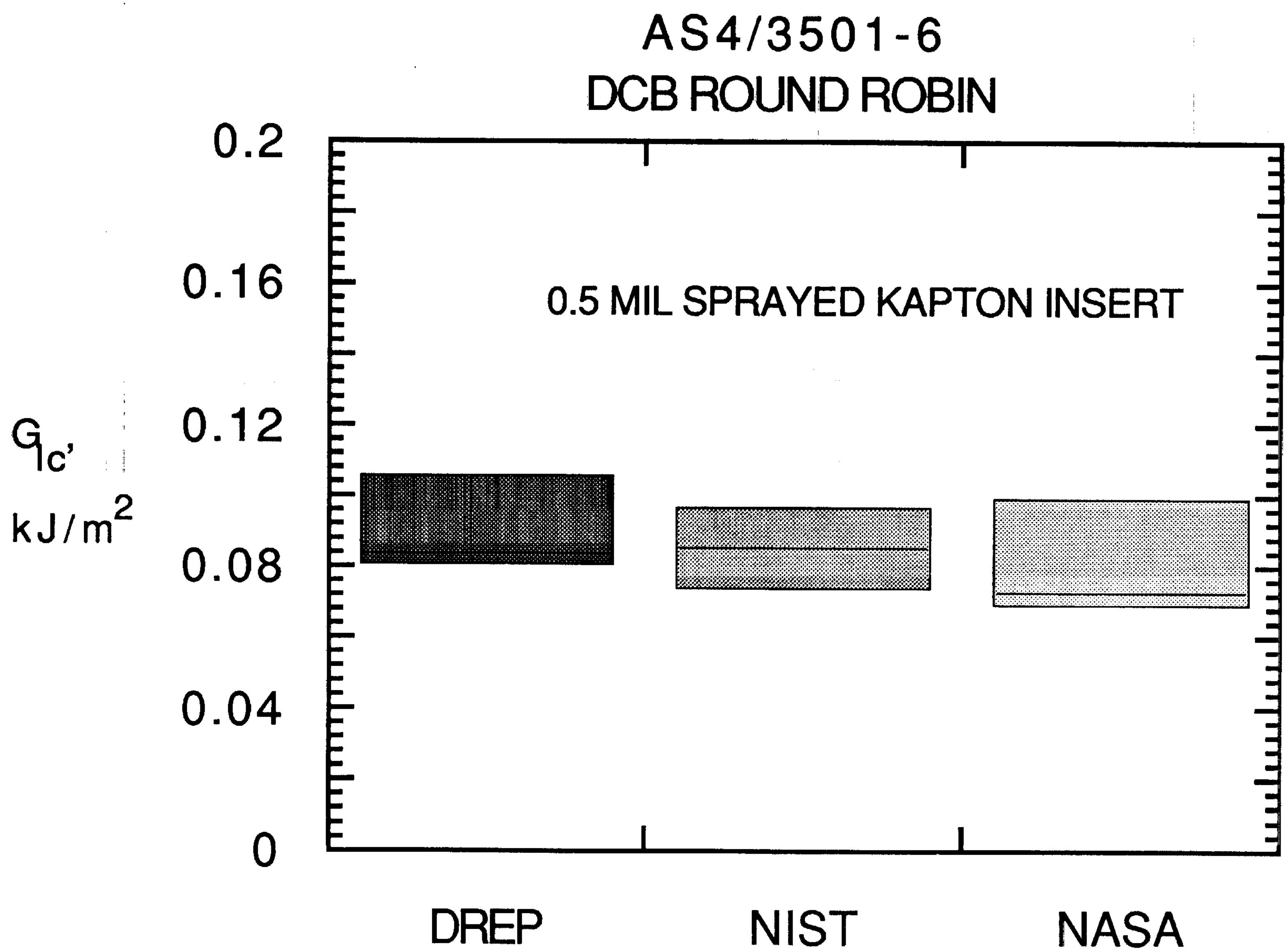


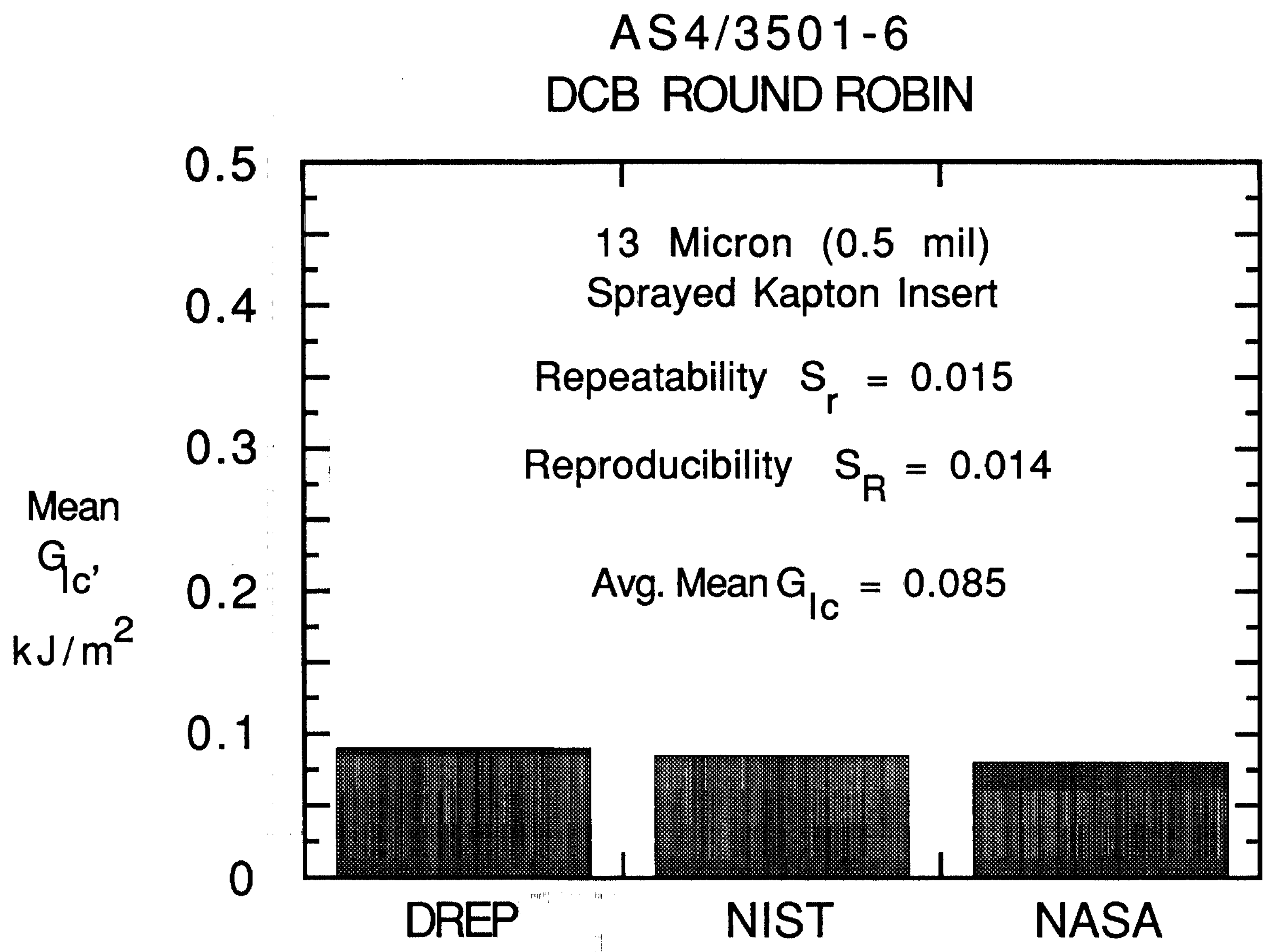
AS4/BP907
DCB ROUND ROBIN
T-tabs
Repeatability S_r = 0.041
Reproducibility S_R = 0.052
Avg. Mean G_{Ic} = 0.410
Mean G_{Ic}, kJ/m^2
0
0.2
0.4
0.6
0.8
1
Rohr
Tex. A&M
U.Del.
DREP
U.Comp.
RAE
Shell


AS4/3501-6
DCB ROUND ROBIN
DELAMINATION ONSET
0.3
0.2
0.1
0
$G_{Ic'}$
kJ/m^2
0.5 MIL S
0.5 MIL F
1.0 MIL S
1.0 MIL F
INSERT TYPE

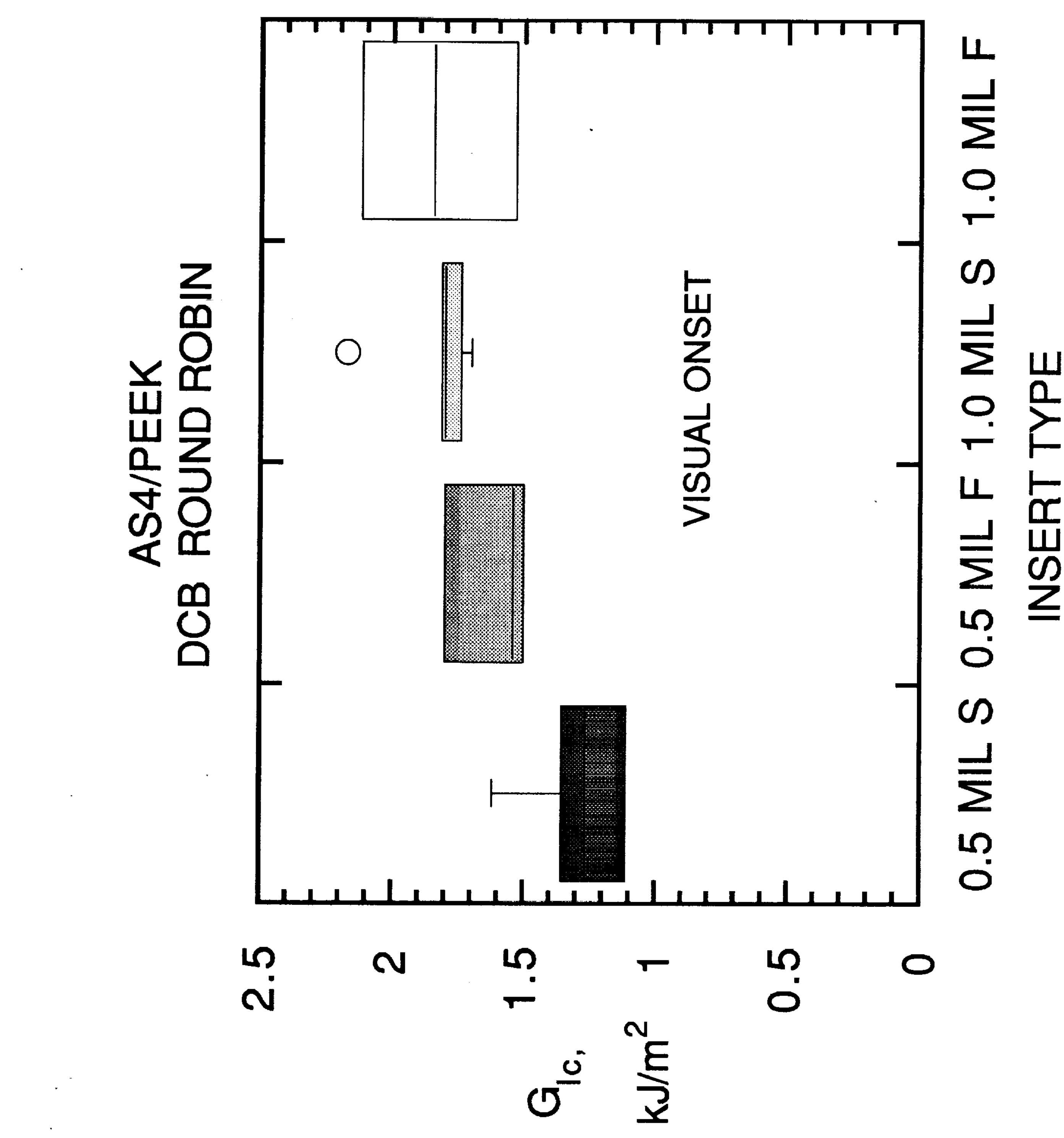

AS4/3501-6
DCB ROUND ROBIN
Mean
G_{Ic}'
kJ/m^2
0.5
0.4
0.3
0.2
0.1
0
0.5 MIL S
0.5 MIL F
1.0 MIL S
1.0 MIL F
DREP
NIST
NASA

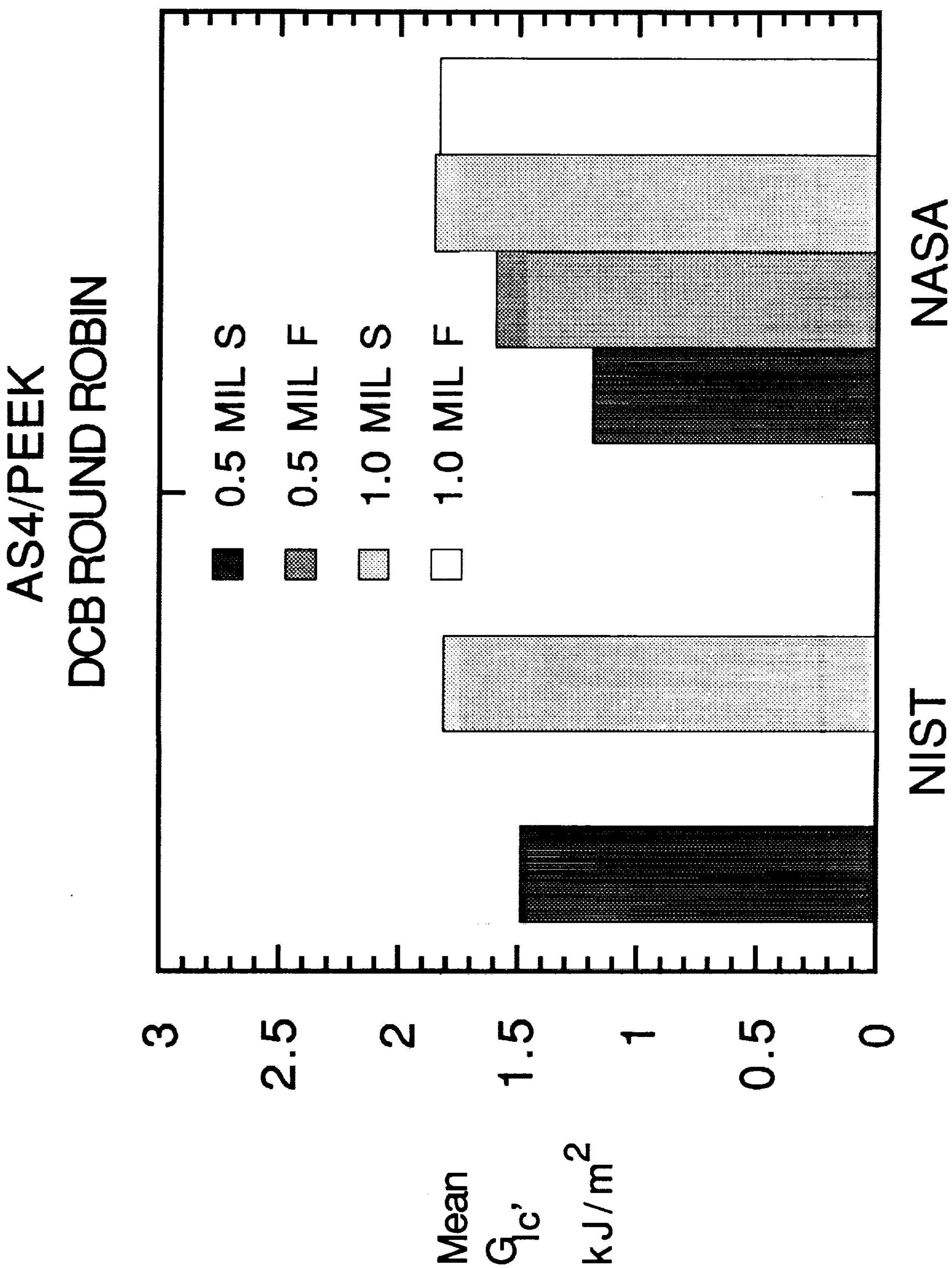

AS4/3501-6
DCB ROUND ROBIN
STANDARD DEVIATION
0.1
0.08
0.06
0.04
0.02
0
0.5 MIL S
0.5 MIL F
1.0 MIL S
1.0 MIL F
DREP
NIST
NASA


AS4/3501-6
DCB ROUND ROBIN
0.5 MIL SPRAYED KAPTON INSERT
G_{Ic}, kJ/m^2
0.2
0.16
0.12
0.08
0.04
0
DREP
NIST
NASA



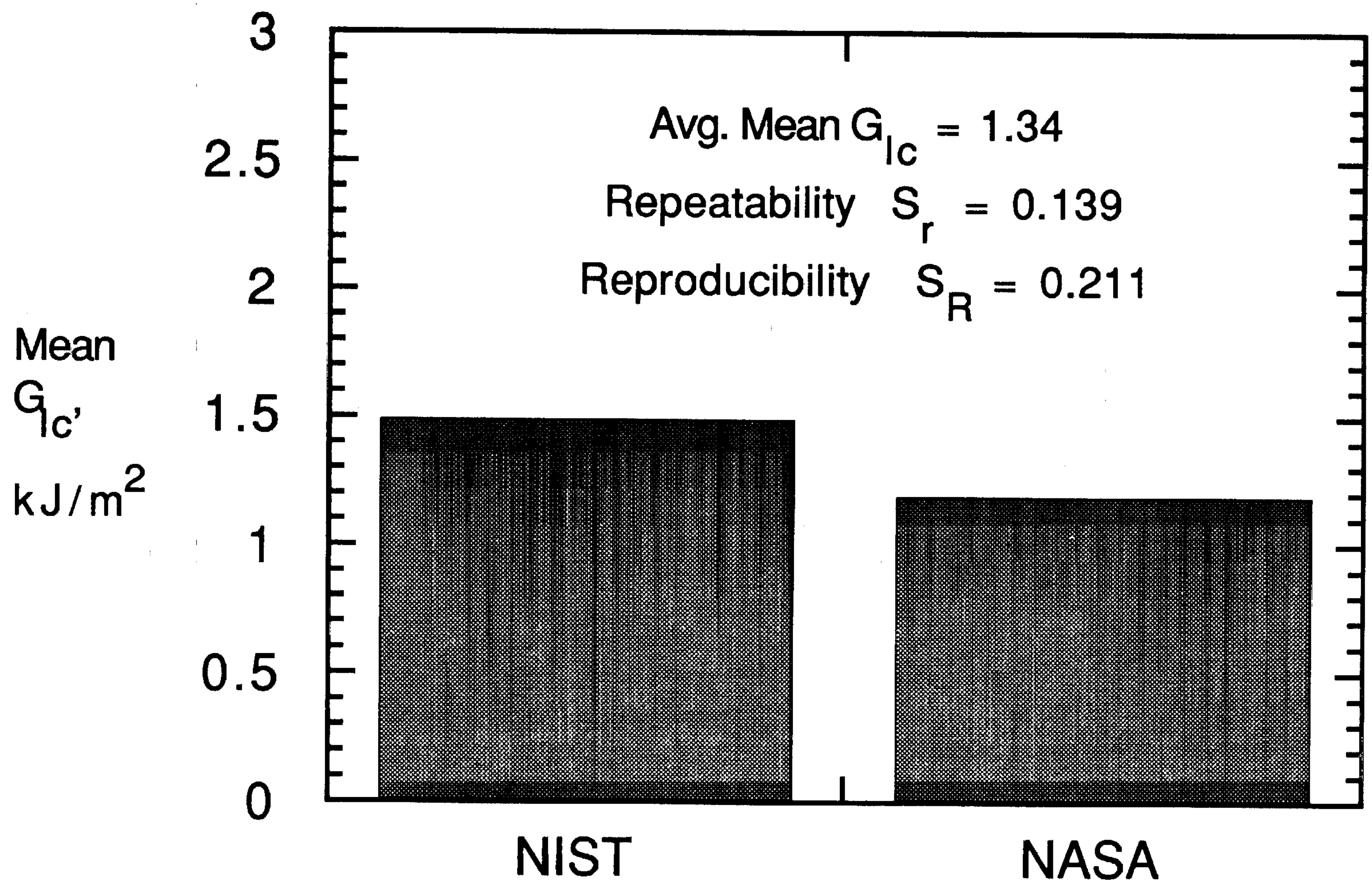
AS4/3501-6
DCB ROUND ROBIN
13 Micron (0.5 mil)
Sprayed Kapton Insert
Repeatability S_r = 0.015
Reproducibility S_R = 0.014
Avg. Mean G_{Ic} = 0.085
Mean G_{Ic}, kJ/m^2
0.5
0.4
0.3
0.2
0.1
0
DREP
NIST
NASA


AS4/PEEK
DCB ROUND ROBIN
VISUAL ONSET
G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
2.5
0.5 MIL S
0.5 MIL F
1.0 MIL F
1.0 MIL S
1.0 MIL F
INSERT TYPE


AS4/PEEK
DCB ROUND ROBIN
0.5 MIL S
0.5 MIL F
1.0 MIL S
1.0 MIL F
NIST
NASA
Mean
G_{Ic}'
kJ/m^2
3
2.5
2
1.5
1
0.5
0

AS4/PEEK DCB ROUND ROBIN

0.5 MIL SPRAYED KAPTON INSERT



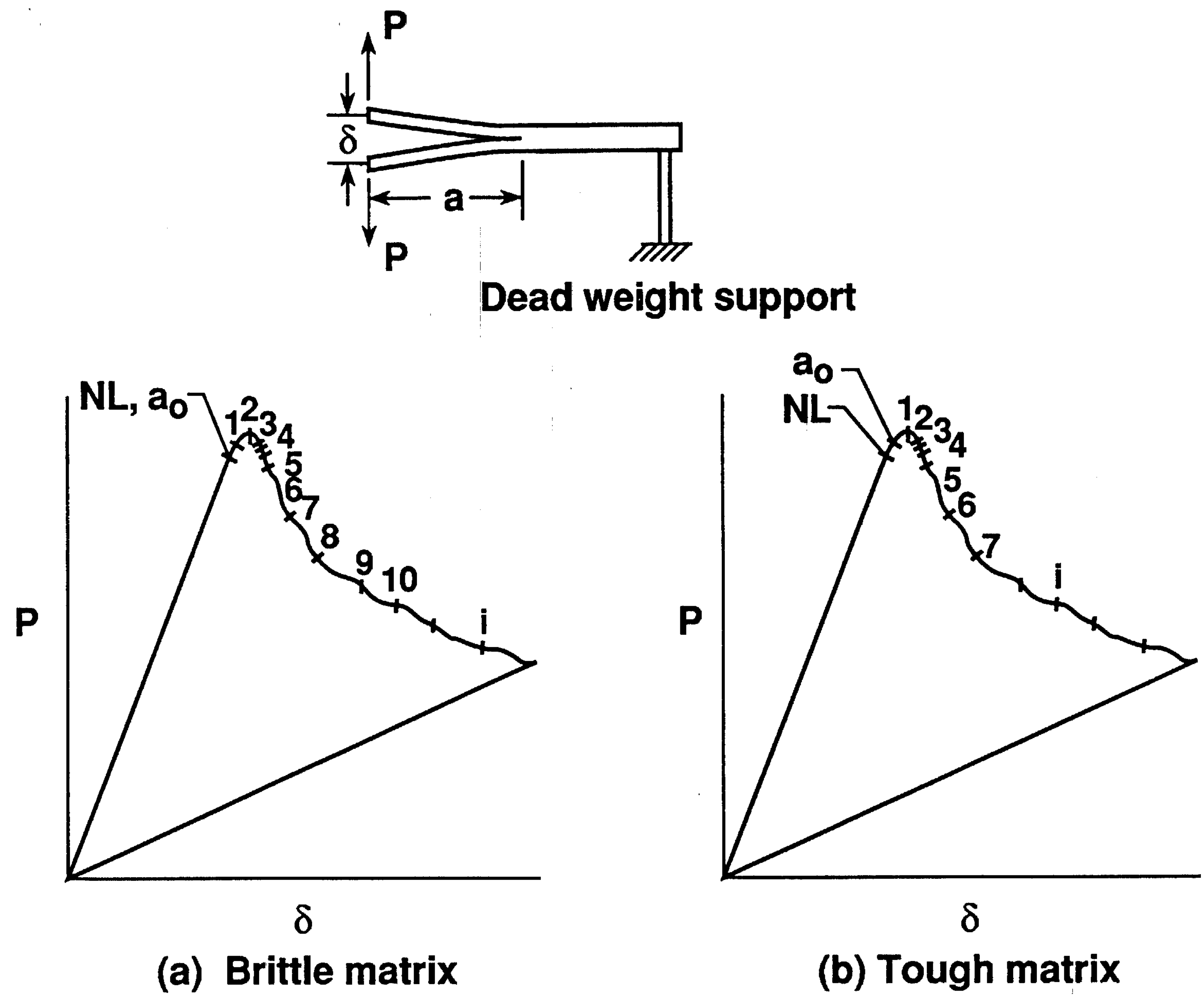
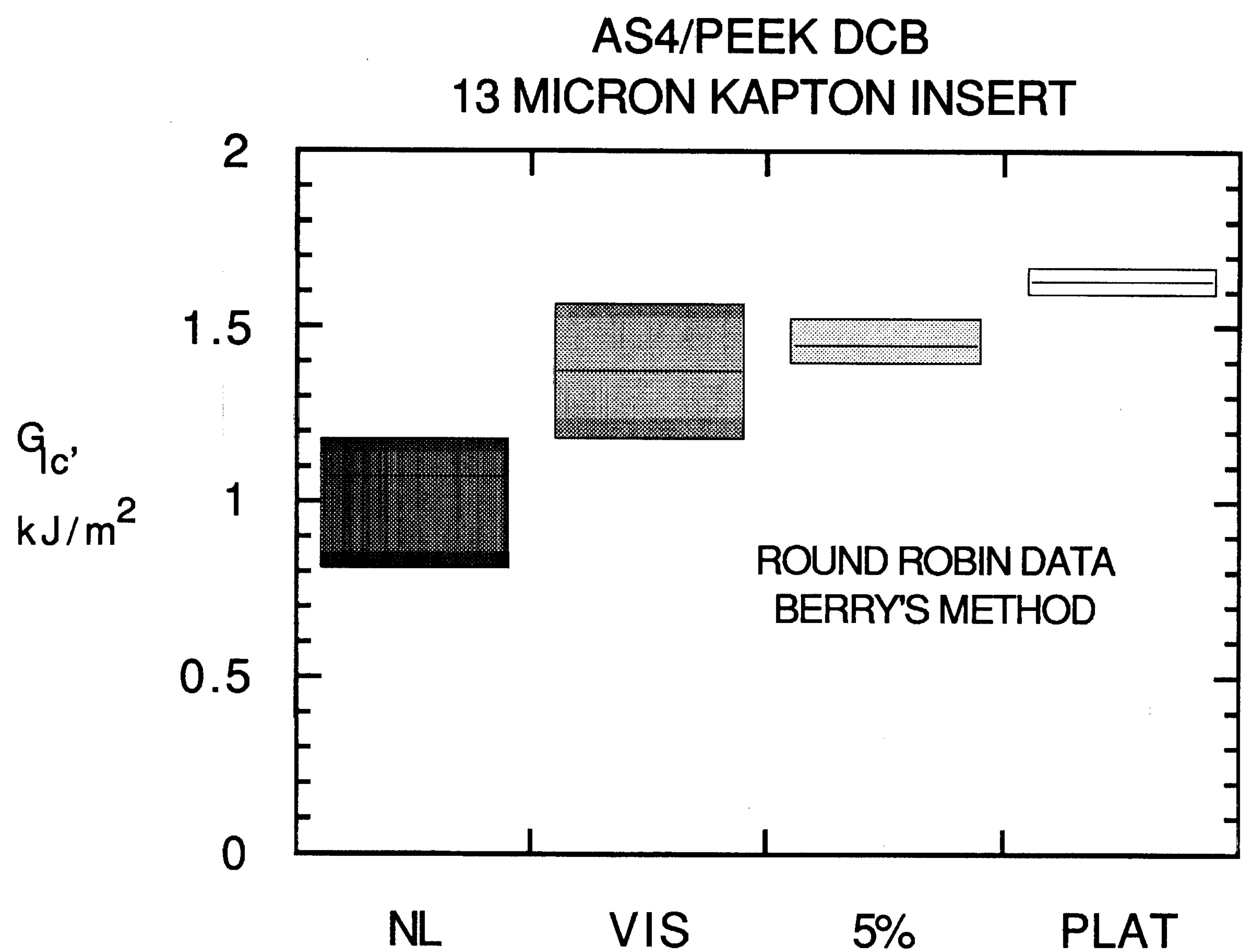
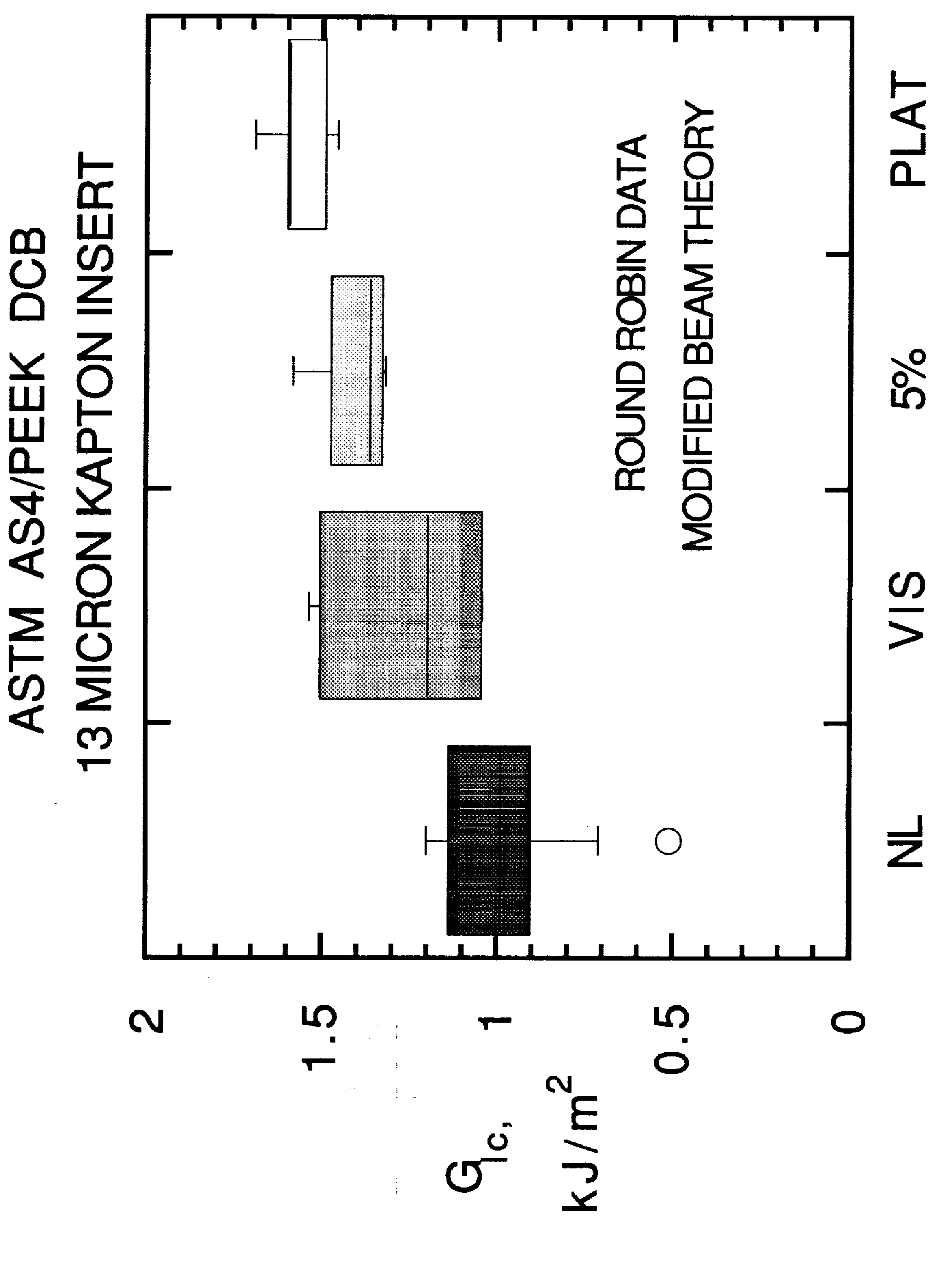
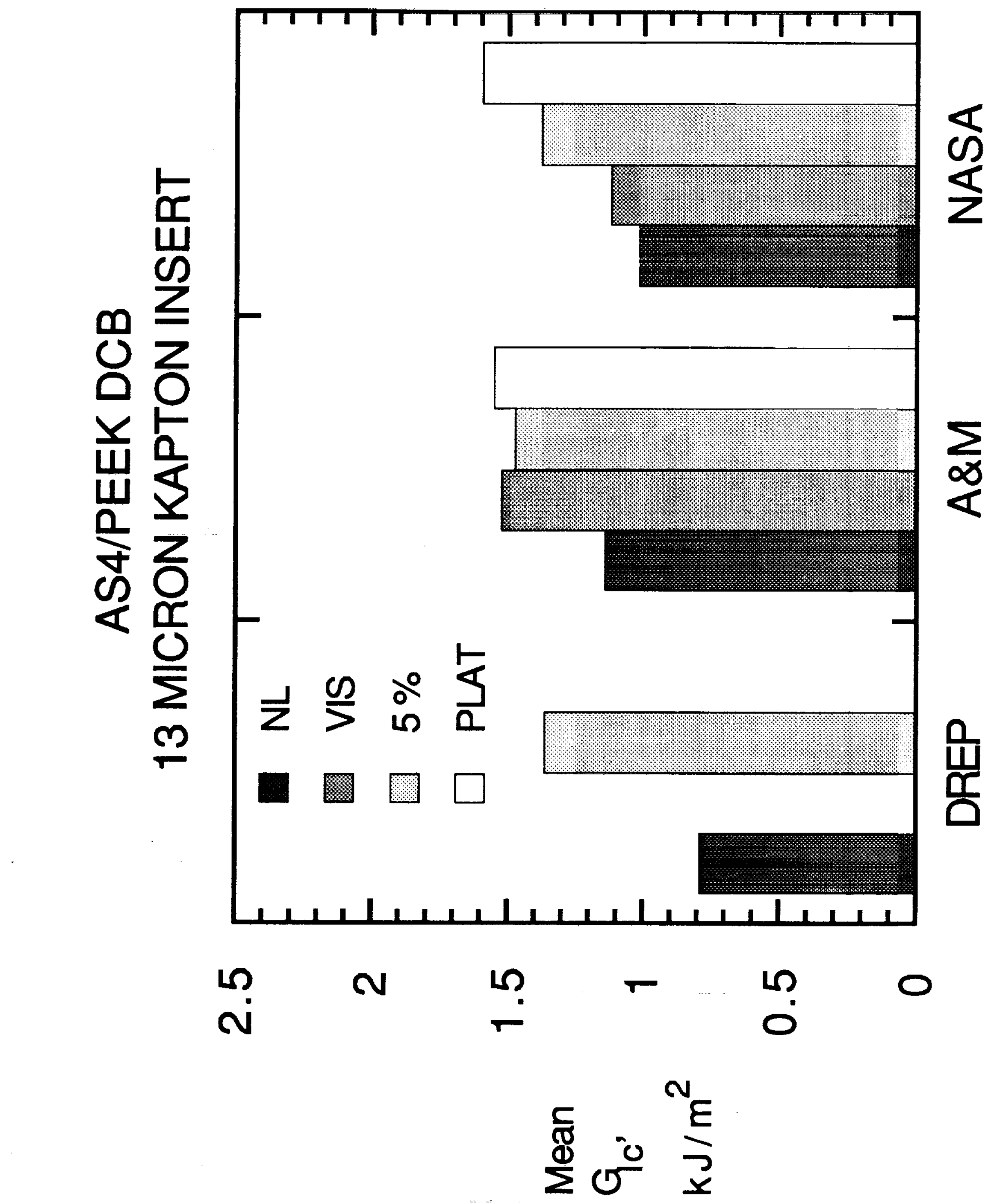


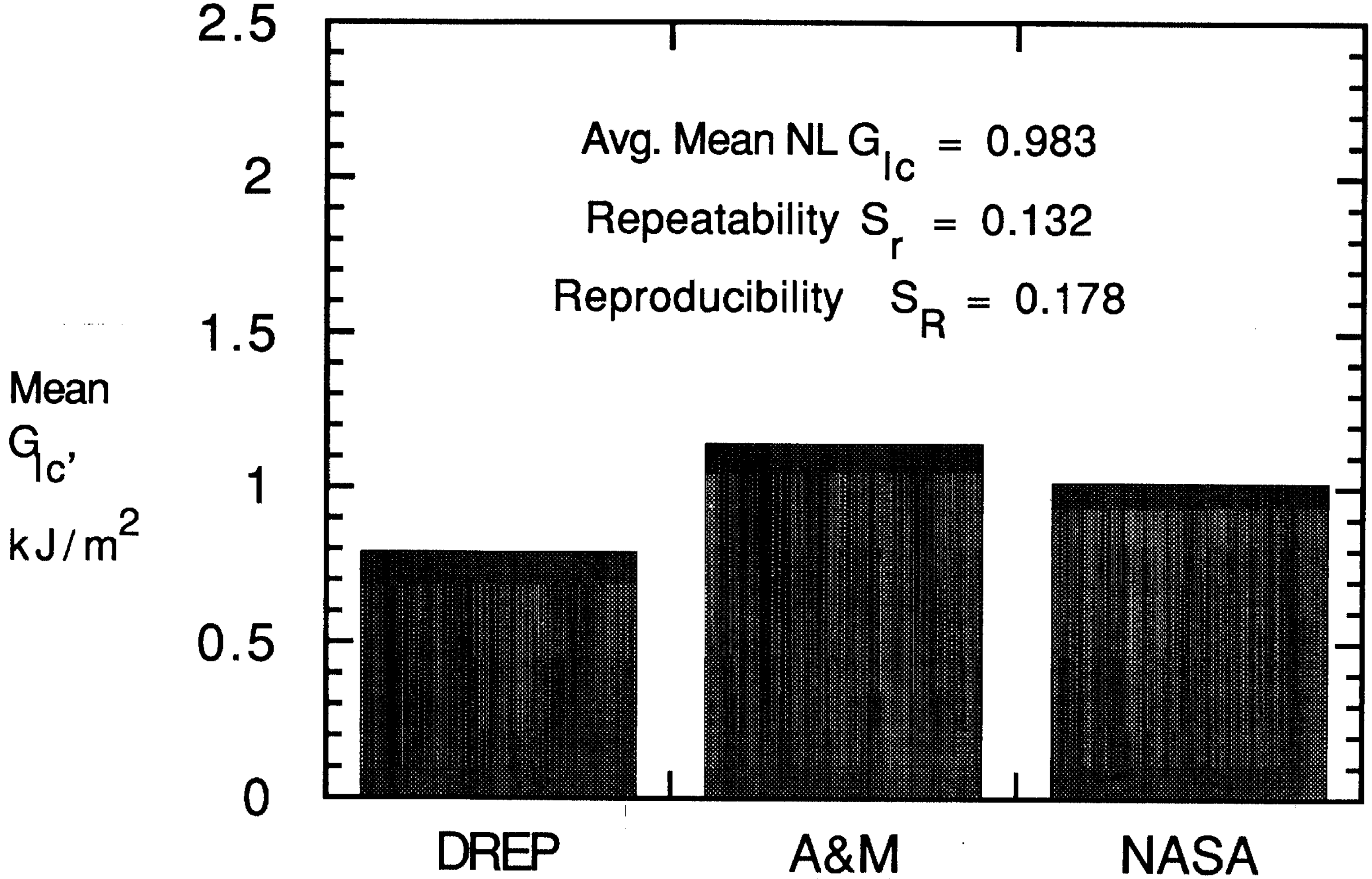
Figure 18. - Load displacement trace from DCB test

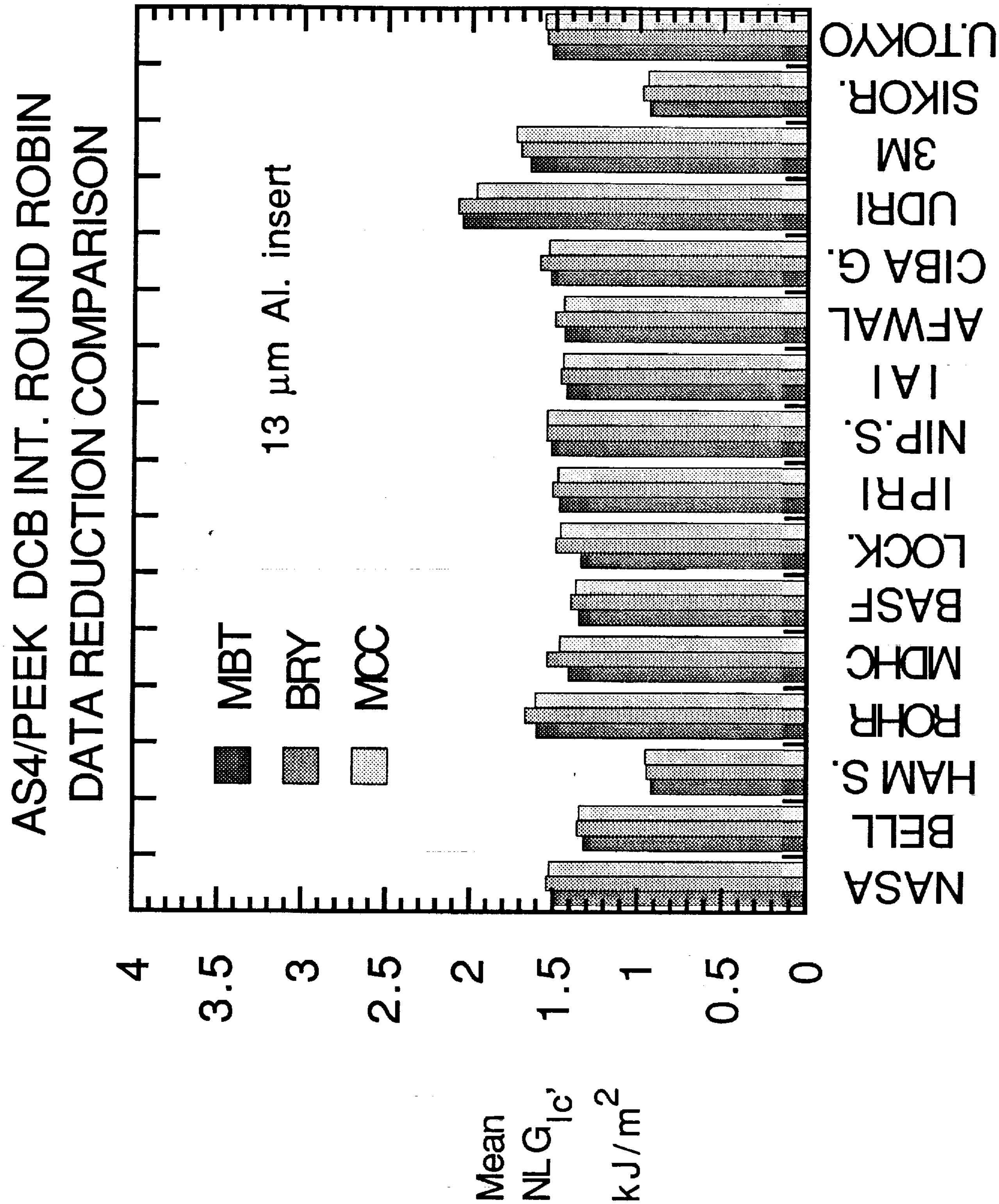

AS4/PEEK DCB
13 MICRON KAPTON INSERT
G_Ic'
kJ/m^2
2
1.5
1
0.5
0
ROUND ROBIN DATA
BERRY'S METHOD
NL
VIS
5%
PLAT


ASTM AS4/PEEK DCB
13 MICRON KAPTON INSERT
ROUND ROBIN DATA
MODIFIED BEAM THEORY
G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
NL
VIS
5%
PLAT

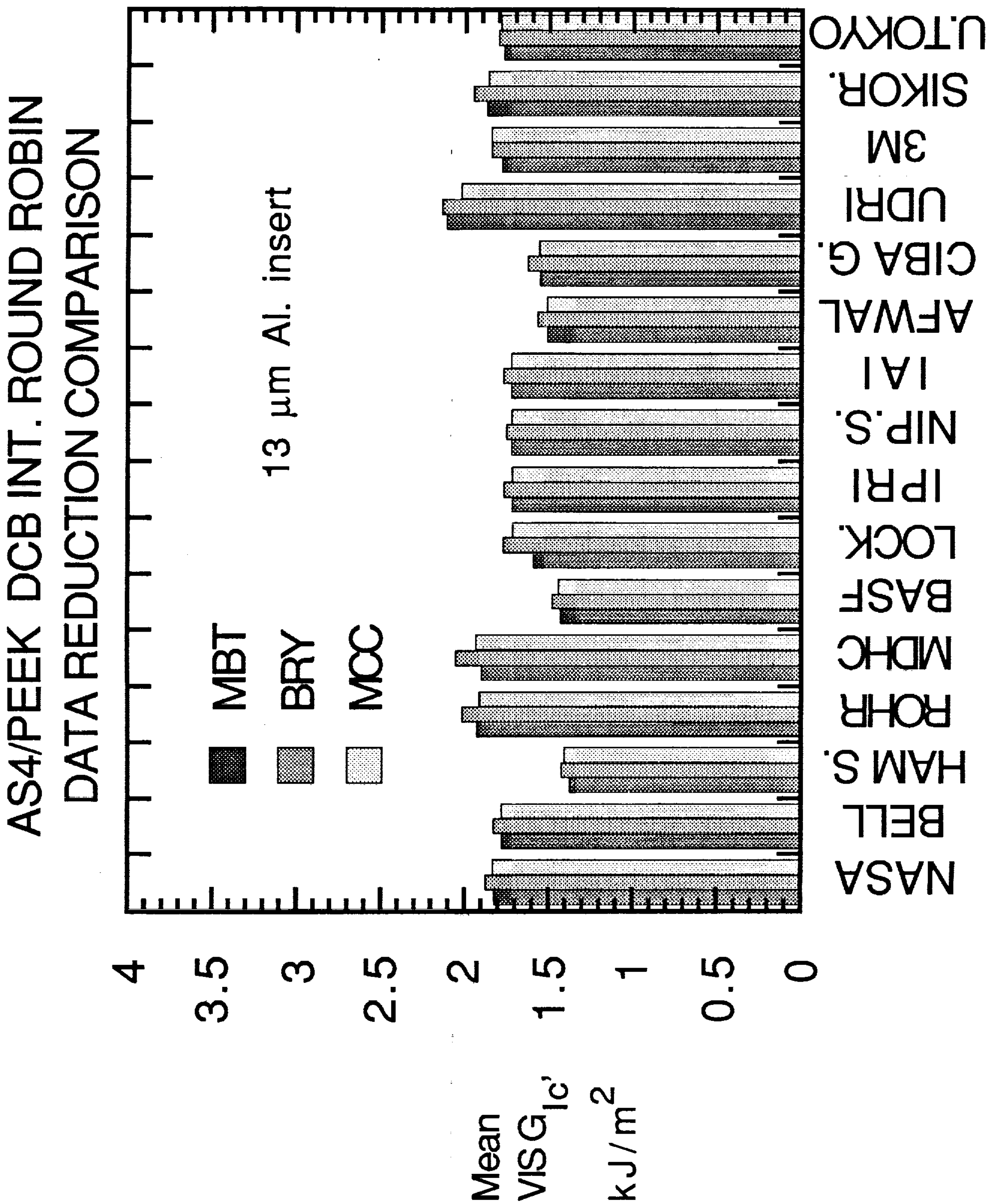

AS4/PEEK DCB
13 MICRON KAPTON INSERT
NL
VIS
5 %
PLAT
Mean
G_Ic'
kJ/m^2
2.5
2
1.5
1
0.5
0
DREP
A&M
NASA

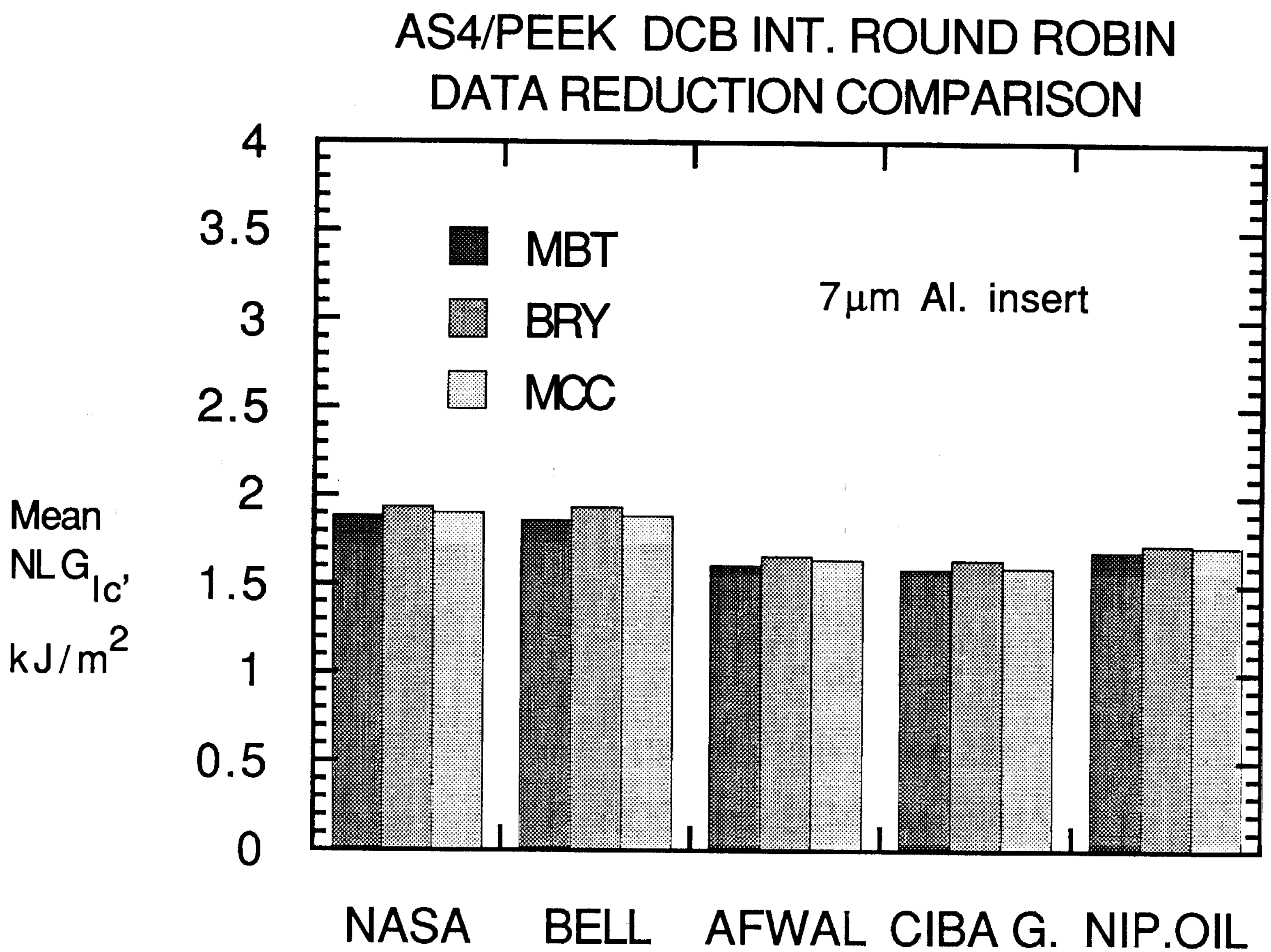
AS4/PEEK DCB
13 MICRON KAPTON INSERT

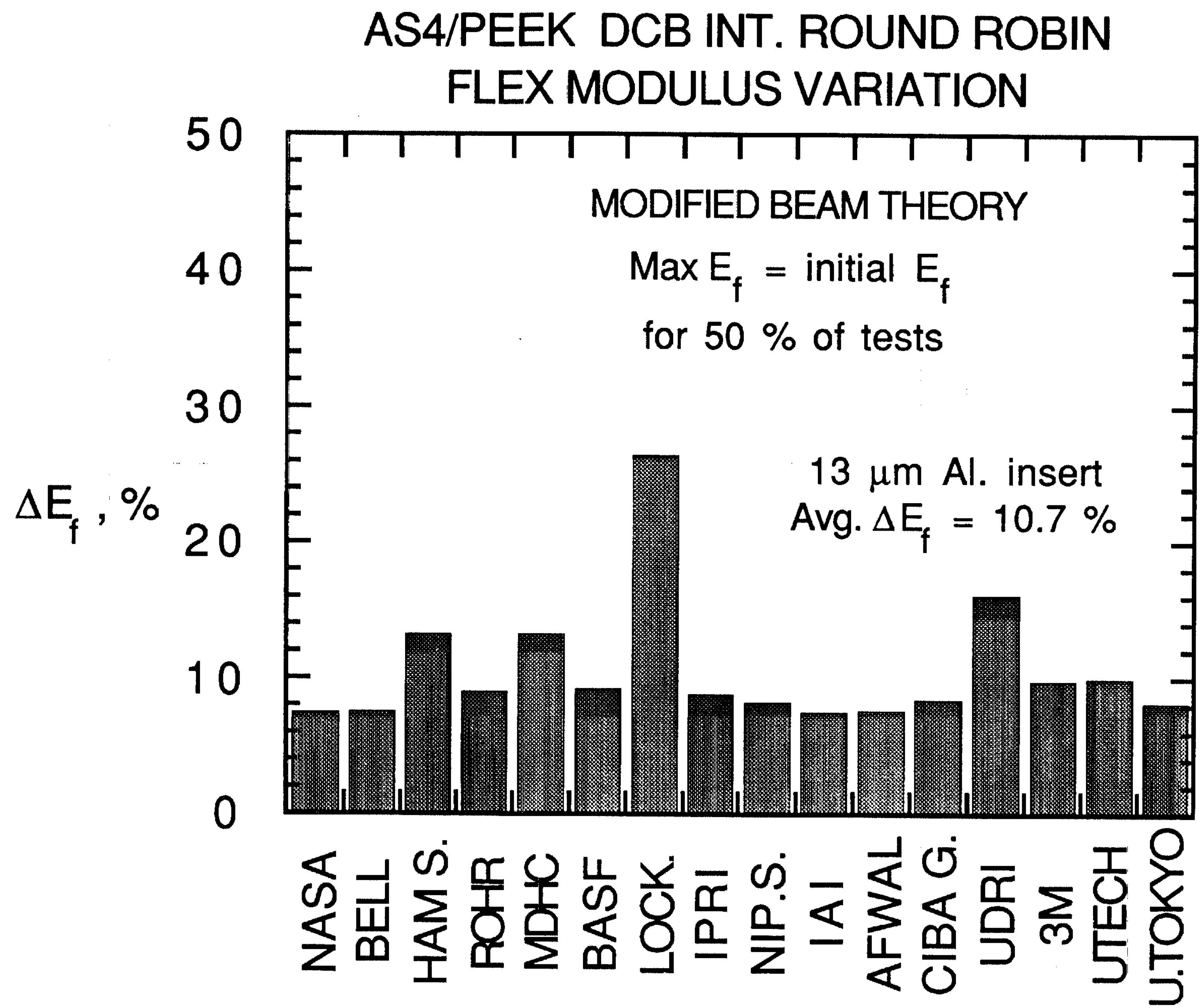


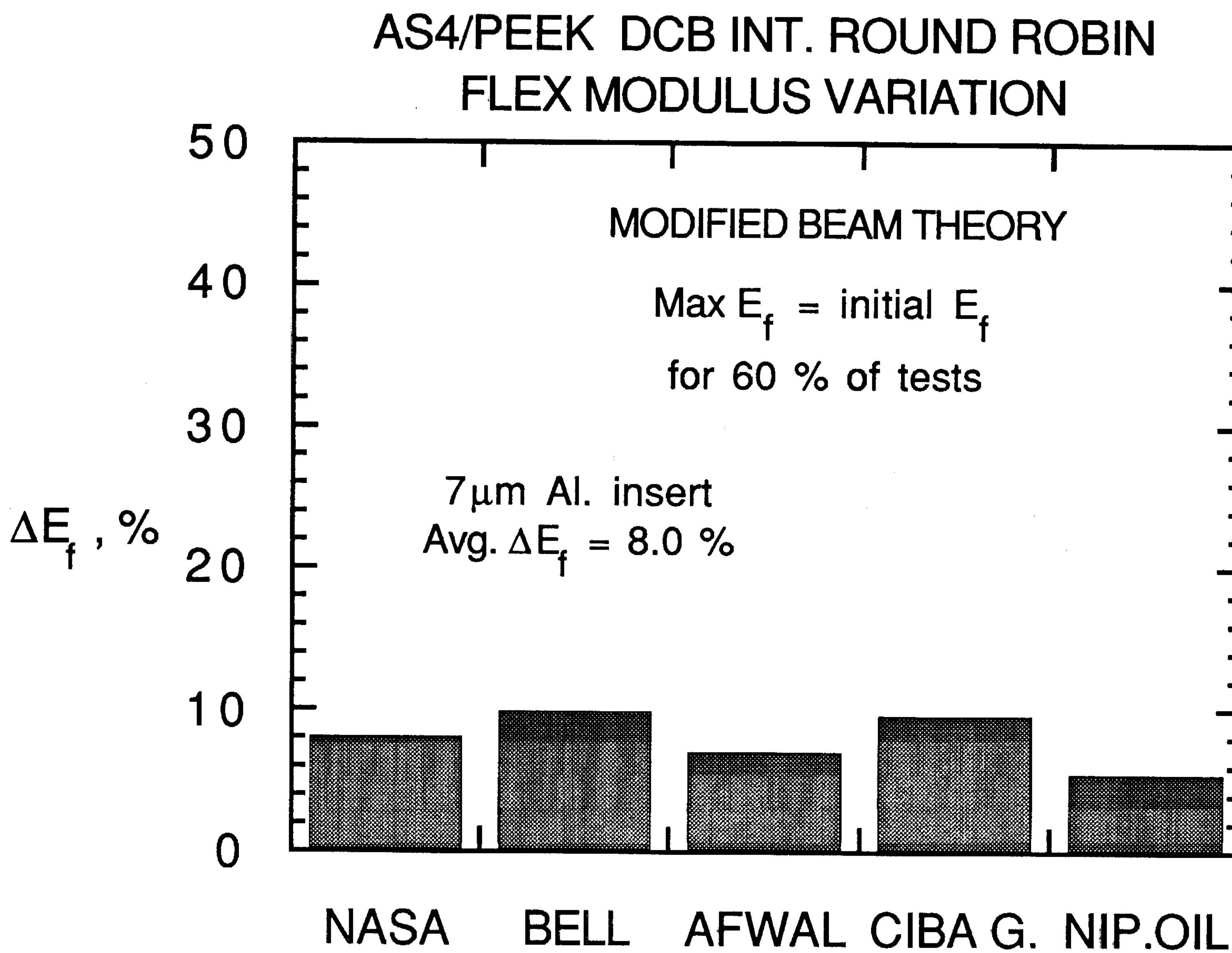


AS4/PEEK DCB INT. ROUND ROBIN
DATA REDUCTION COMPARISON
13 μm Al. insert
MBT
BRY
MCC
Mean
NL G$_{Ic}$,
kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
IAI
AFWAL
CIBA G.
UDRI
3M
SIKOR.
U.TOKYO


AS4/PEEK DCB INT. ROUND ROBIN
DATA REDUCTION COMPARISON
MBT
BRY
MCC
13 µm Al. insert
Mean
VIS G_{Ic}'
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
SIKOR.
U.TOKYO

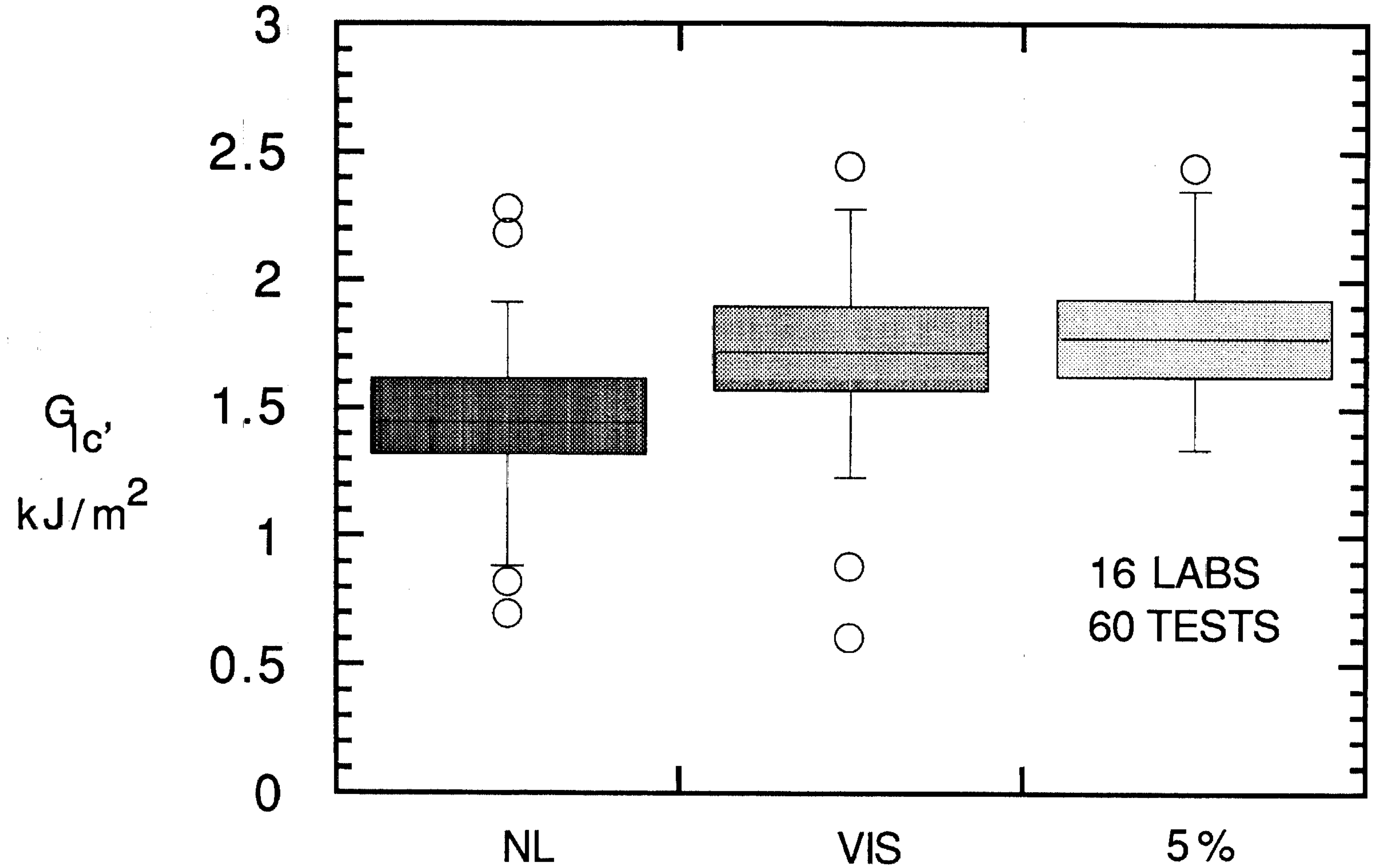

AS4/PEEK DCB INT. ROUND ROBIN
DATA REDUCTION COMPARISON
Mean
NL G_{Ic},
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
MBT
BRY
MCC
7μm Al. insert
NASA
BELL
AFWAL
CIBA G.
NIP.OIL

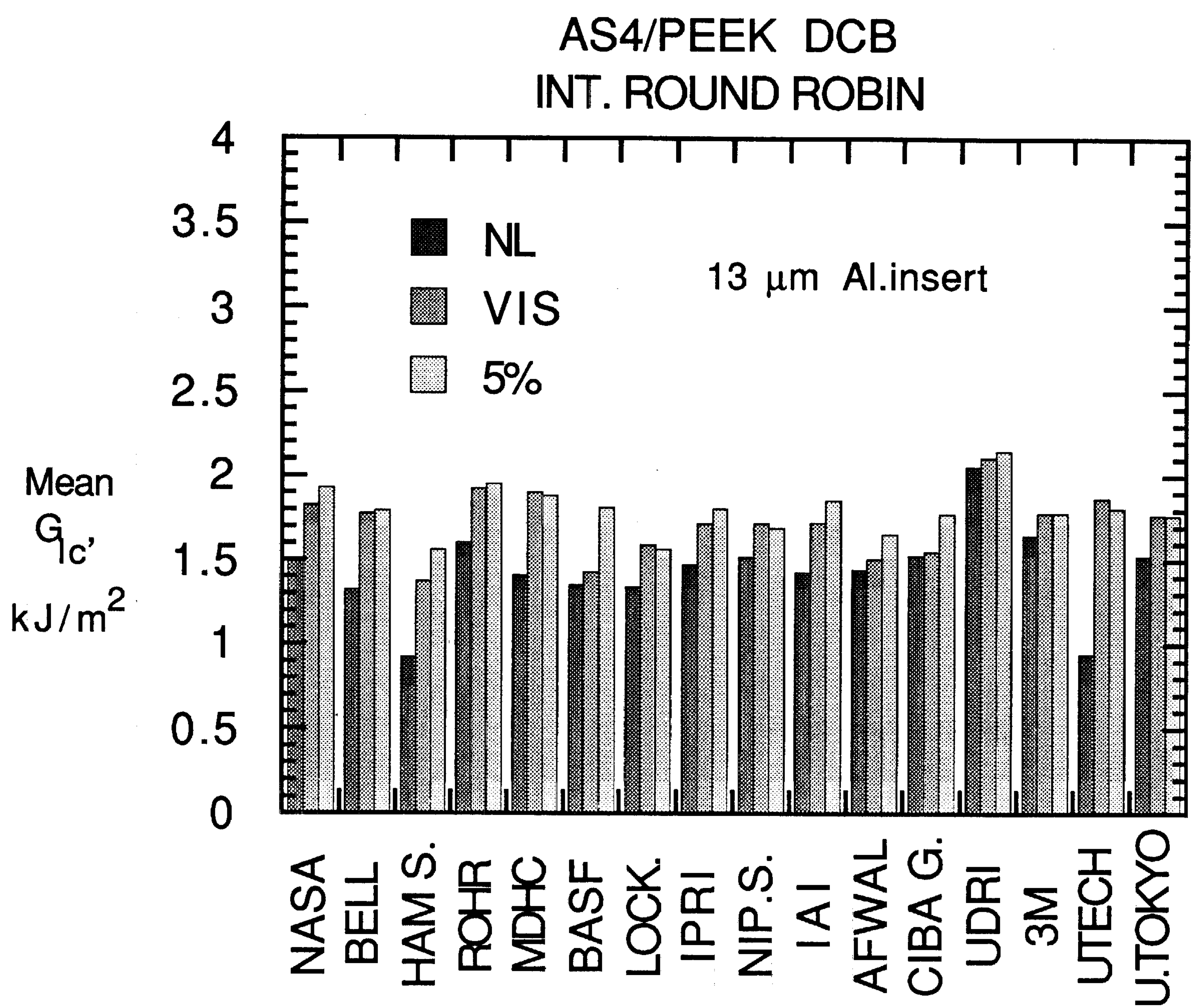

AS4/PEEK DCB INT. ROUND ROBIN
FLEX MODULUS VARIATION
MODIFIED BEAM THEORY
Max E_f = initial E_f
for 50 % of tests
13 μm Al. insert
Avg. ΔE_f = 10.7 %
ΔE_f, %
0
10
20
30
40
50
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO

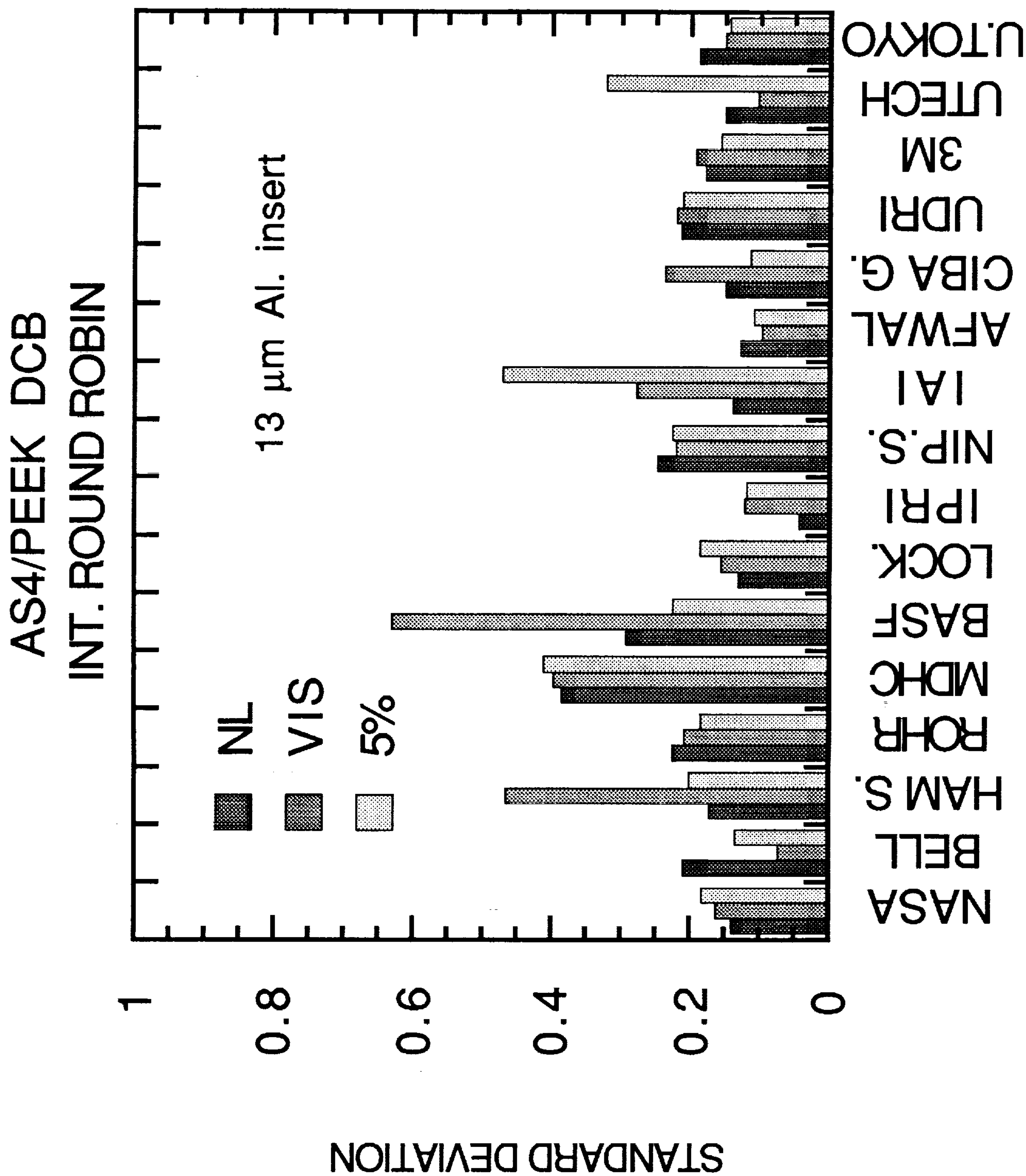


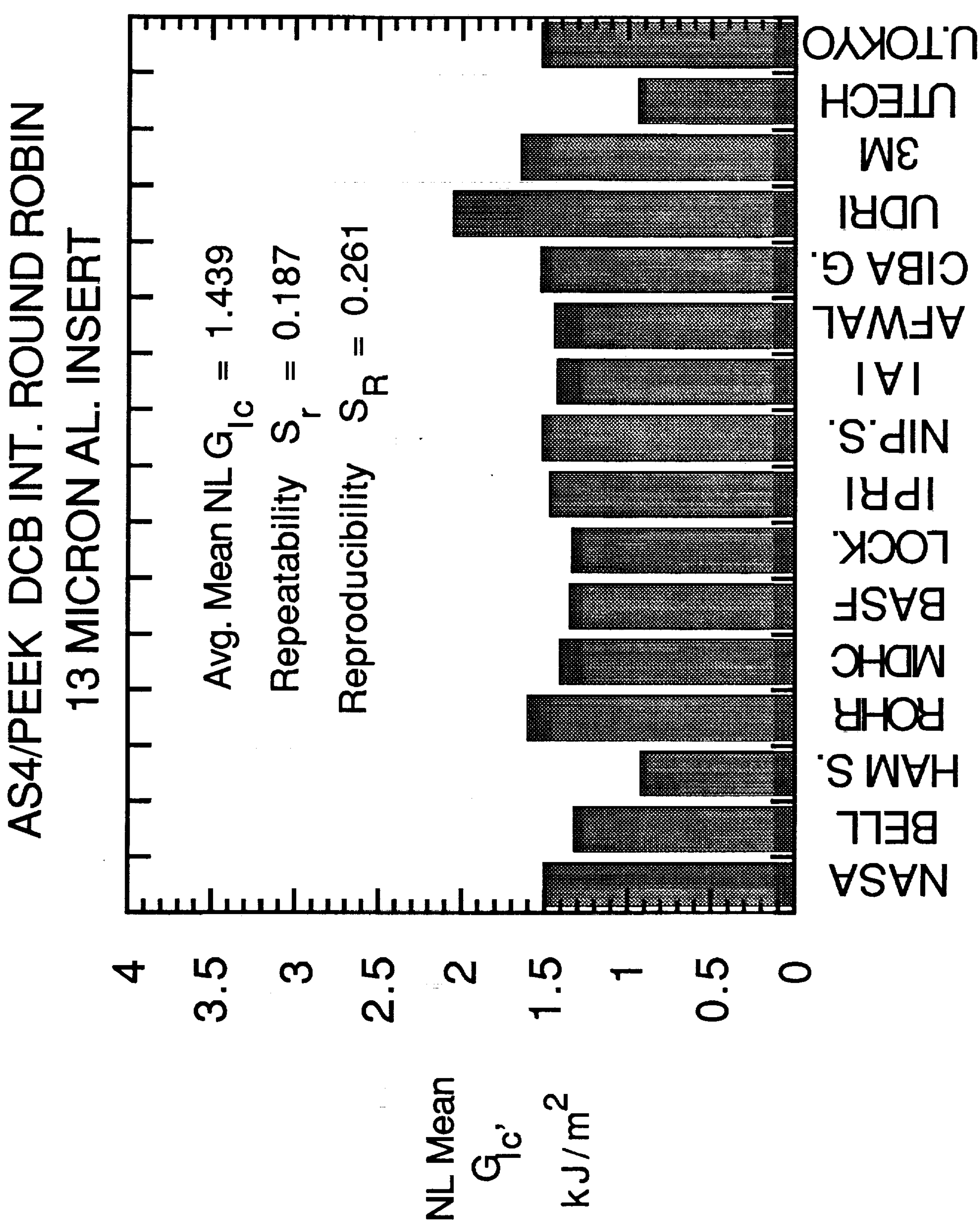
AS4/PEEK DCB INT. ROUND ROBIN
FLEX MODULUS VARIATION
MODIFIED BEAM THEORY
Max E_f = initial E_f
for 60 % of tests
7 µm Al. insert
Avg. ΔE_f = 8.0 %
ΔE_f , %
50
40
30
20
10
0
NASA
BELL
AFWAL
CIBA G.
NIP.OIL

AS4/PEEK DCB INT. ROUND ROBIN
13 MICRON AL. INSERT

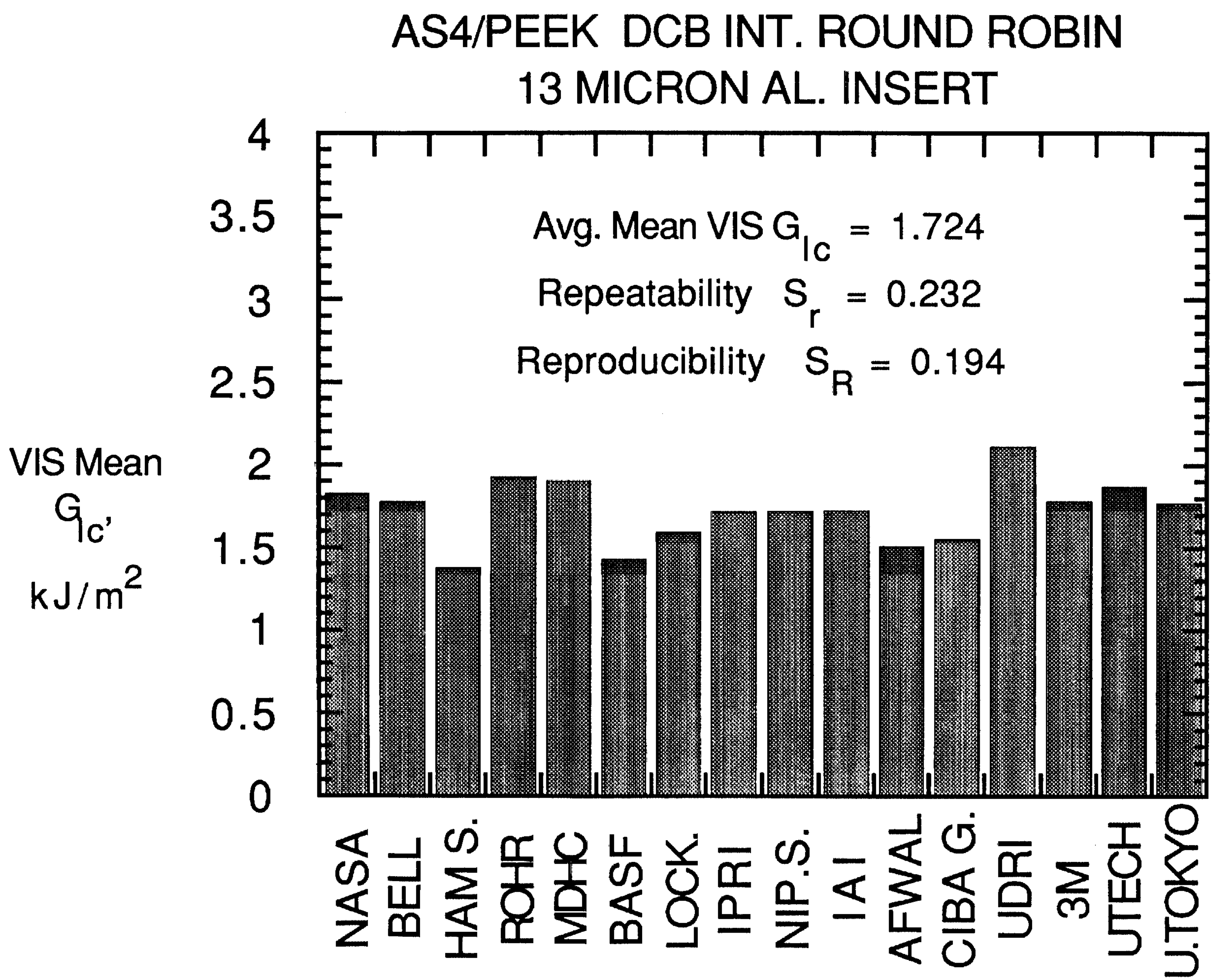



AS4/PEEK DCB
INT. ROUND ROBIN
Mean
G_{Ic},
kJ/m^2
NL
VIS
5%
13 µm Al.insert
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO

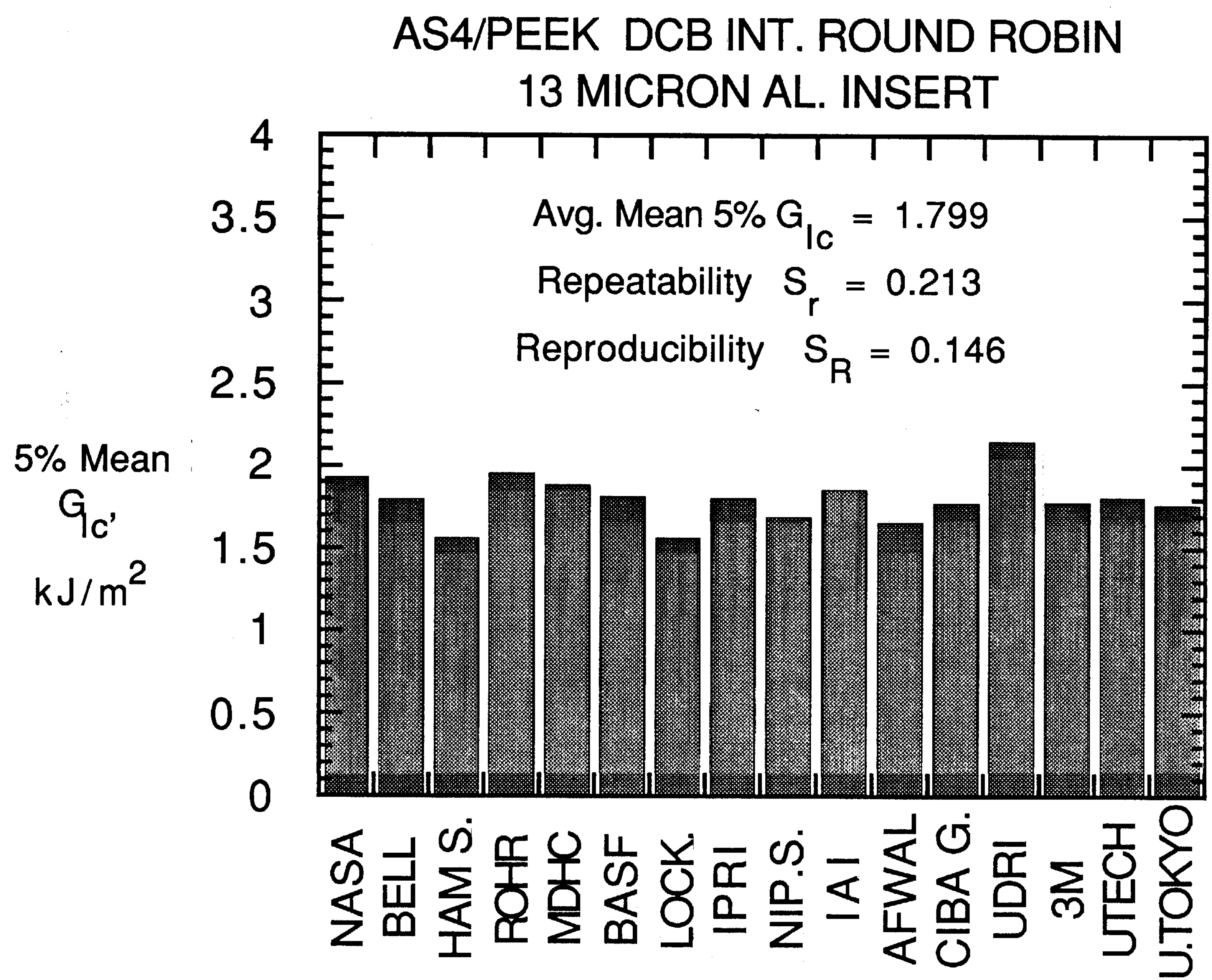

AS4/PEEK DCB
INT. ROUND ROBIN
13 µm Al. insert
NL
VIS
5%
STANDARD DEVIATION
0
0.2
0.4
0.6
0.8
1
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
IAI
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO

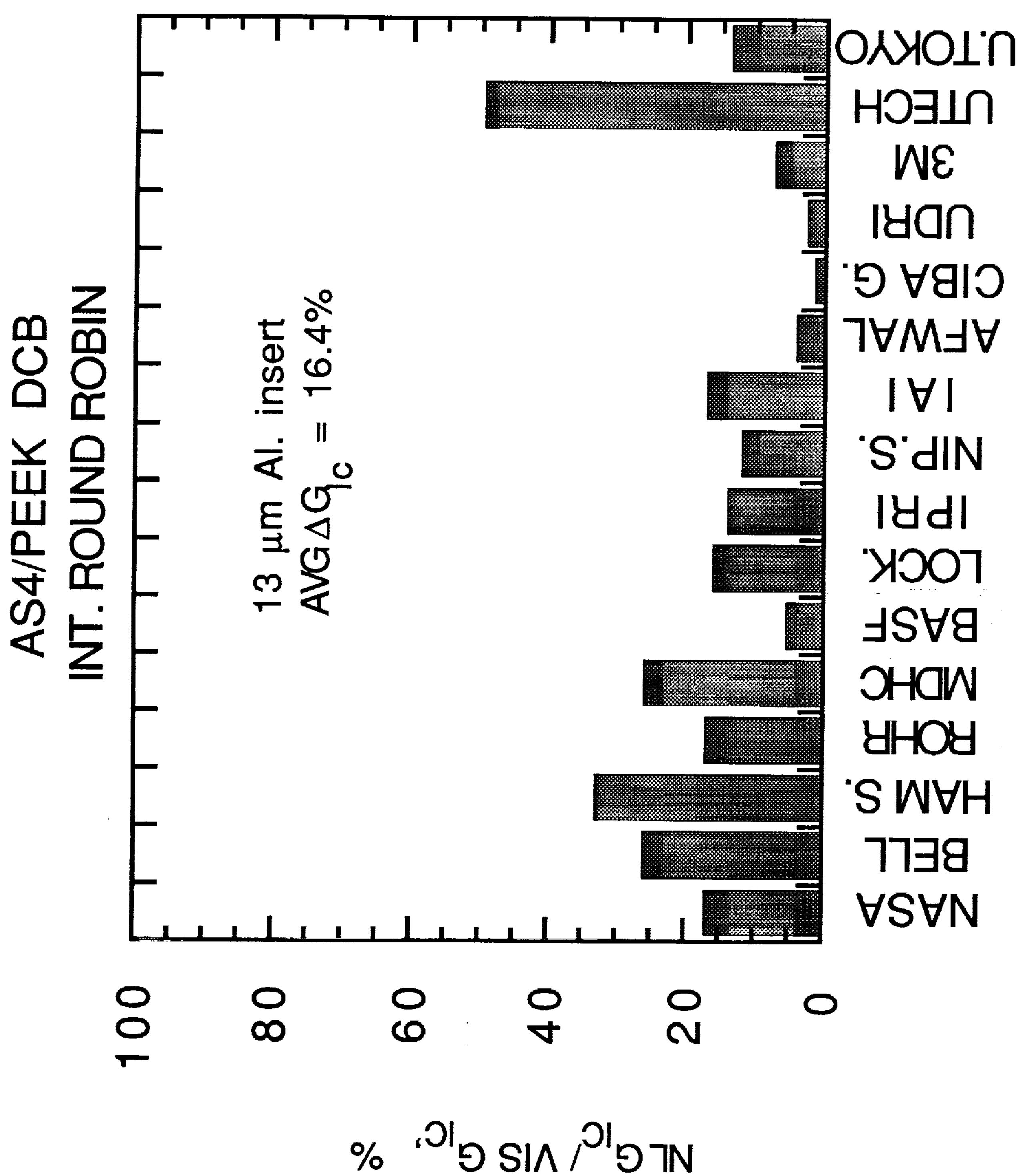


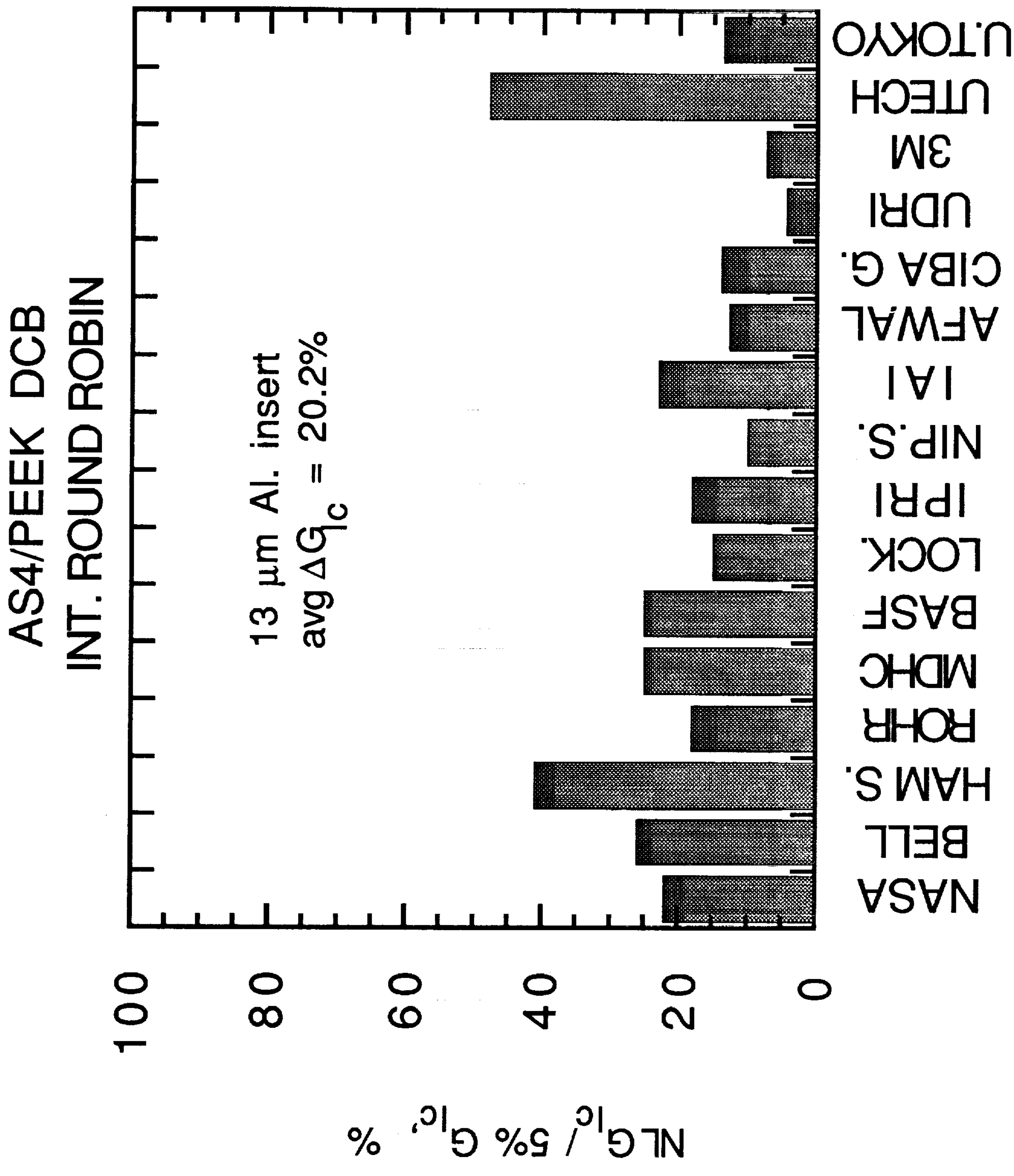
AS4/PEEK DCB INT. ROUND ROBIN
13 MICRON AL. INSERT
Avg. Mean NL G_{Ic} = 1.439
Repeatability S_r = 0.187
Reproducibility S_R = 0.261
NL Mean
G_{Ic},
kJ / m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO



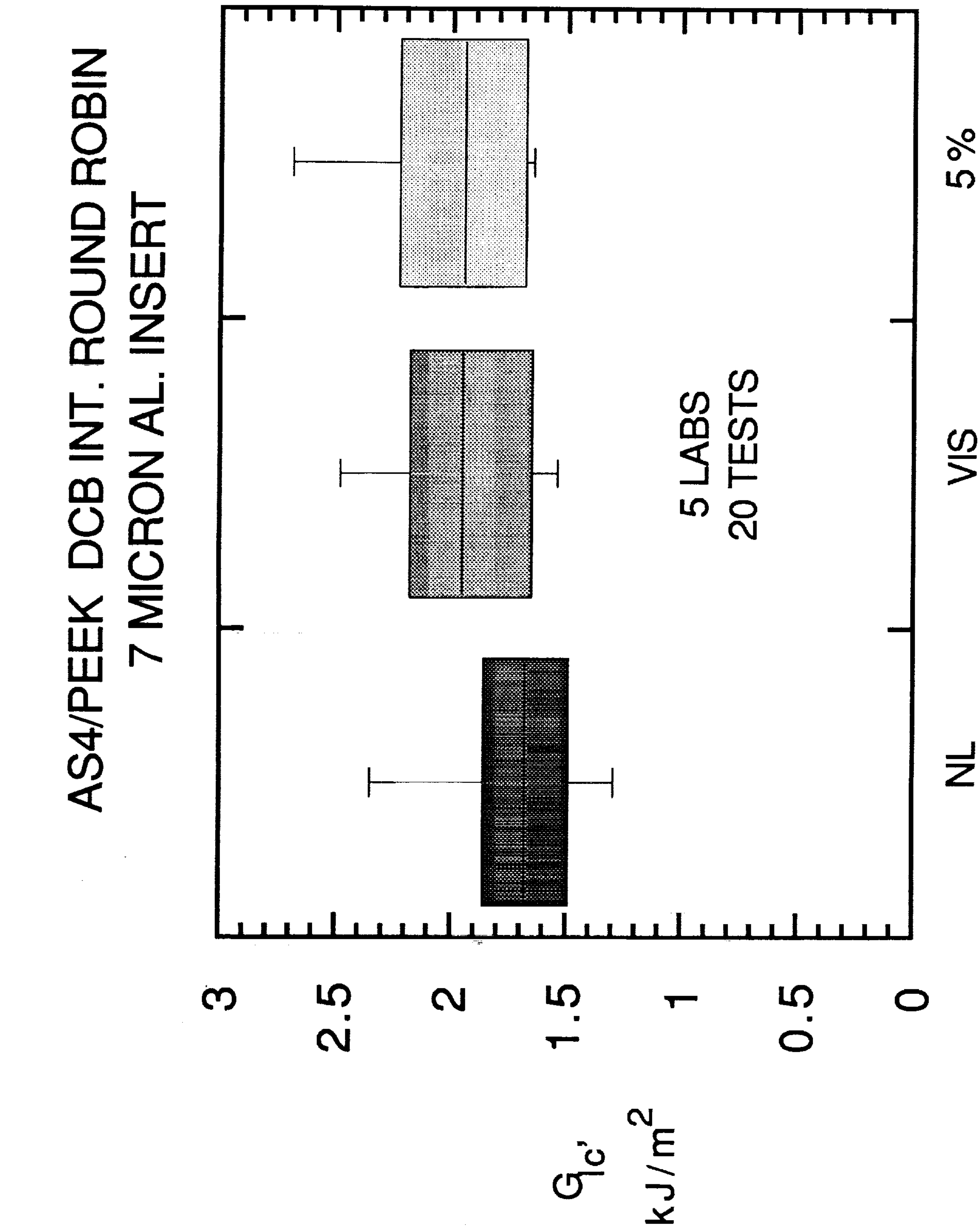
AS4/PEEK DCB INT. ROUND ROBIN
13 MICRON AL. INSERT
Avg. Mean VIS G_{Ic} = 1.724
Repeatability S_r = 0.232
Reproducibility S_R = 0.194
VIS Mean
G_{Ic},
kJ / m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO

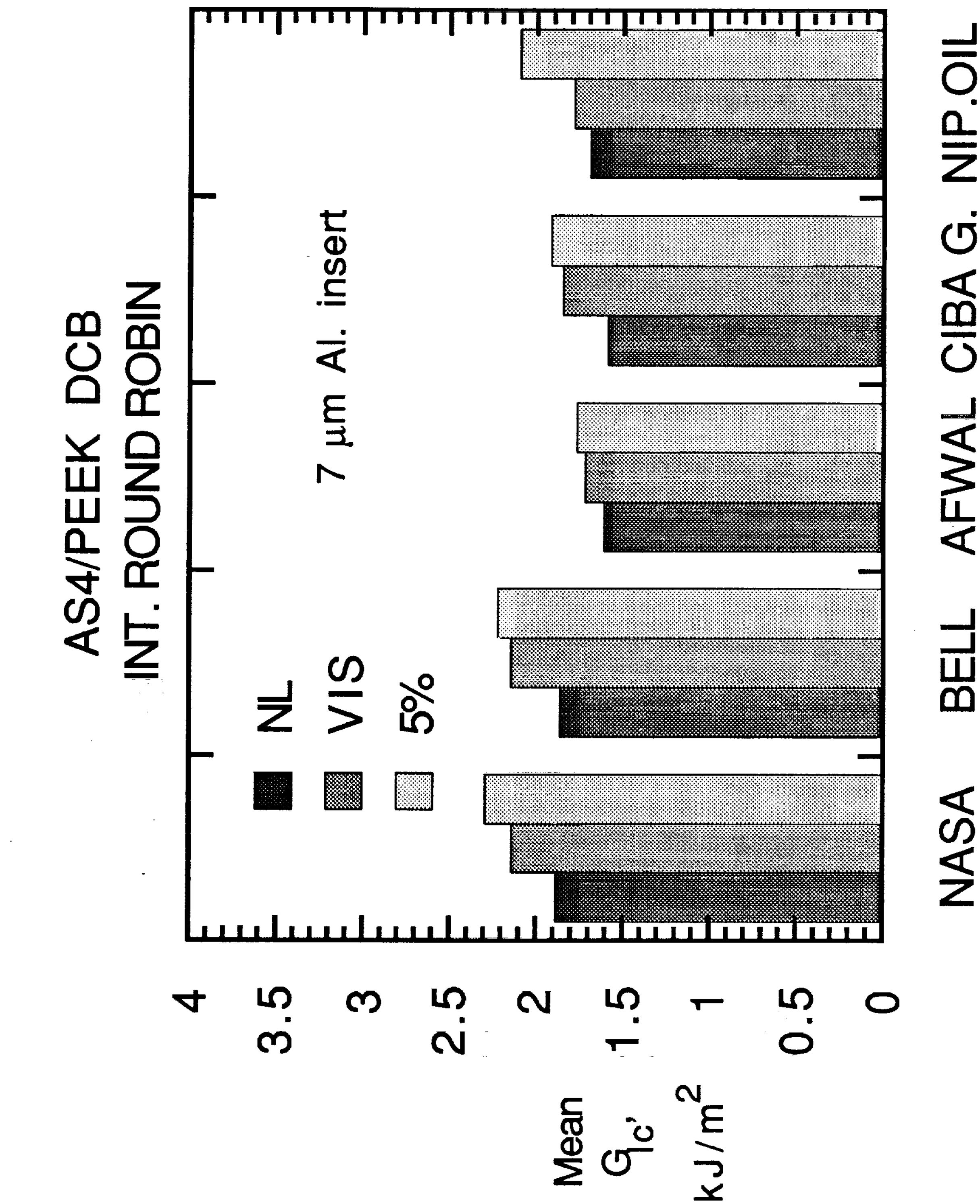

AS4/PEEK DCB INT. ROUND ROBIN
13 MICRON AL. INSERT
Avg. Mean 5% G_{Ic} = 1.799
Repeatability S_r = 0.213
Reproducibility S_R = 0.146
5% Mean G_{Ic}, kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO

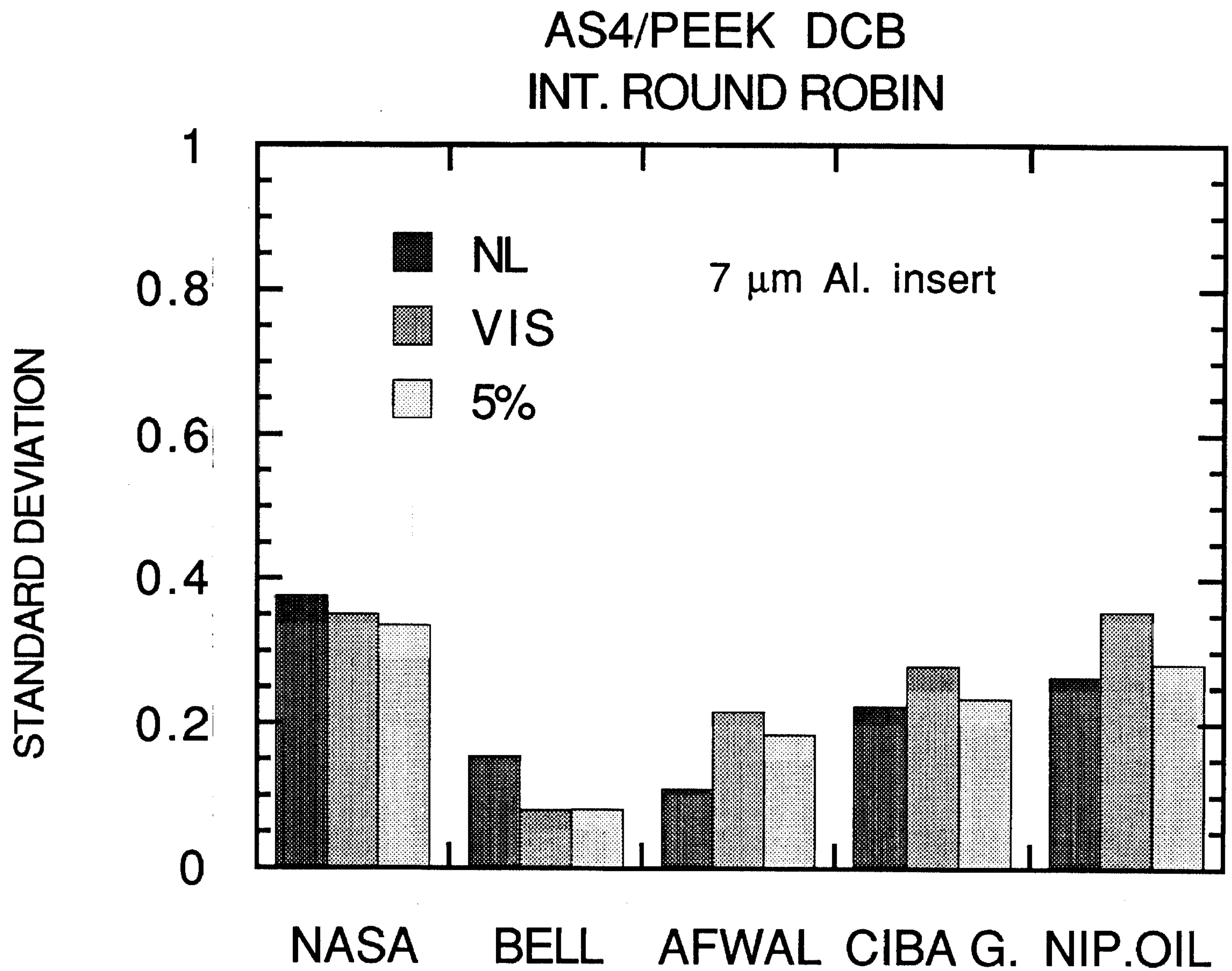

AS4/PEEK DCB
INT. ROUND ROBIN
13 µm Al. insert
AVG ΔG_Ic = 16.4%
NL G_IC/ VIS G_IC, %
100
80
60
40
20
0
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO

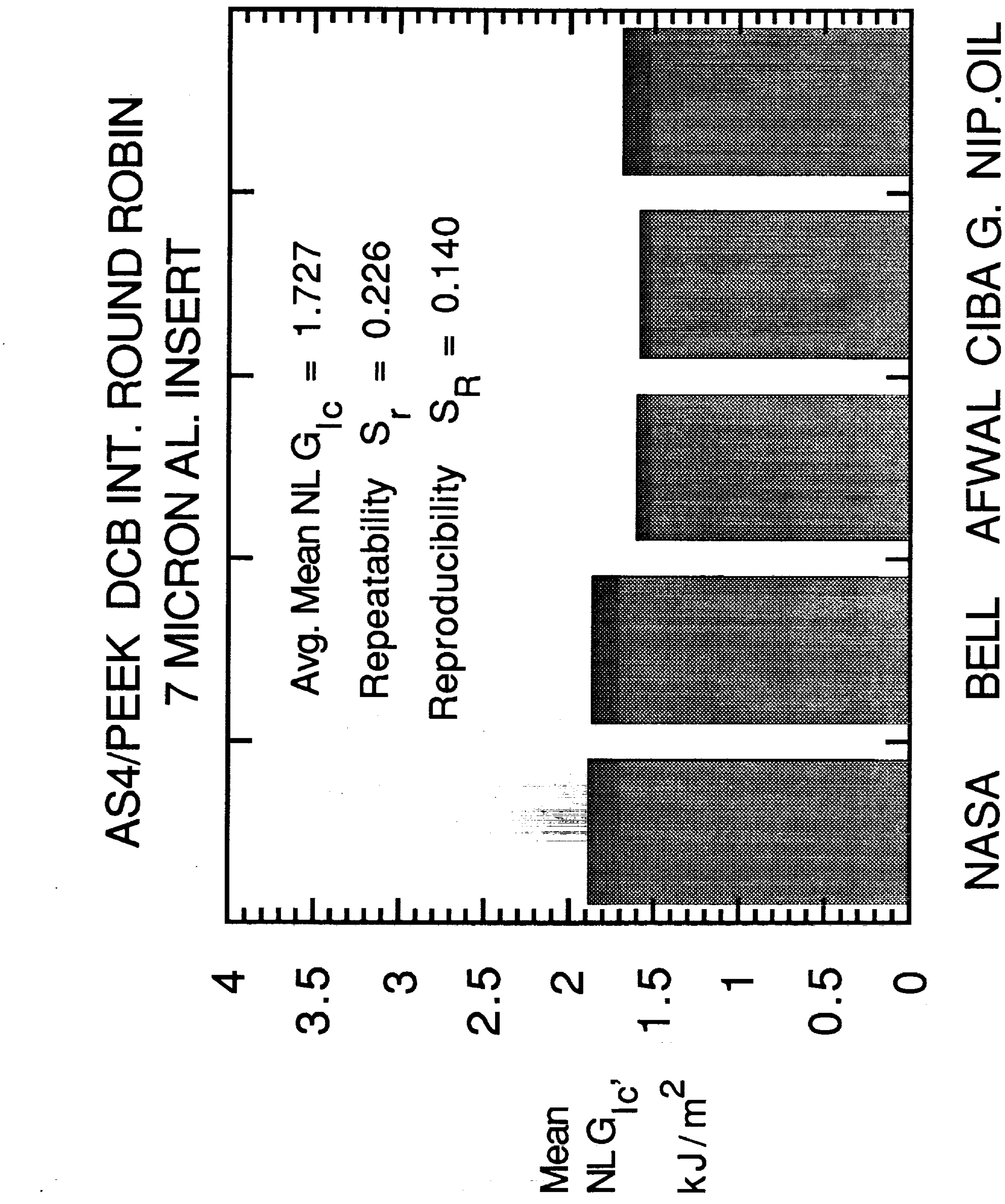


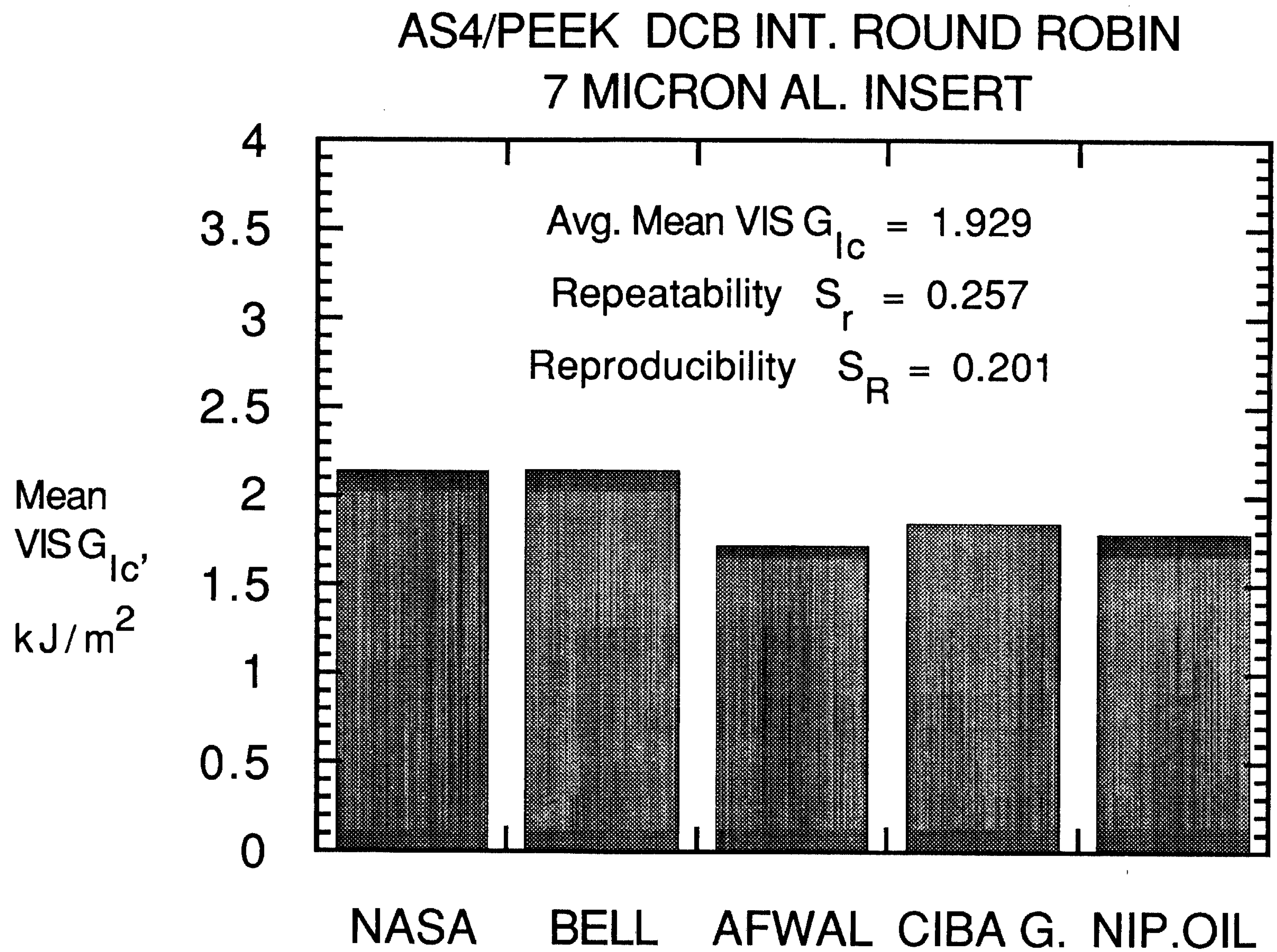
AS4/PEEK DCB
INT. ROUND ROBIN
13 µm Al. insert
avg ΔG_{Ic} = 20.2%
NL G_{Ic} / 5% G_{Ic}, %
0
20
40
60
80
100
NASA
BELL
HAM S.
ROHR
MDHC
BASF
LOCK.
IPRI
NIP.S.
I A I
AFWAL
CIBA G.
UDRI
3M
UTECH
U.TOKYO


AS4/PEEK DCB INT. ROUND ROBIN
7 MICRON AL. INSERT
G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
2.5
3
NL
VIS
5%
5 LABS
20 TESTS

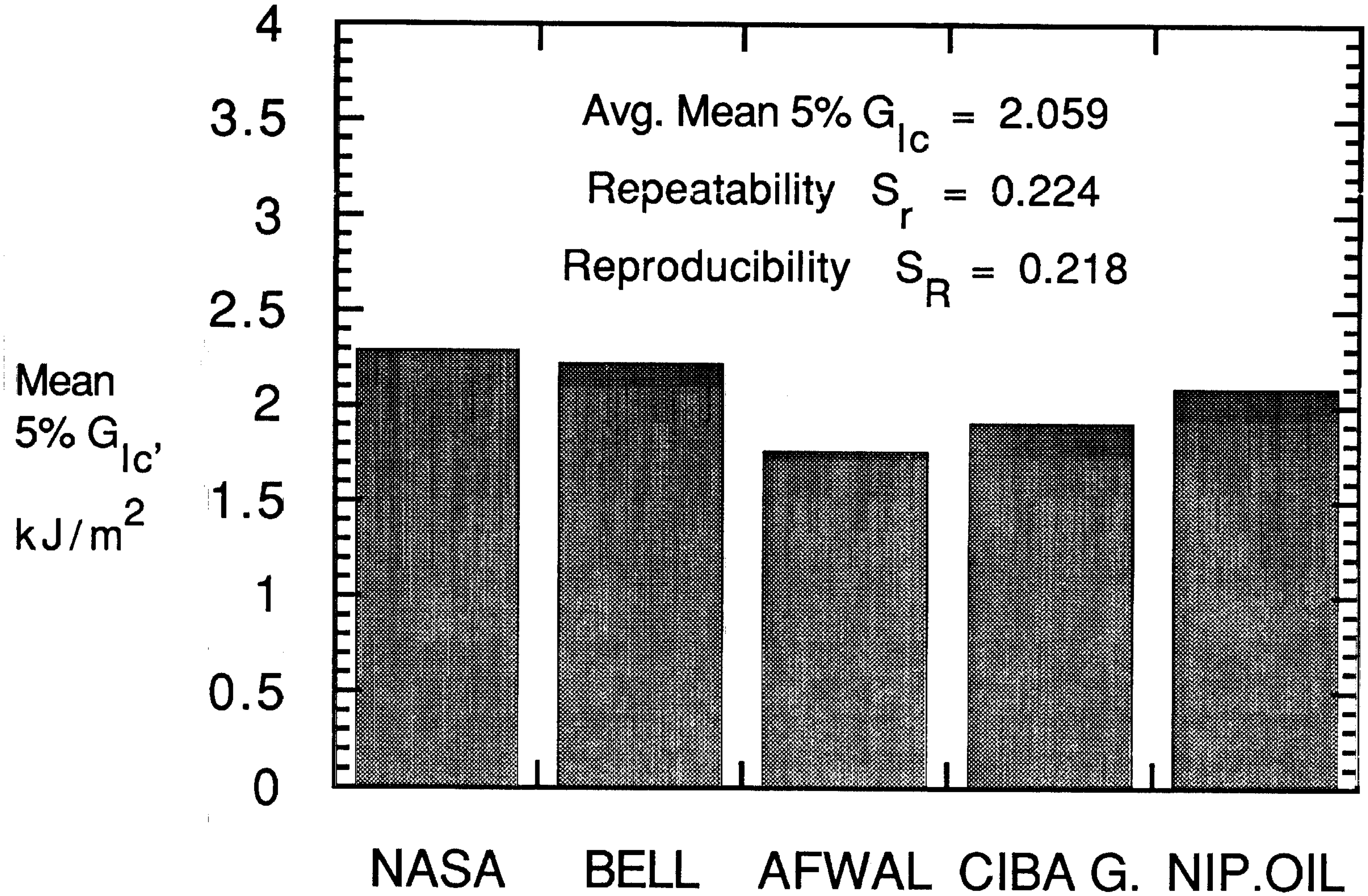

AS4/PEEK DCB
INT. ROUND ROBIN
7 μm Al. insert
NL
VIS
5%
NASA
BELL
AFWAL
CIBA G.
NIP.OIL
0
0.5
1
1.5
2
2.5
3
3.5
4
Mean
G_{Ic}',
kJ/m^2

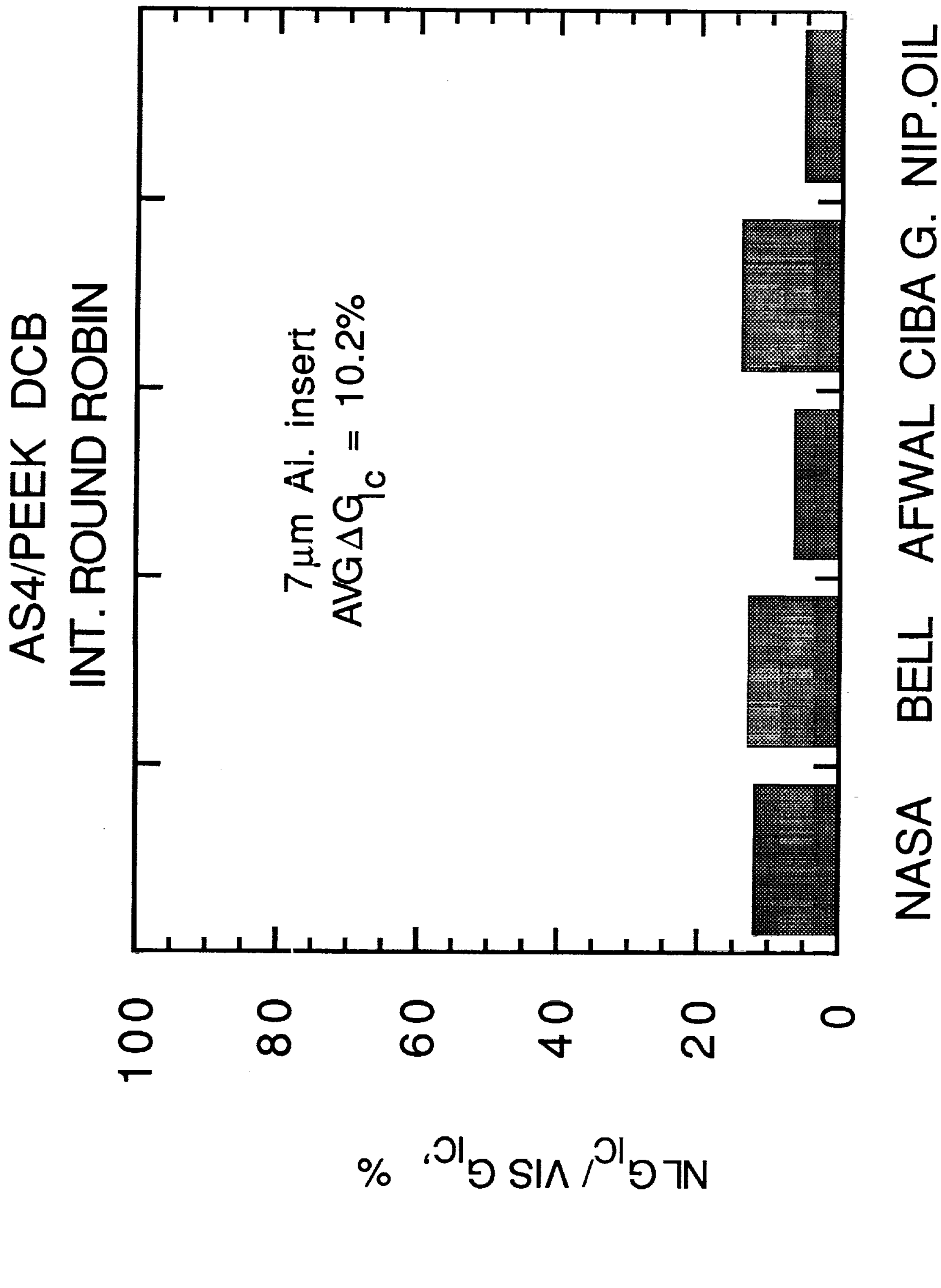

AS4/PEEK DCB
INT. ROUND ROBIN
STANDARD DEVIATION
0
0.2
0.4
0.6
0.8
1
NL
VIS
5%
7 μm Al. insert
NASA
BELL
AFWAL
CIBA G.
NIP.OIL


AS4/PEEK DCB INT. ROUND ROBIN
7 MICRON AL. INSERT
Avg. Mean NL G_{Ic} = 1.727
Repeatability S_r = 0.226
Reproducibility S_R = 0.140
Mean NL G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
NASA
BELL
AFWAL
CIBA G.
NIP.OIL

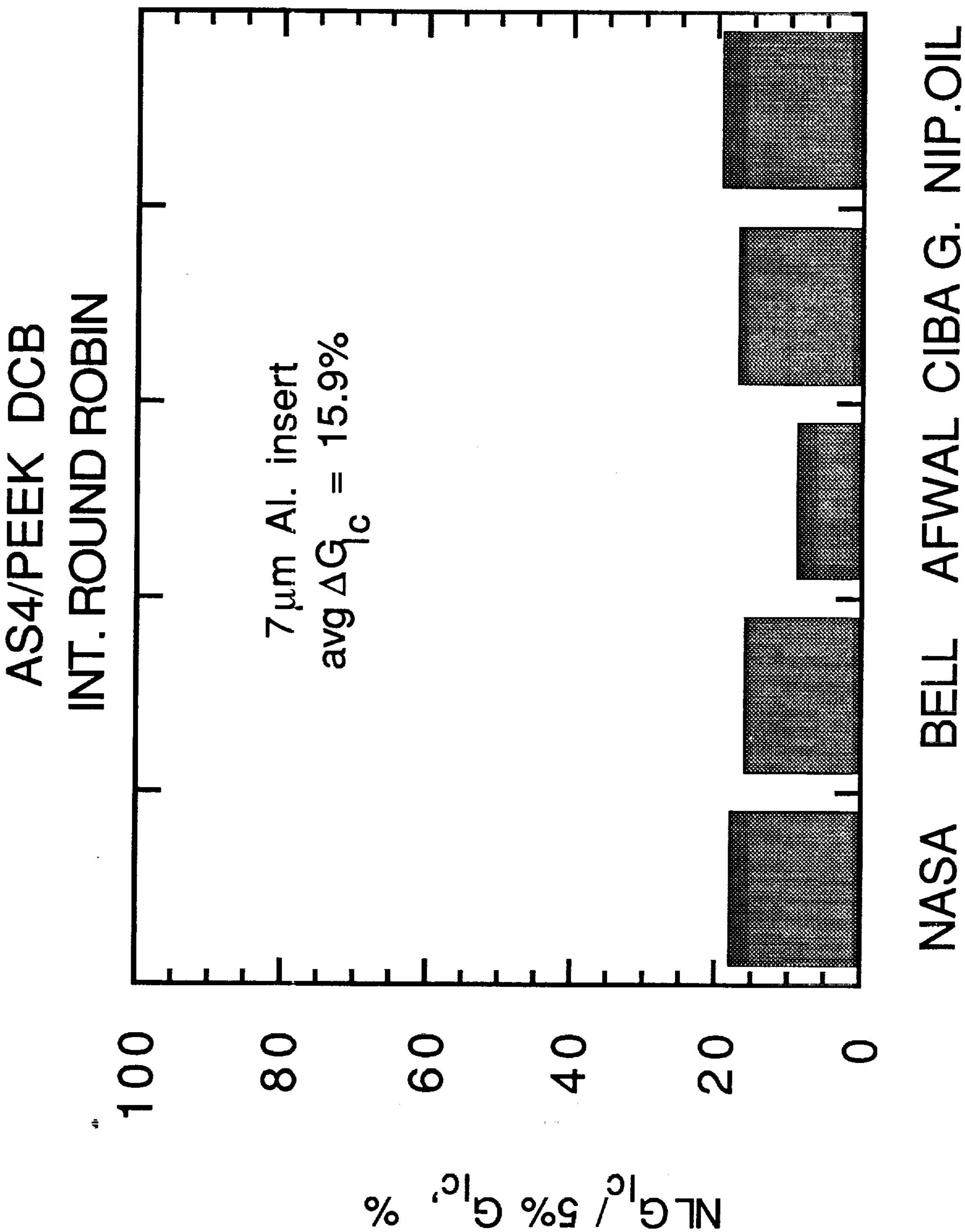

AS4/PEEK DCB INT. ROUND ROBIN
7 MICRON AL. INSERT
Avg. Mean VIS G_{Ic} = 1.929
Repeatability S_r = 0.257
Reproducibility S_R = 0.201
Mean
VIS G_{Ic},
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
AFWAL
CIBA G.
NIP.OIL

AS4/PEEK DCB INT. ROUND ROBIN
7 MICRON AL. INSERT





AS4/PEEK DCB
INT. ROUND ROBIN
7μm Al. insert
AVG ΔG_{Ic} = 10.2%
100
80
60
40
20
0
NL G_{IC} / VIS G_{IC}, %
NASA
BELL
AFWAL
CIBA G.
NIP.OIL


AS4/PEEK DCB
INT. ROUND ROBIN
7μm Al. insert
avg ΔG_Ic = 15.9%
NLG_Ic / 5% G_Ic, %
0
20
40
60
80
100
NASA
BELL
AFWAL
CIBA G.
NIP.OIL

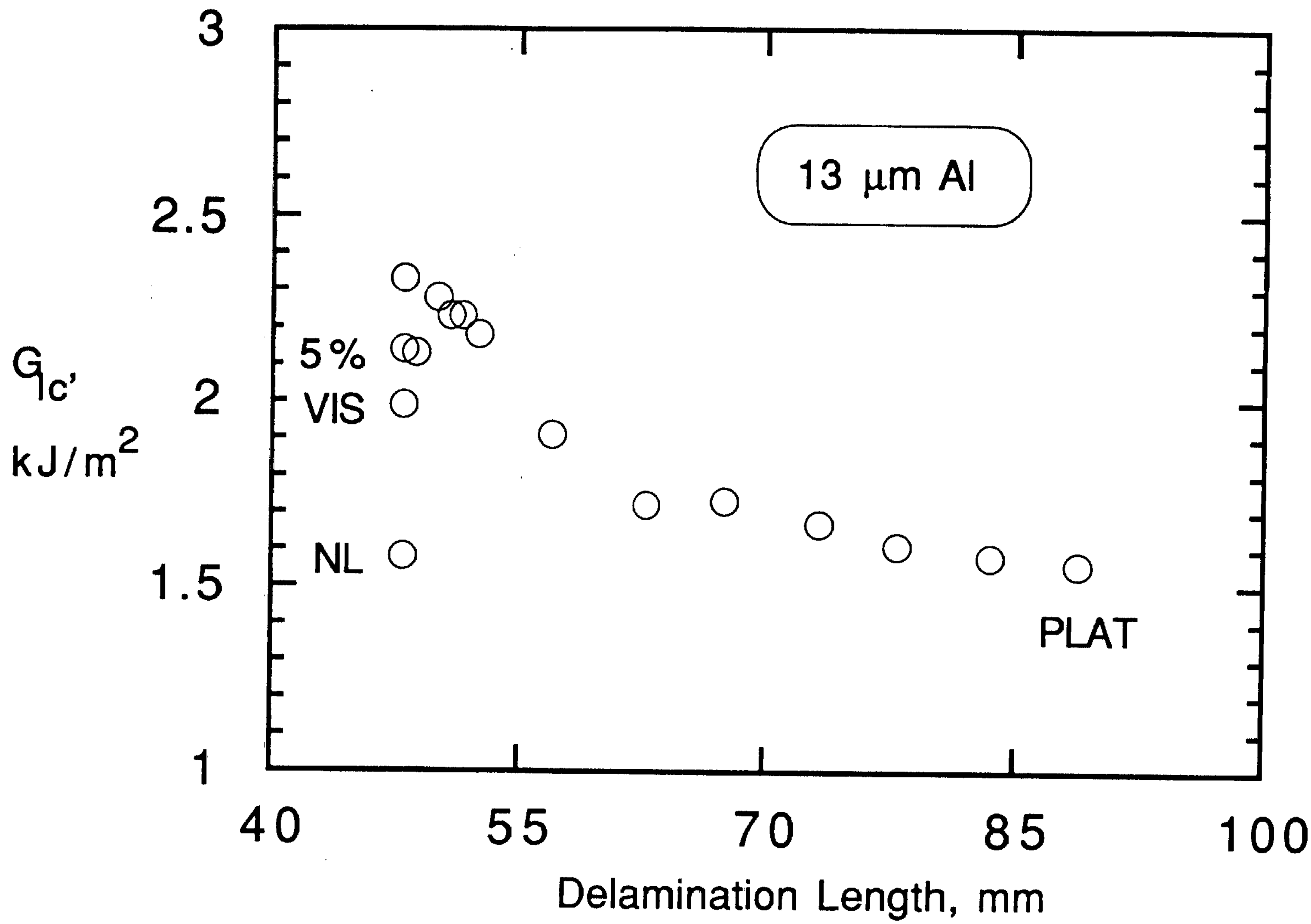


Fig. 5a R-curve for AS4/PEEK with 13 μm Aluminum insert

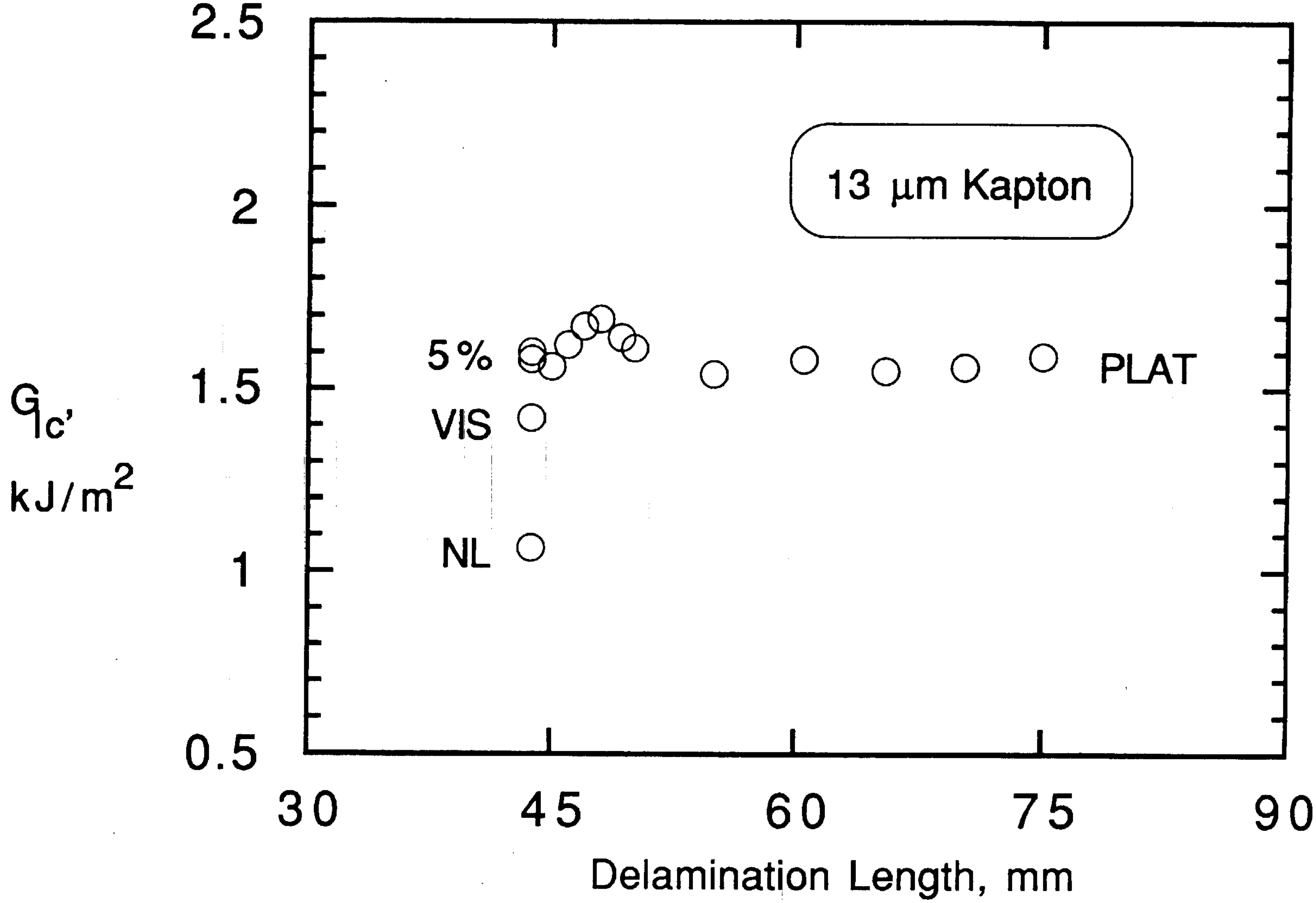
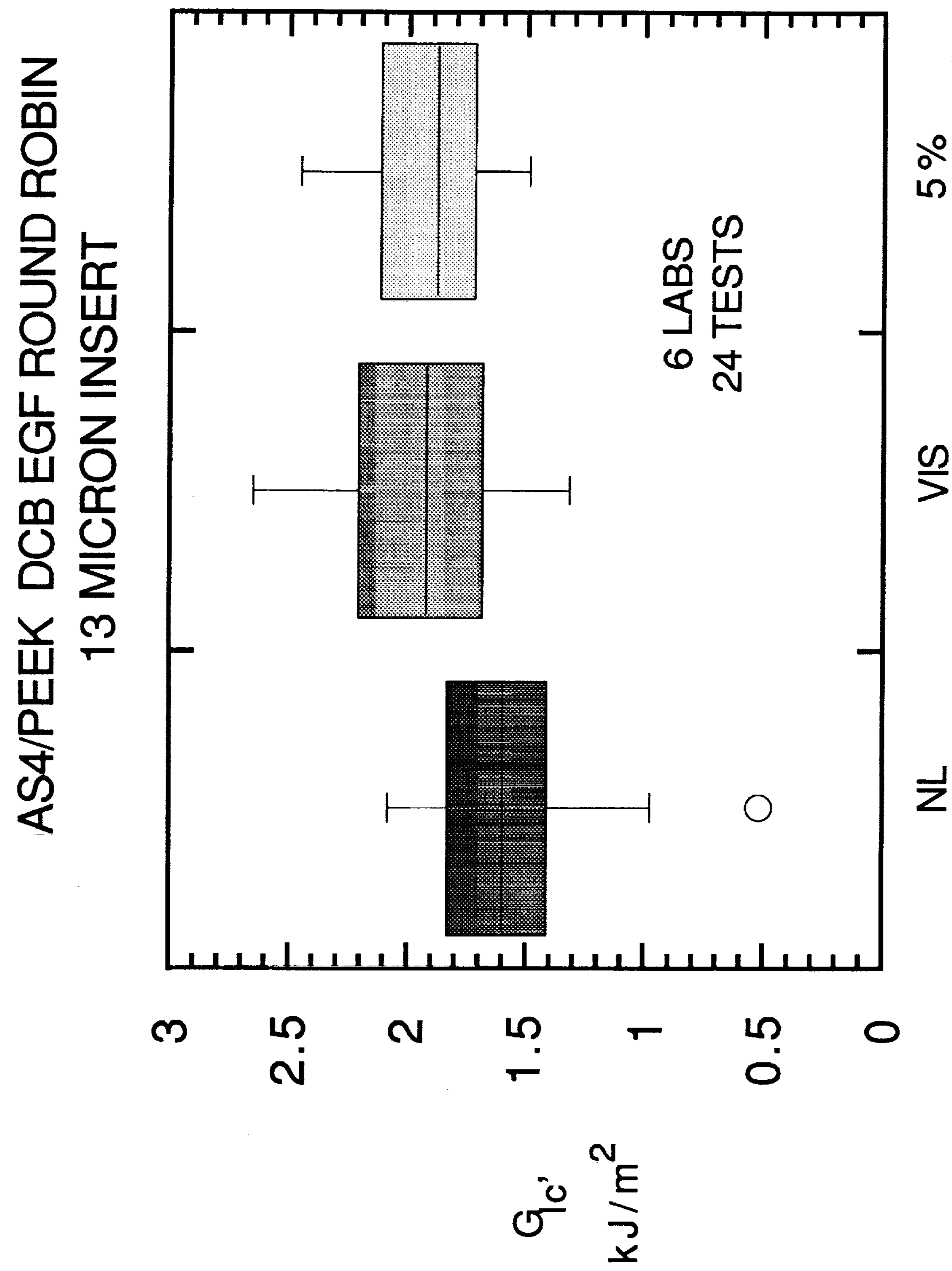
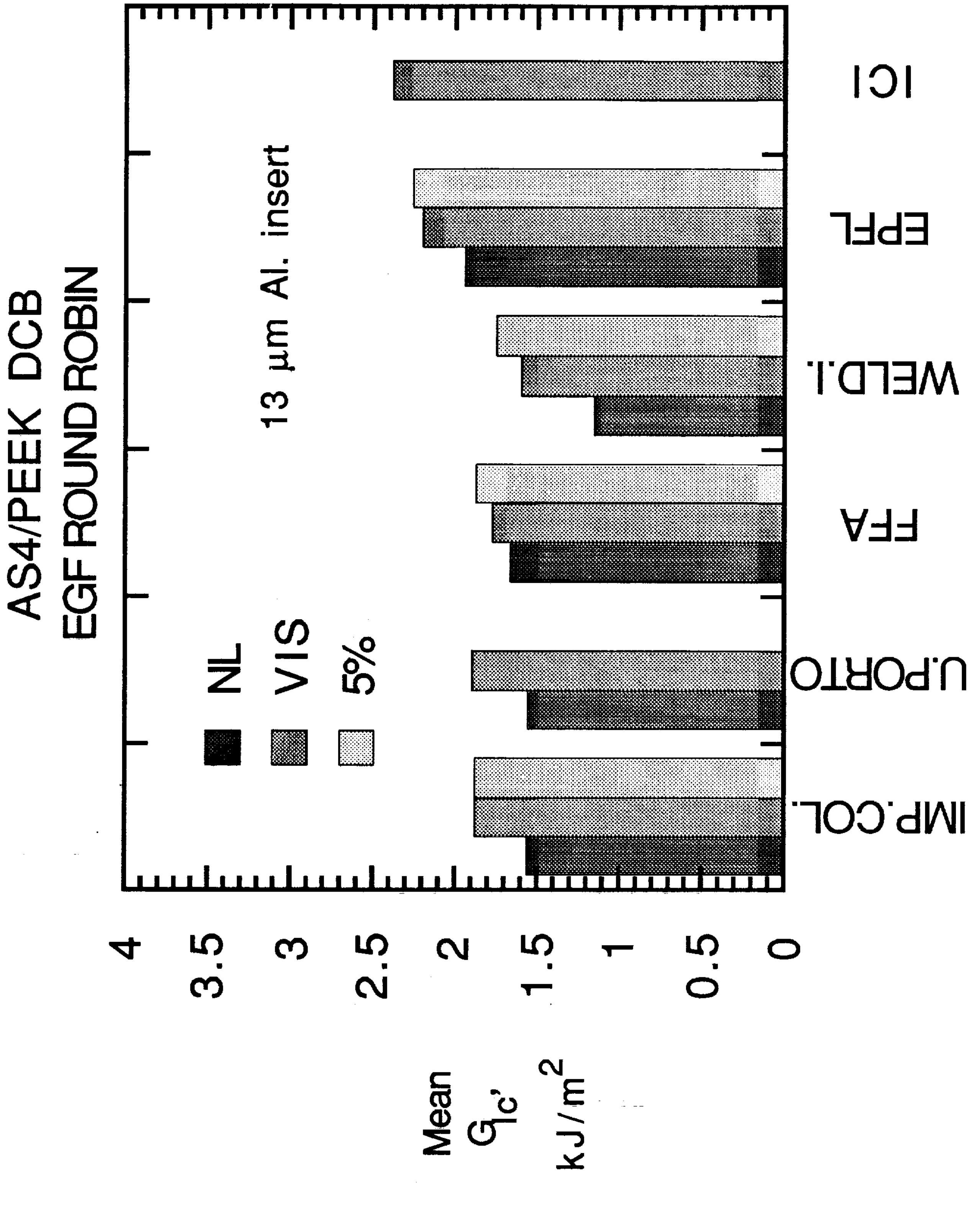
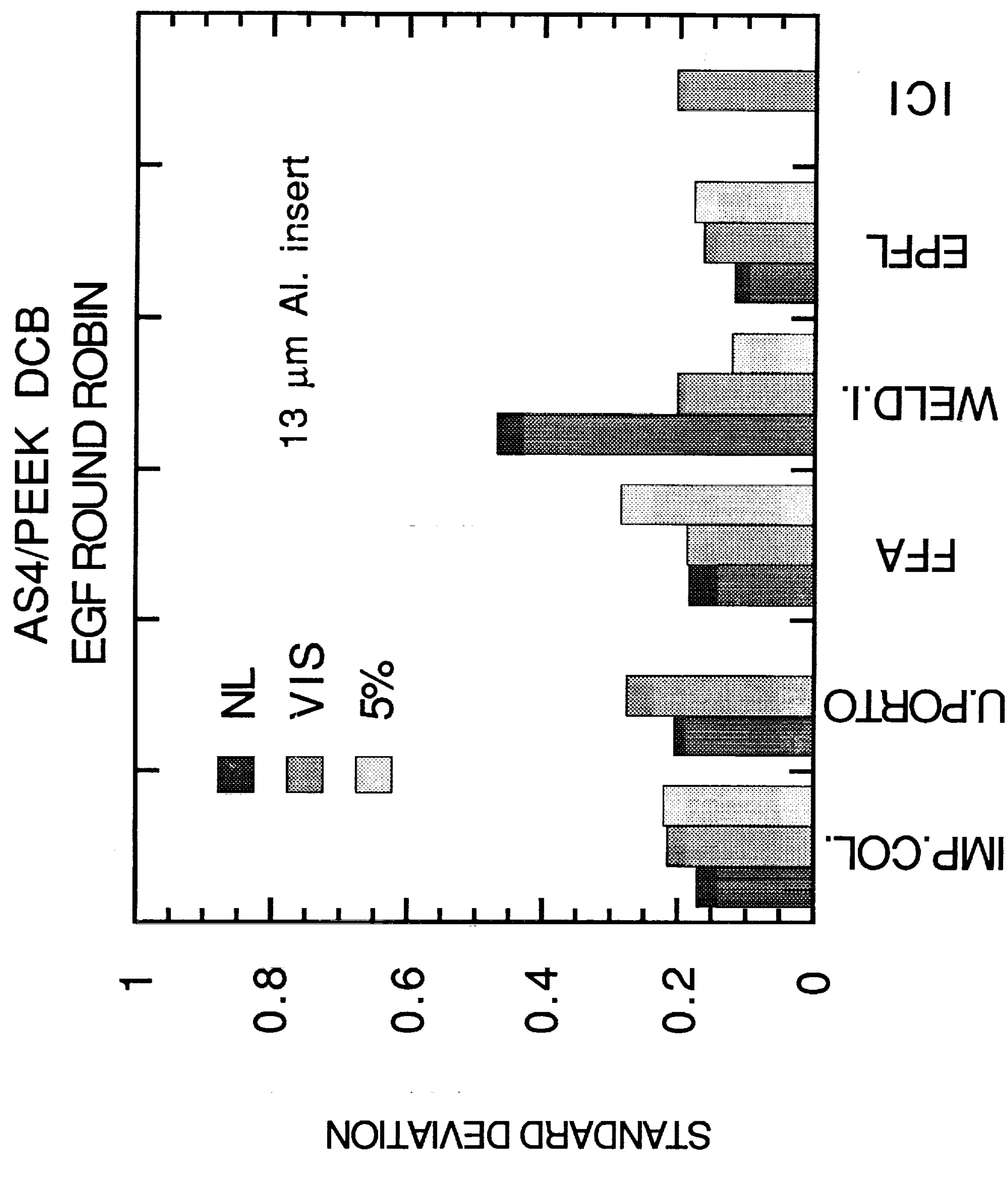
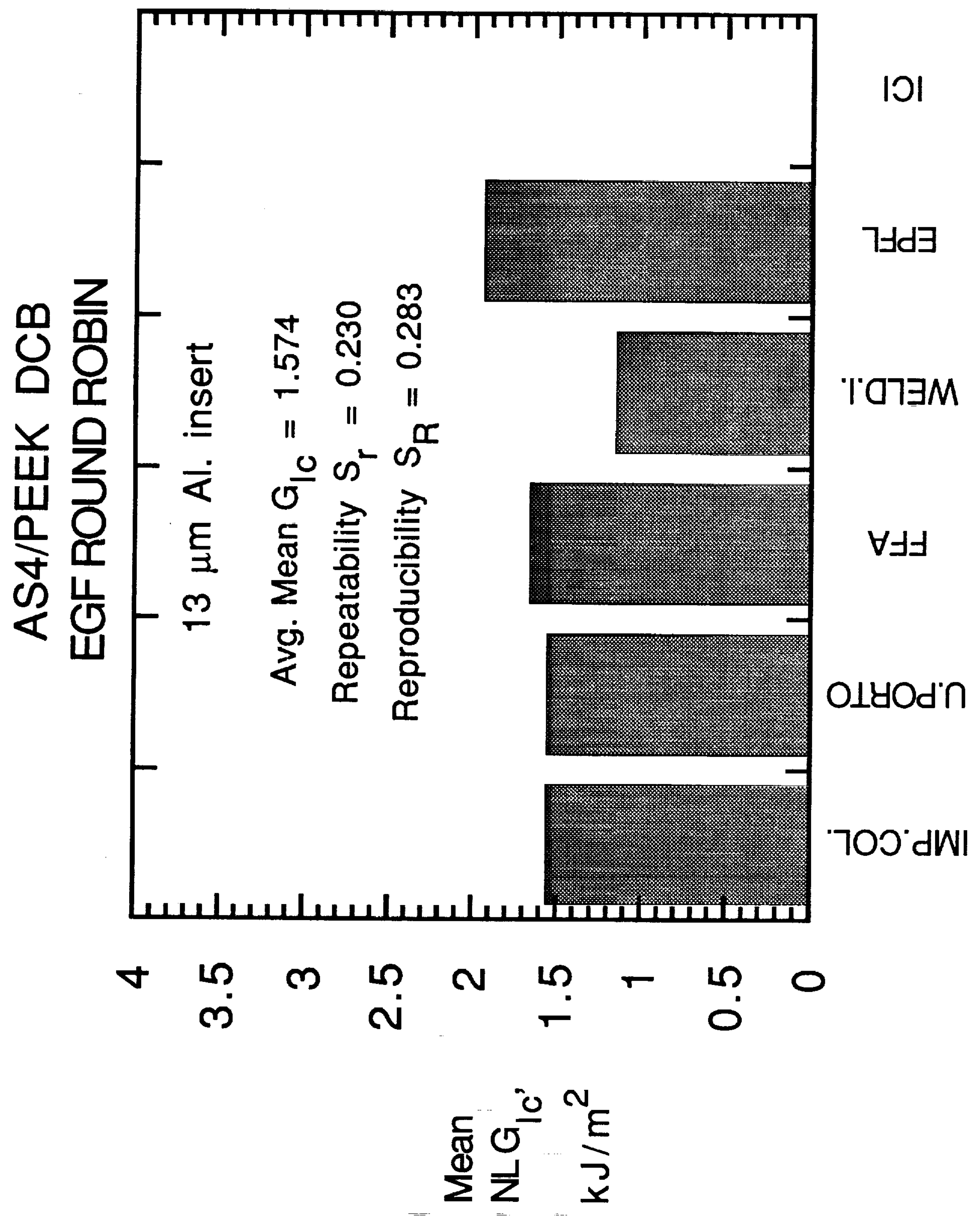


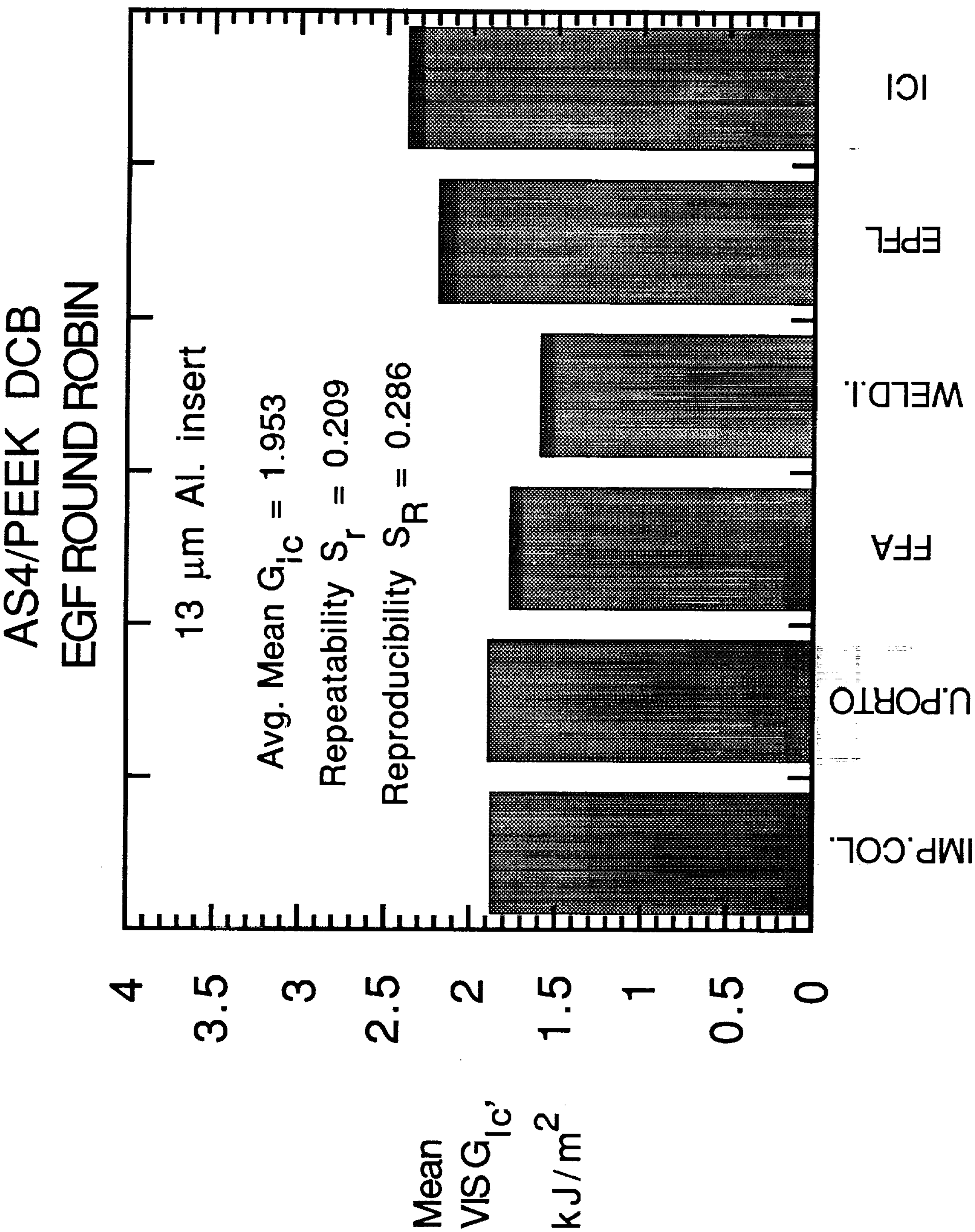
Fig. 5b R-curve for AS4/PEEK with 13 μm Kapton insert

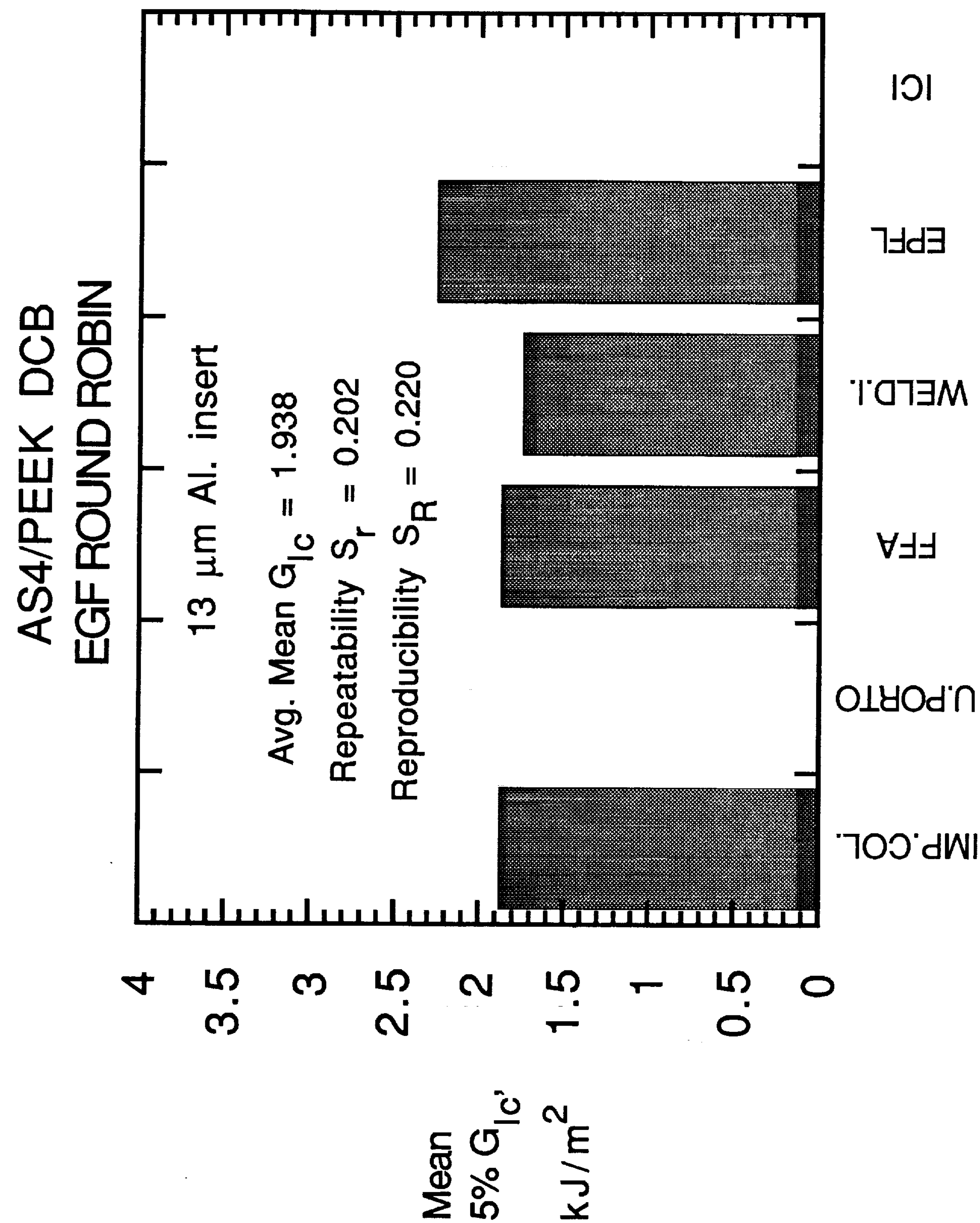

AS4/PEEK DCB EGF ROUND ROBIN
13 MICRON INSERT
6 LABS
24 TESTS
G_{Ic}'
kJ/m^2
3
2.5
2
1.5
1
0.5
0
NL
VIS
5%

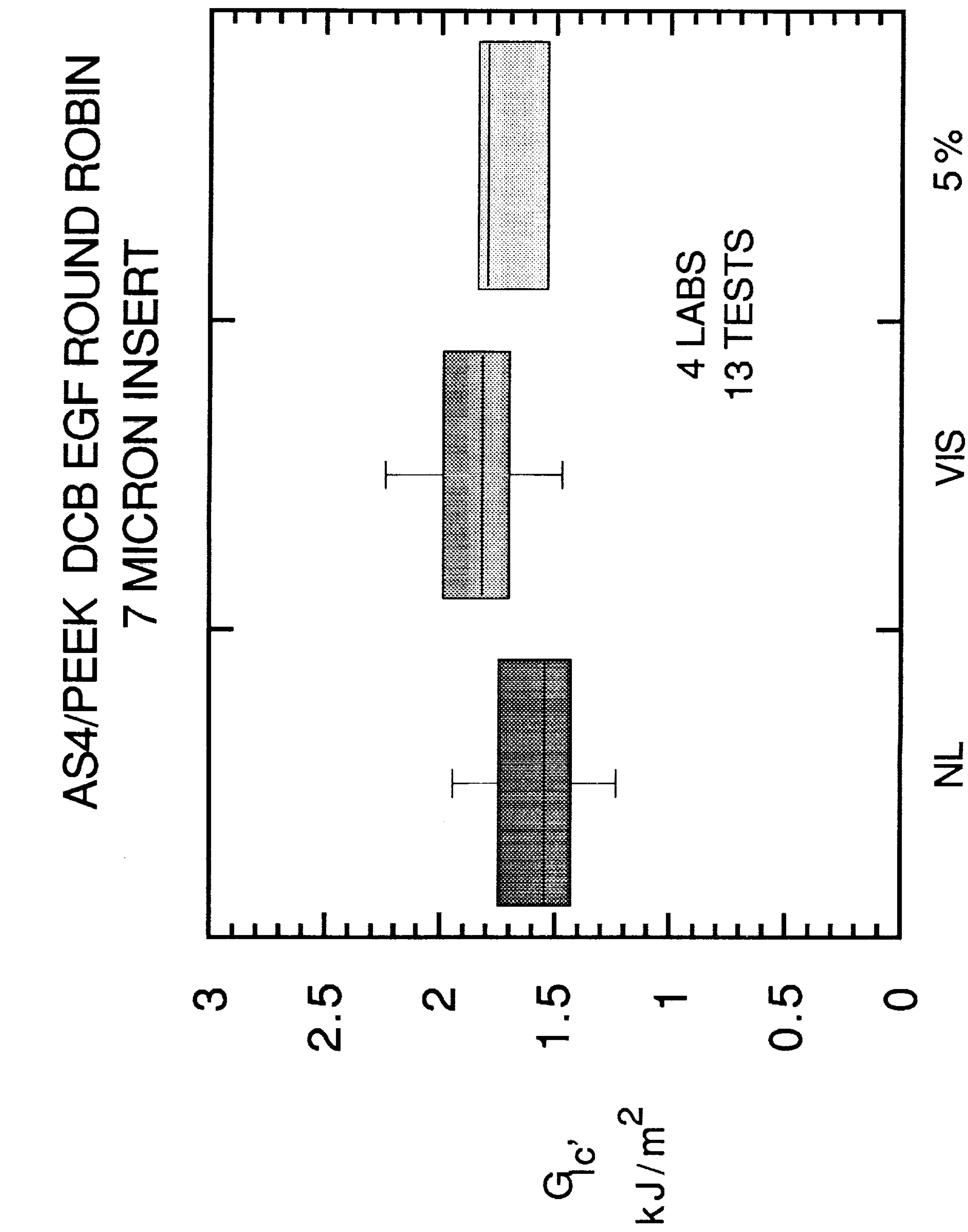

AS4/PEEK DCB
EGF ROUND ROBIN
13 μm Al. insert
NL
VIS
5%
IMP.COL.
U.PORTO
FFA
WELD.I.
EPFL
ICI
0
0.5
1
1.5
2
2.5
3
3.5
4
Mean
G$_{Ic}$'
kJ/m^2


AS4/PEEK DCB
EGF ROUND ROBIN
13 μm Al. insert
NL
VIS
5%
IMP.COL.
U.PORTO
FFA
WELD.I.
EPFL
ICI
0
0.2
0.4
0.6
0.8
1
STANDARD DEVIATION

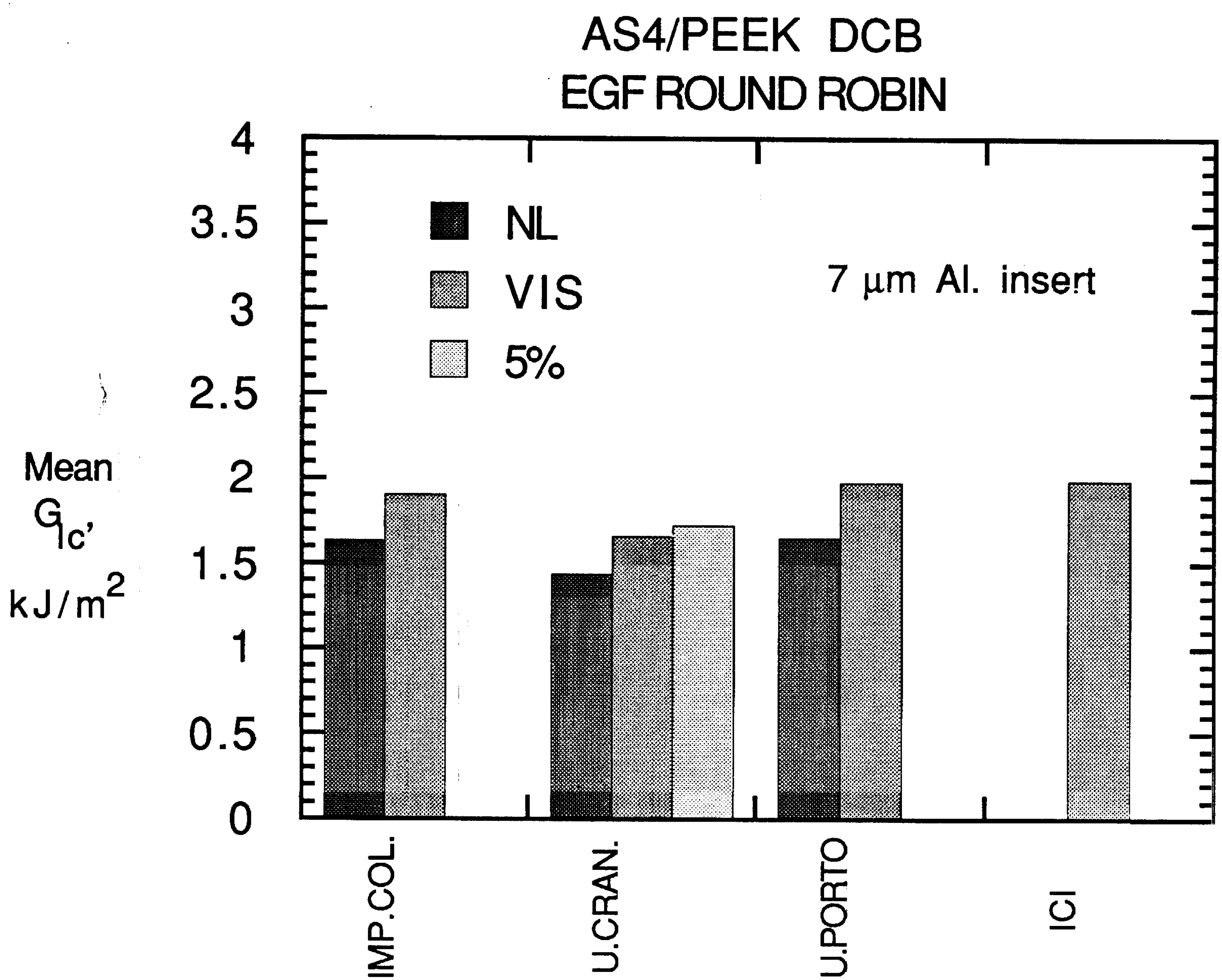

AS4/PEEK DCB
EGF ROUND ROBIN
13 μm Al. insert
Avg. Mean G_{Ic} = 1.574
Repeatability S_r = 0.230
Reproducibility S_R = 0.283
Mean NL G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
IMP.COL.
U.PORTO
FFA
WELD.I.
EPFL
ICI

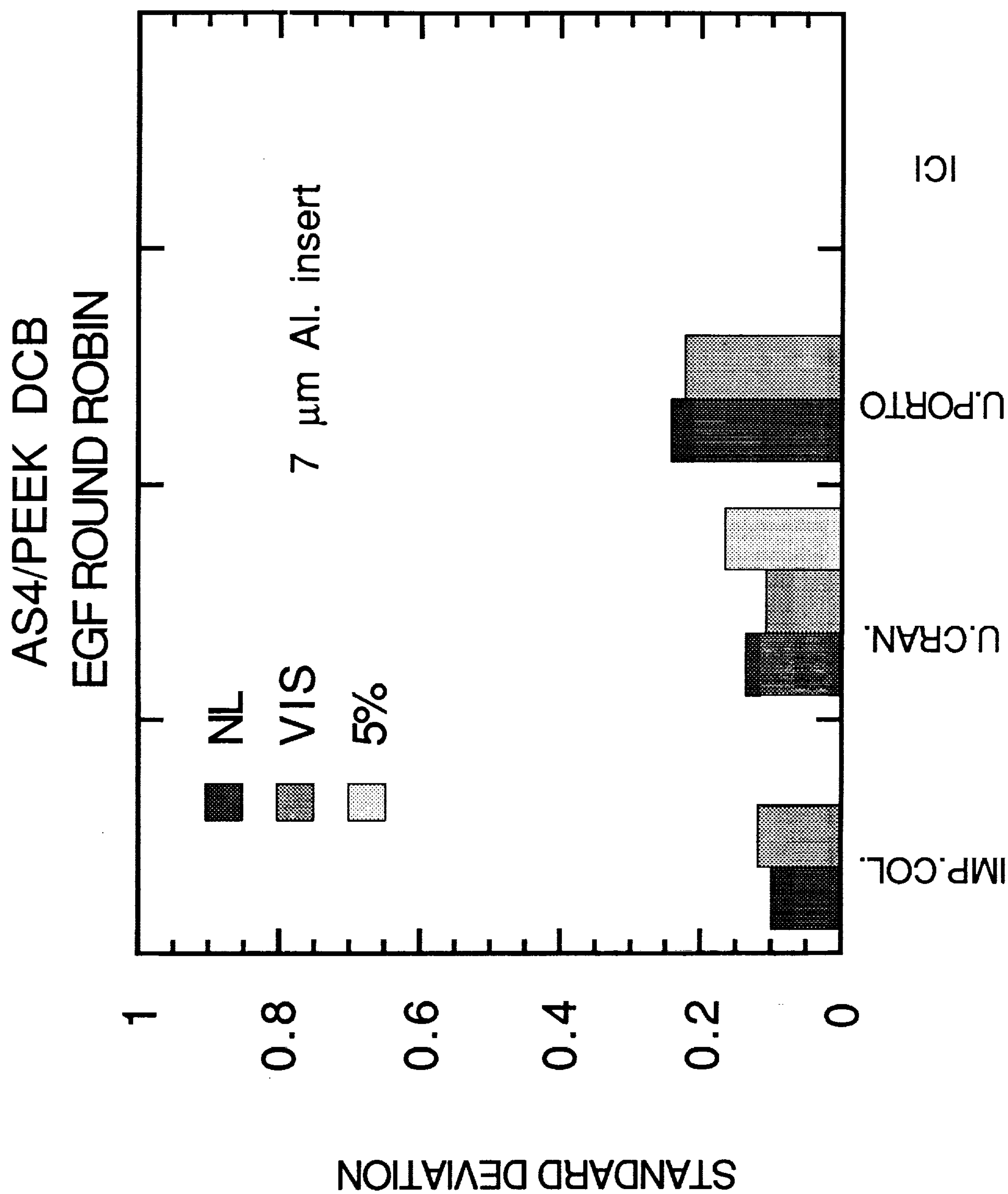

AS4/PEEK DCB
EGF ROUND ROBIN
13 μm Al. insert
Avg. Mean G_{Ic} = 1.953
Repeatability S_r = 0.209
Reproducibility S_R = 0.286
Mean
VIS G_{Ic}',
kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
IMP.COL.
U.PORTO
FFA
WELD.I.
EPFL
ICI

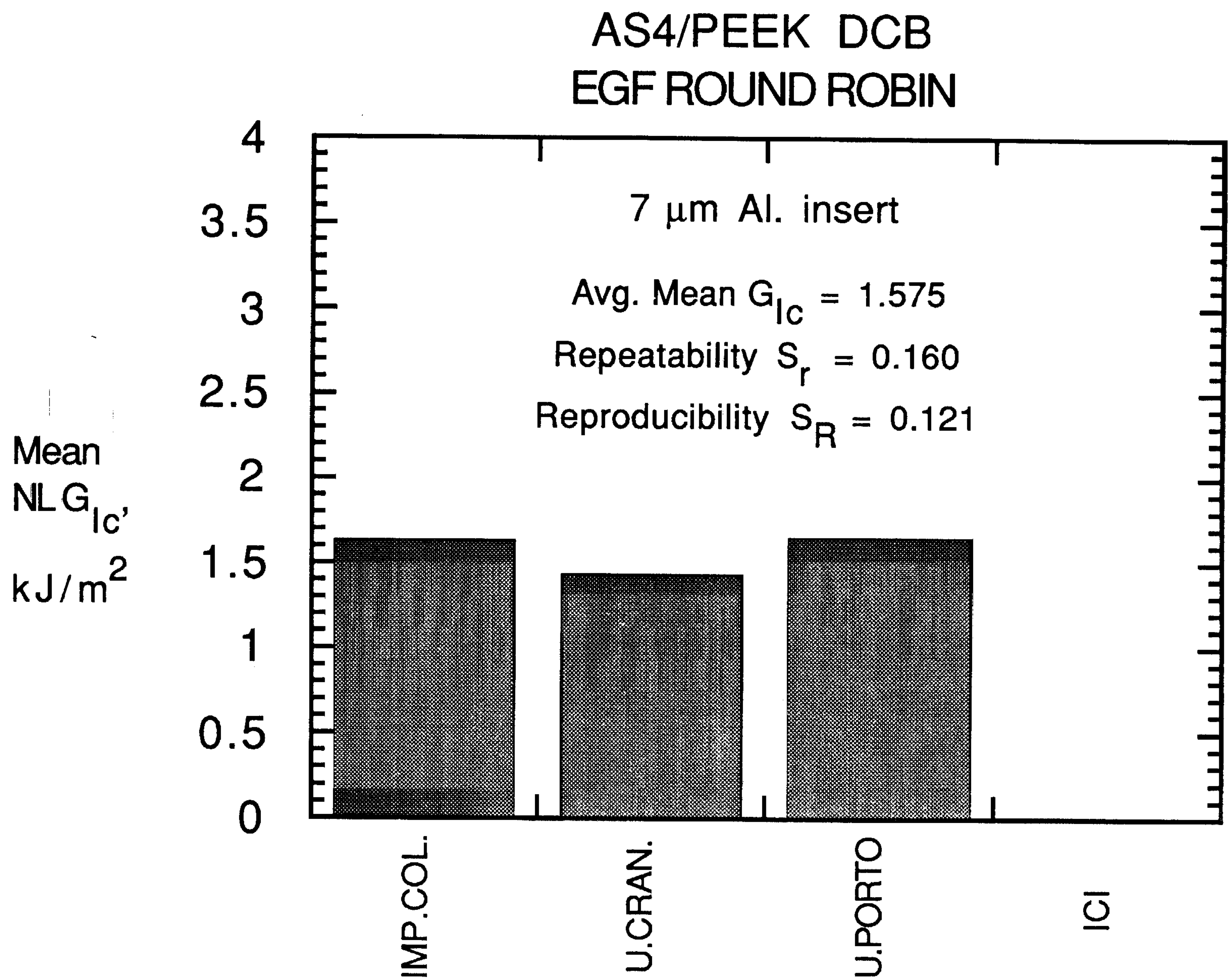

AS4/PEEK DCB
EGF ROUND ROBIN
13 μm Al. insert
Avg. Mean G_{Ic} = 1.938
Repeatability S_r = 0.202
Reproducibility S_R = 0.220
Mean 5% G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
IMP.COL.
U.PORTO
FFA
WELD.I.
EPFL
ICI

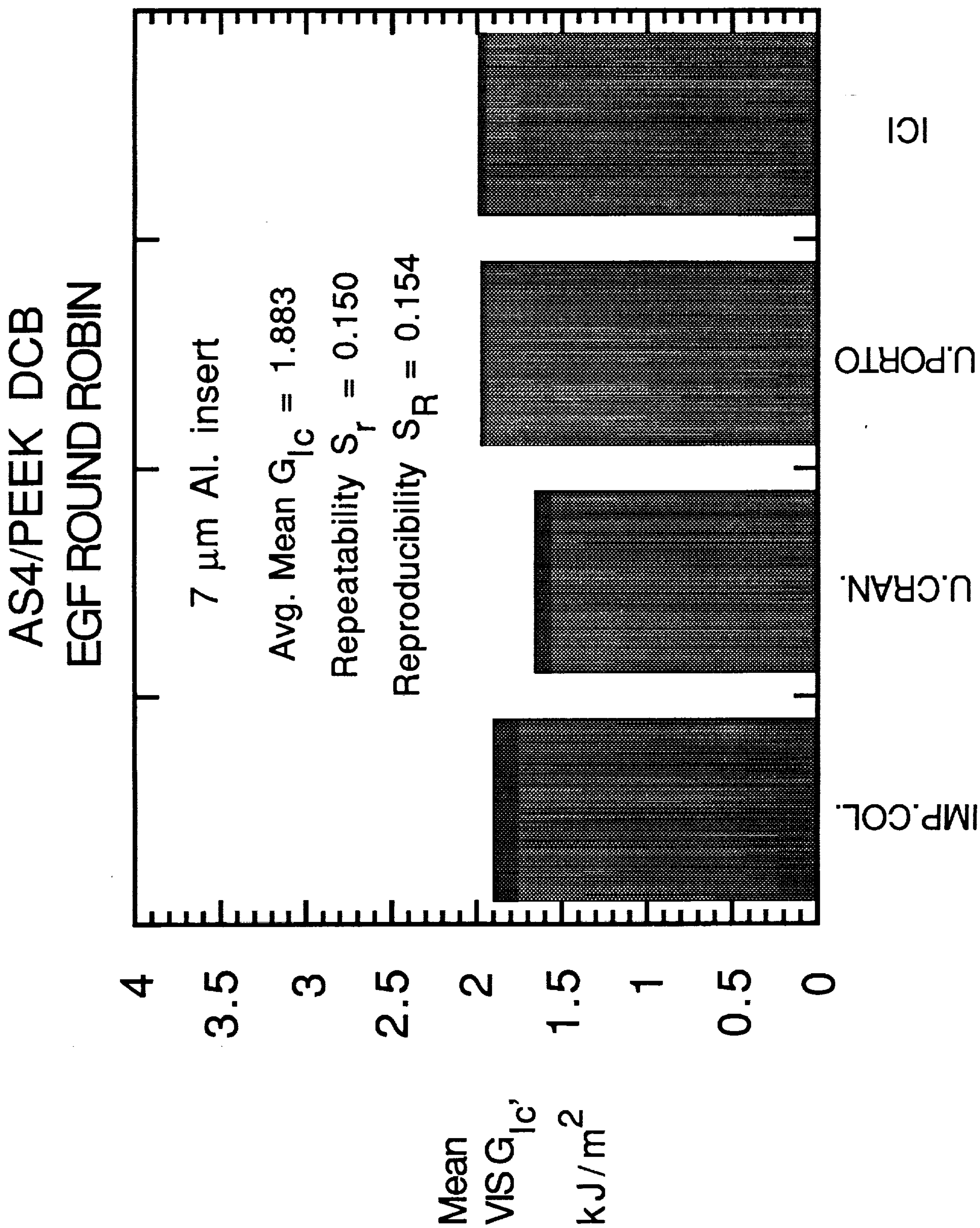

AS4/PEEK DCB EGF ROUND ROBIN
7 MICRON INSERT
G_{Ic}'
kJ/m^2
0
0.5
1
1.5
2
2.5
3
NL
VIS
5%
4 LABS
13 TESTS

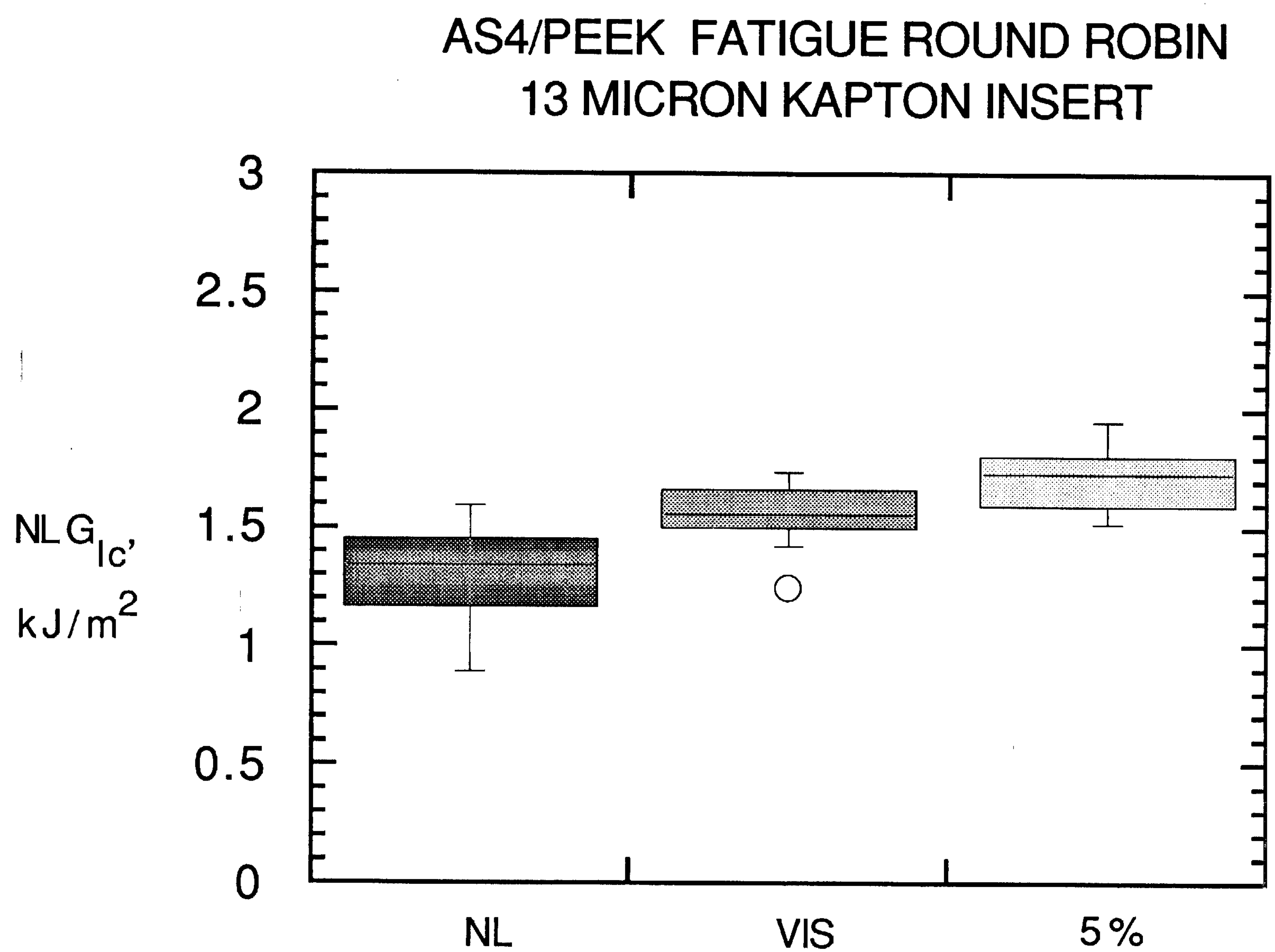
C-2

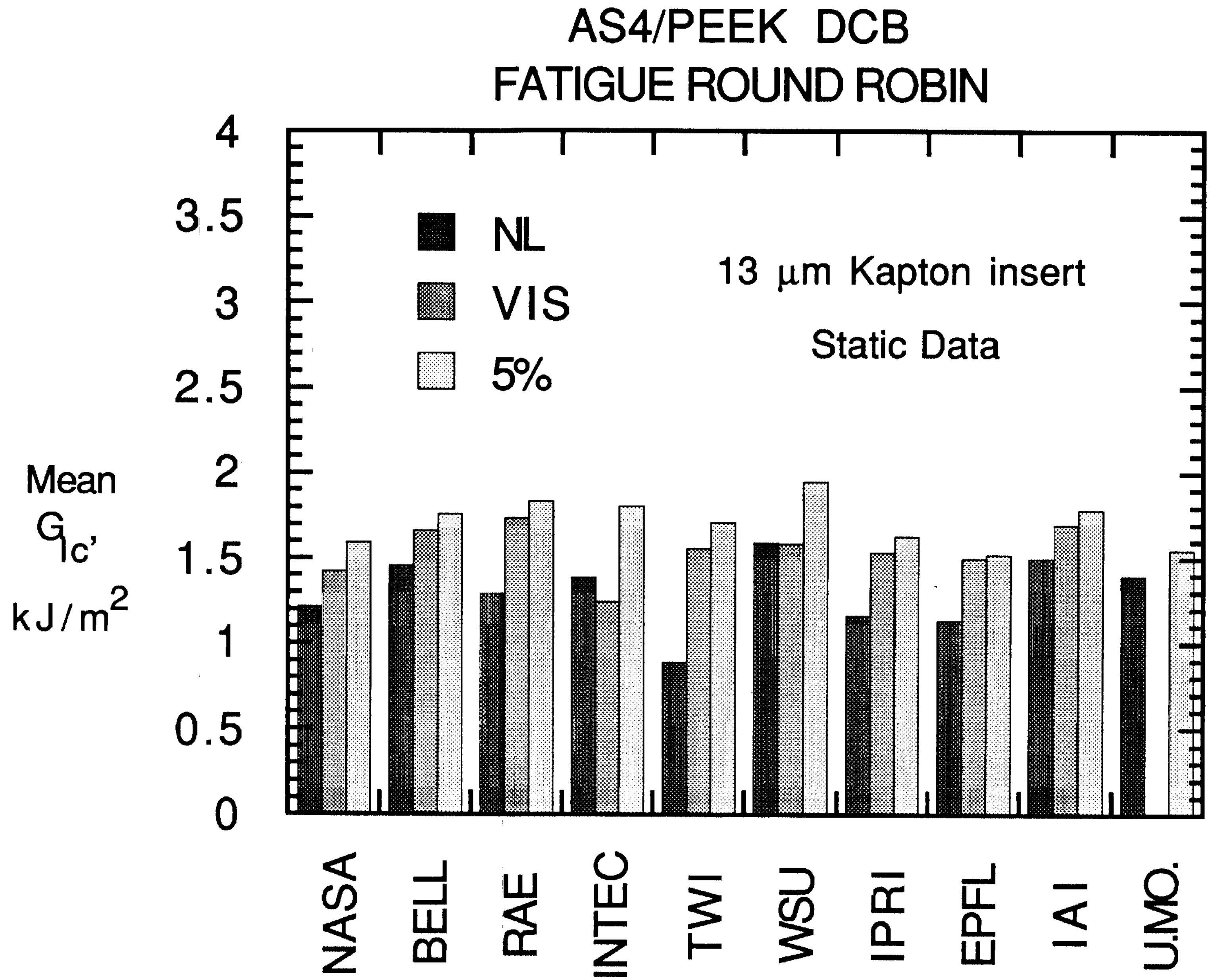

AS4/PEEK DCB
EGF ROUND ROBIN
Mean
G1c',
kJ/m2
4
3.5
3
2.5
2
1.5
1
0.5
0
NL
VIS
5%
7 µm Al. insert
IMP.COL.
U.CRAN.
U.PORTO
ICI

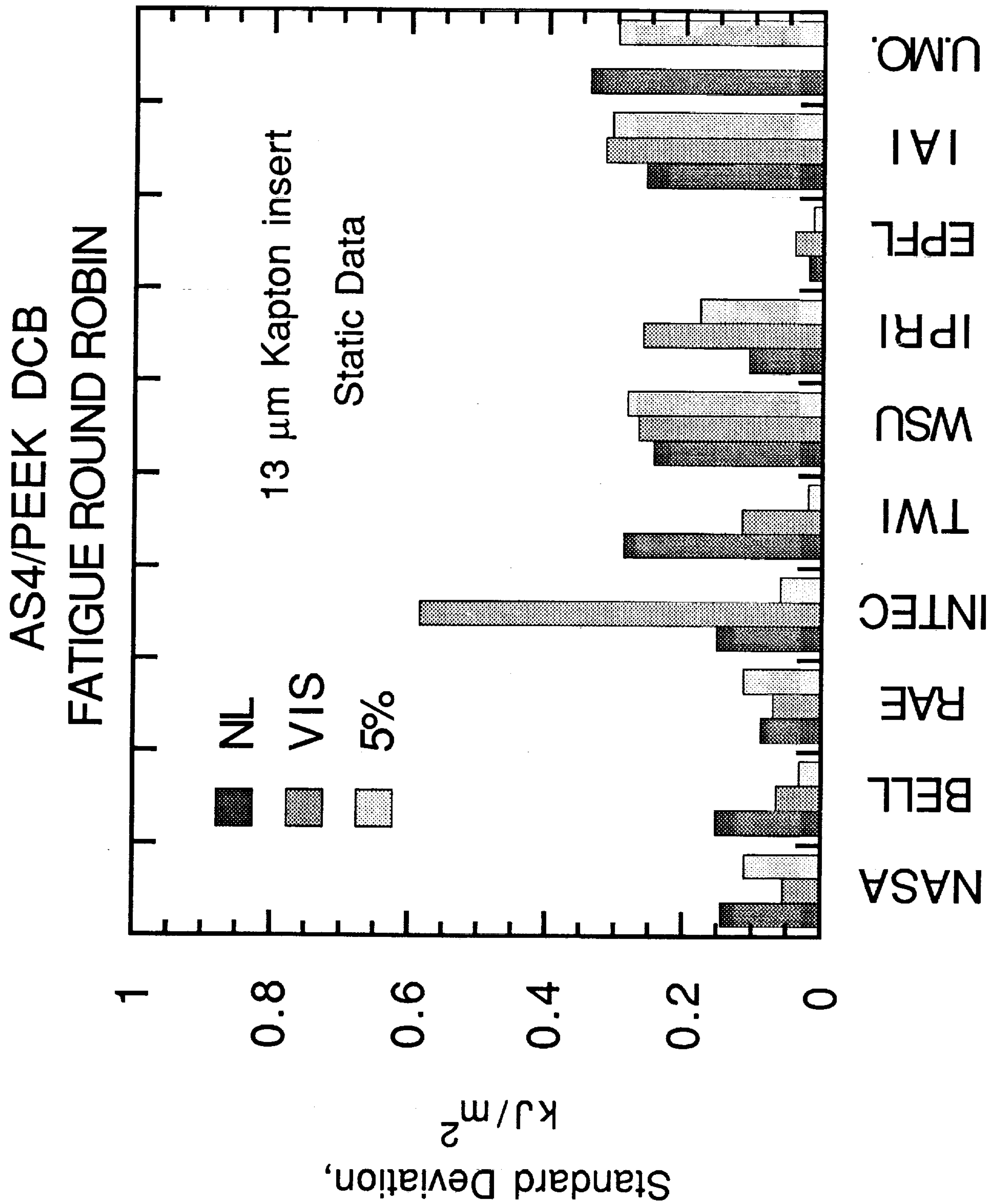

AS4/PEEK DCB
EGF ROUND ROBIN
7 μm Al. insert
NL
VIS
5%
STANDARD DEVIATION
0
0.2
0.4
0.6
0.8
1
IMP.COL.
U.CRAN.
U.PORTO
ICI

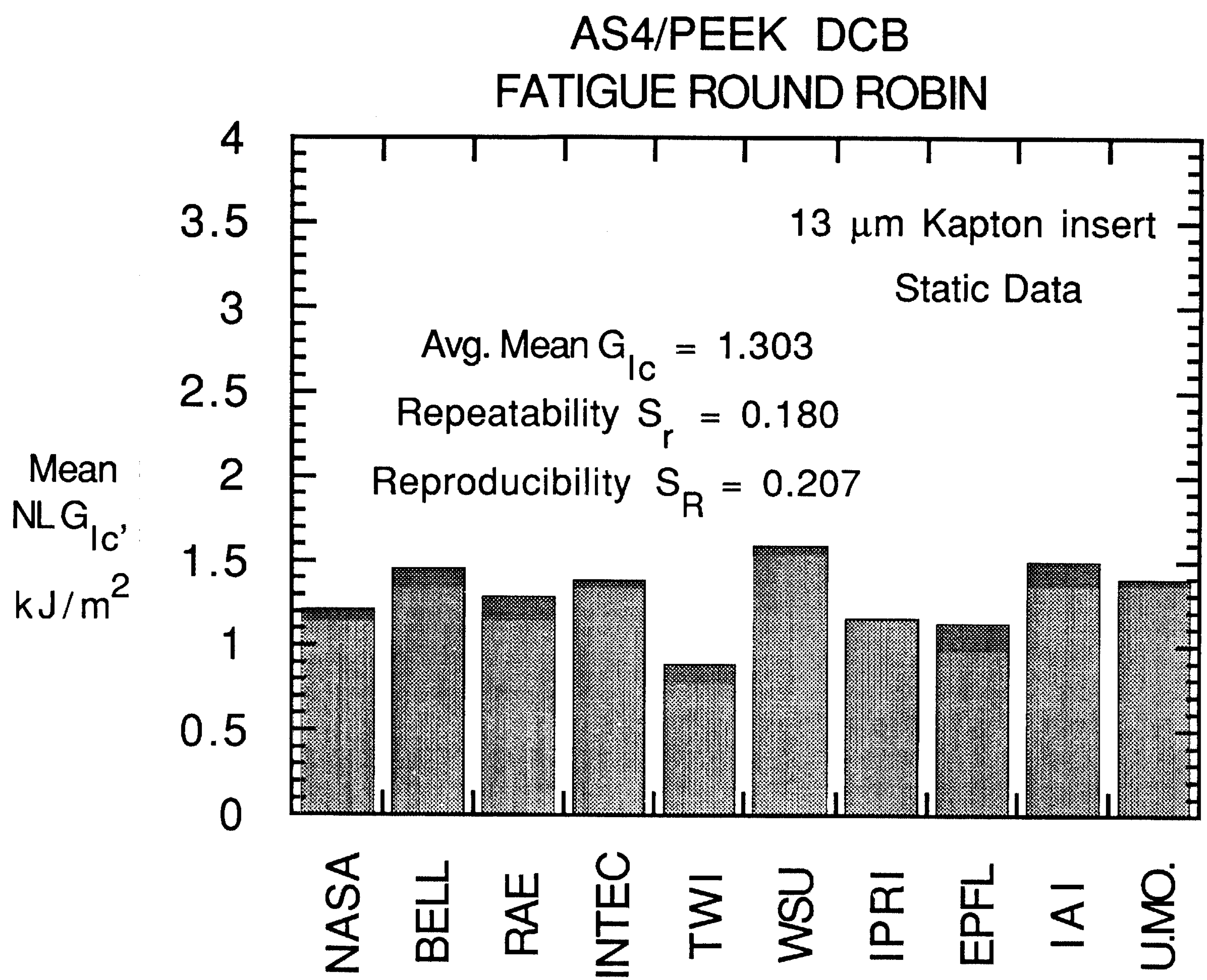

AS4/PEEK DCB
EGF ROUND ROBIN
7 µm Al. insert
Avg. Mean G_{Ic} = 1.575
Repeatability S_r = 0.160
Reproducibility S_R = 0.121
Mean
NL G_{Ic},
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
IMP.COL.
U.CRAN.
U.PORTO
ICI


AS4/PEEK DCB
EGF ROUND ROBIN
7 μm Al. insert
Avg. Mean G$_{Ic}$ = 1.883
Repeatability S$_r$ = 0.150
Reproducibility S$_R$ = 0.154
Mean VIS G$_{Ic}$', kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
IMP.COL.
U.CRAN.
U.PORTO
ICI

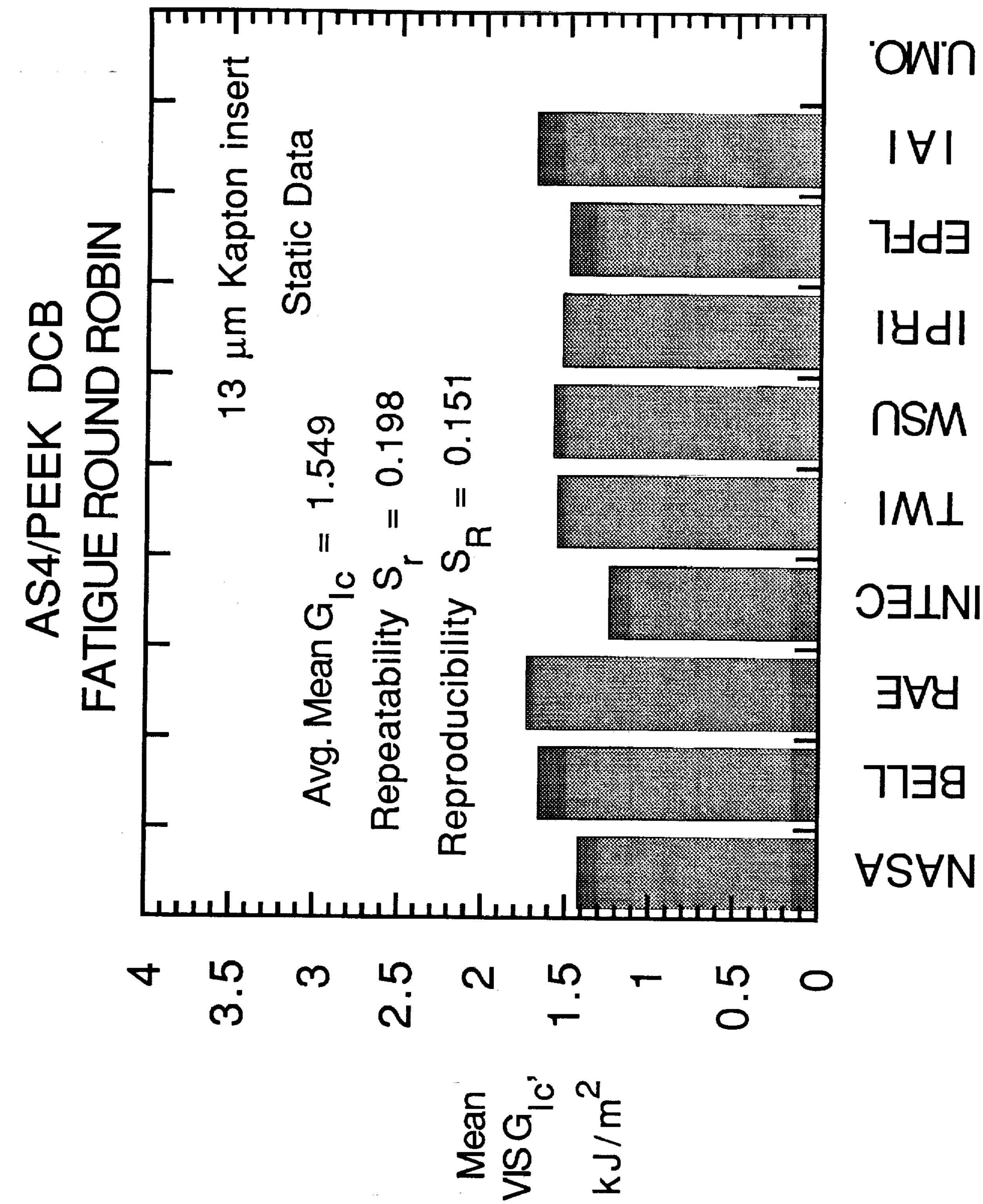

AS4/PEEK FATIGUE ROUND ROBIN
13 MICRON KAPTON INSERT
NL G$_{Ic}$,
kJ/m^2
3
2.5
2
1.5
1
0.5
0
NL
VIS
5%


AS4/PEEK DCB
FATIGUE ROUND ROBIN
Mean
G_{Ic}'
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NL
VIS
5%
13 μm Kapton insert
Static Data
NASA
BELL
RAE
INTEC
TWI
WSU
IPRI
EPFL
IAI
U.MO.

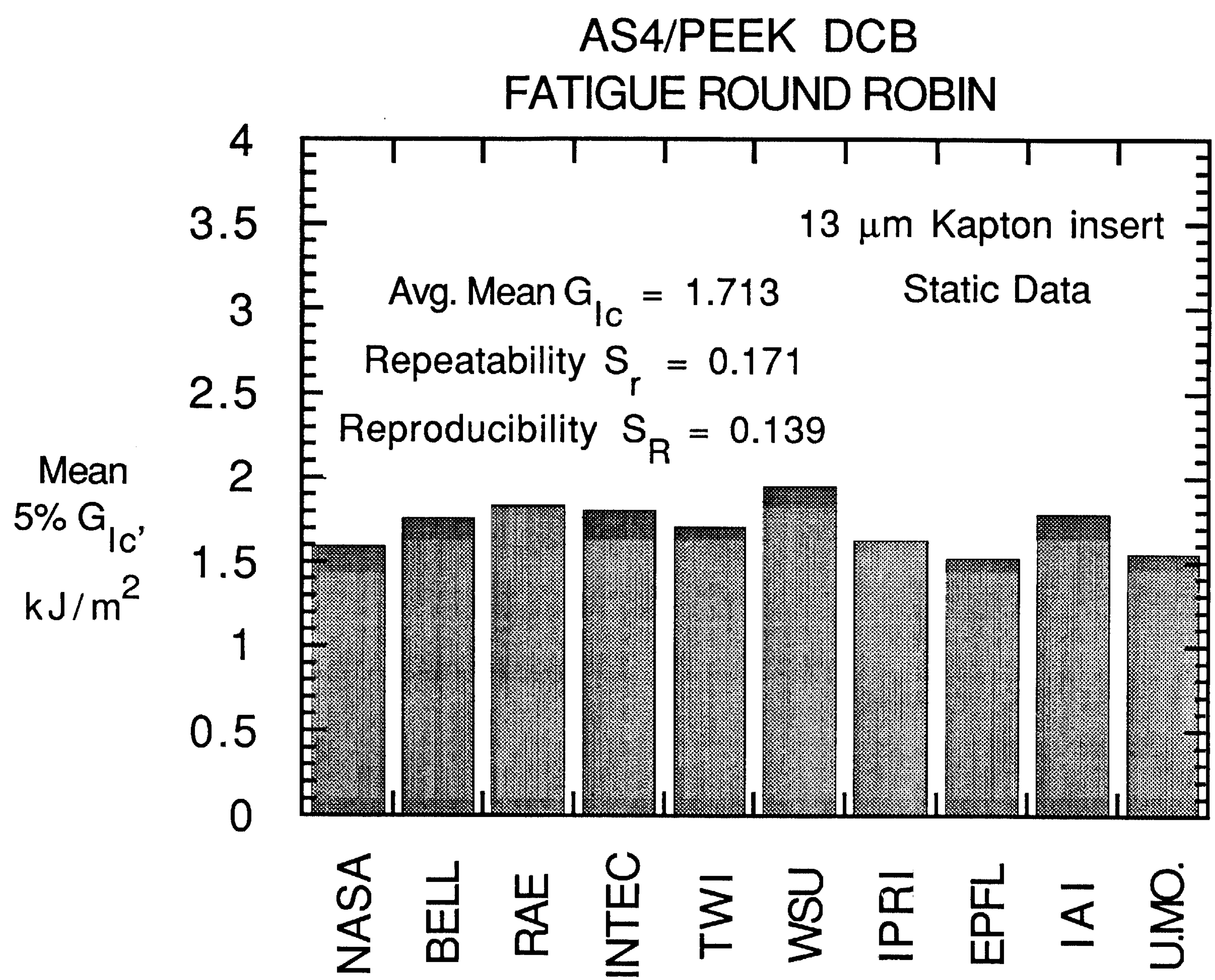

AS4/PEEK DCB
FATIGUE ROUND ROBIN
13 μm Kapton insert
Static Data
NL
VIS
5%
Standard Deviation,
kJ/m²
0
0.2
0.4
0.6
0.8
1
NASA
BELL
RAE
INTEC
TWI
WSU
IPRI
EPFL
IAI
U.MO.

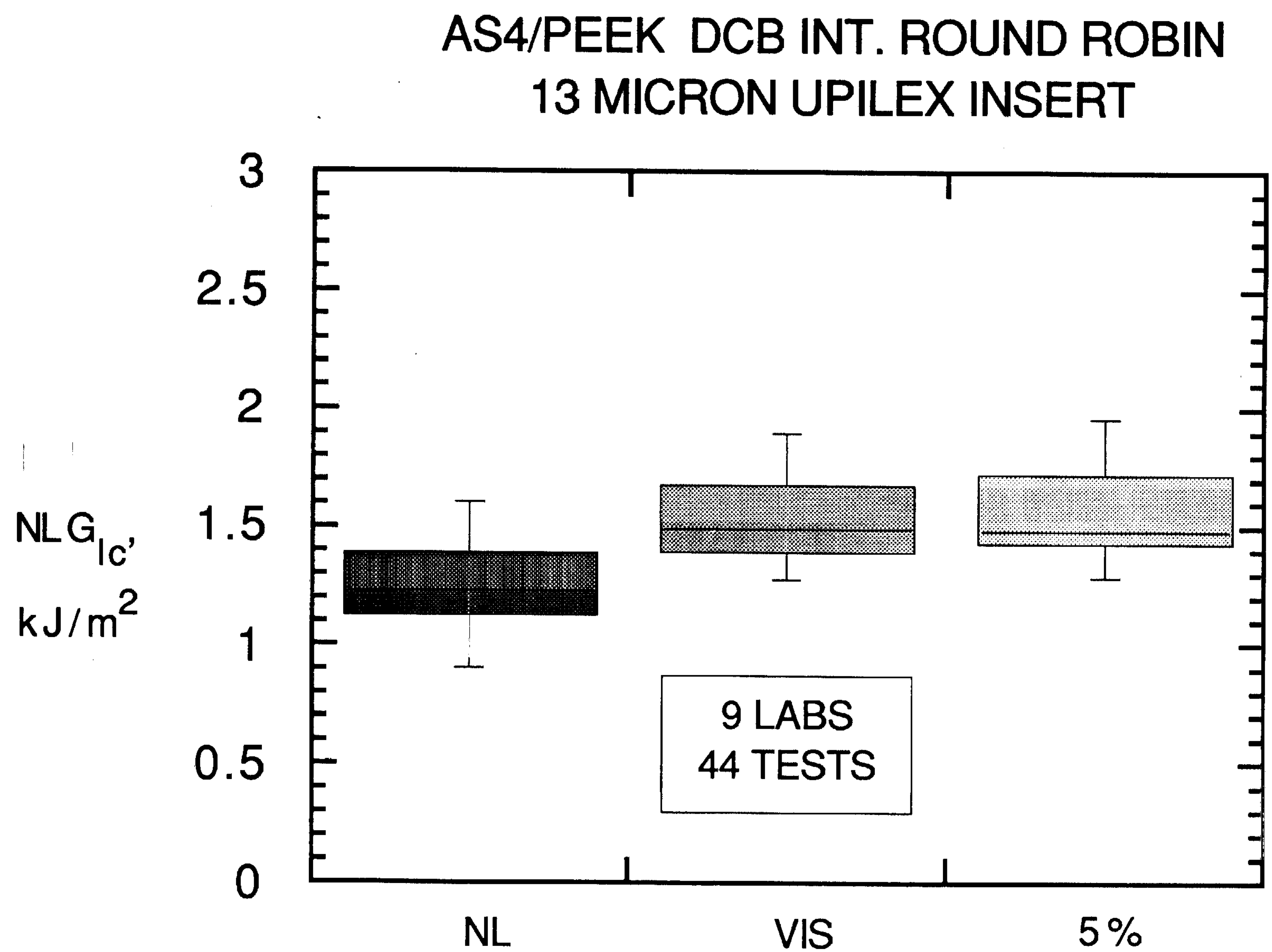


AS4/PEEK DCB
FATIGUE ROUND ROBIN
13 μm Kapton insert
Static Data
Avg. Mean G_Ic = 1.303
Repeatability S_r = 0.180
Reproducibility S_R = 0.207
Mean NL G_Ic', kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
RAE
INTEC
TWI
WSU
IPRI
EPFL
I A I
U.MO.

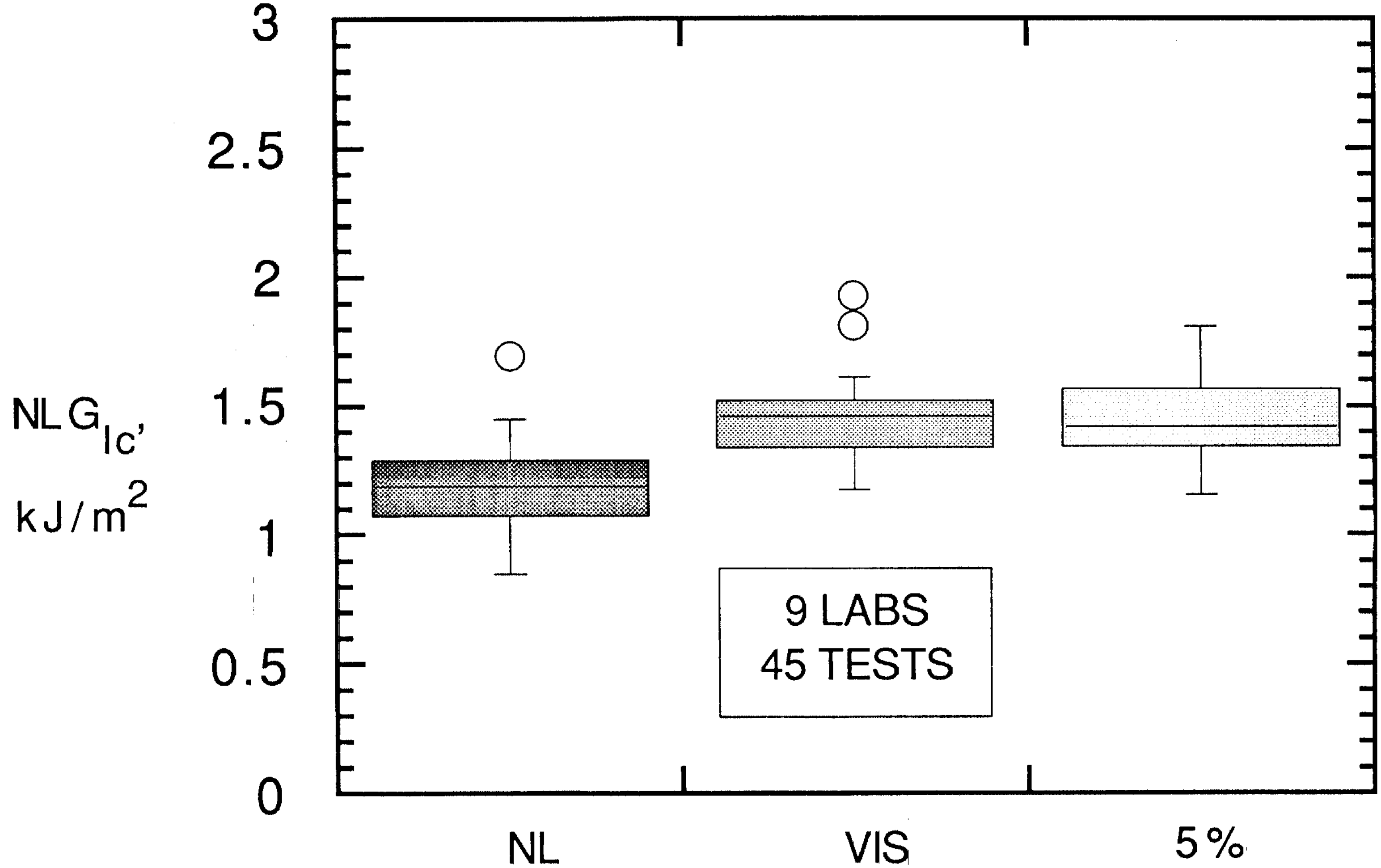


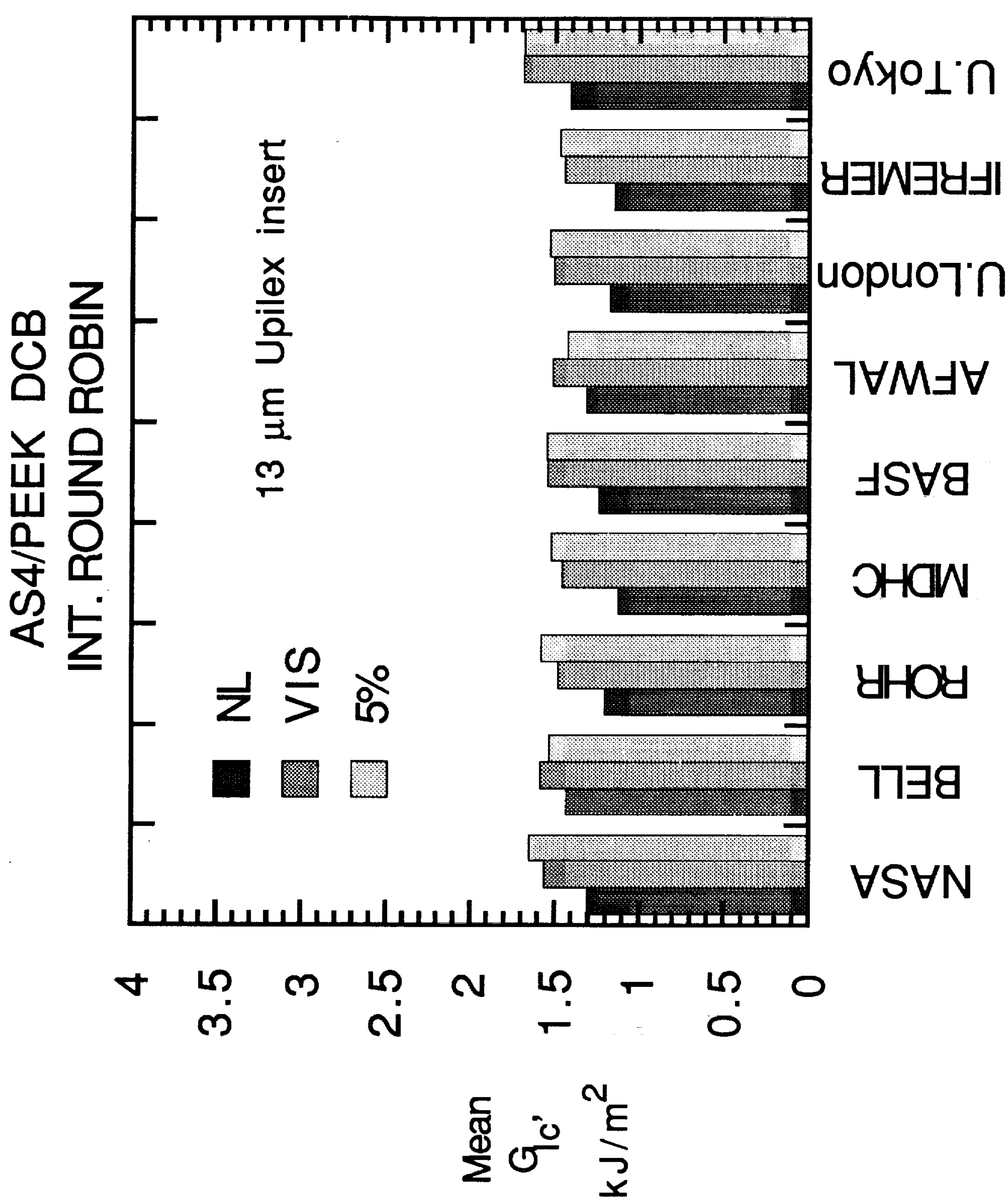
AS4/PEEK DCB
FATIGUE ROUND ROBIN
13 μm Kapton insert
Static Data
Avg. Mean G_{Ic} = 1.549
Repeatability S_r = 0.198
Reproducibility S_R = 0.151
Mean VIS G_{Ic}, kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
RAE
INTEC
TWI
WSU
IPRI
EPFL
IAI
U.MO.


AS4/PEEK DCB
FATIGUE ROUND ROBIN
13 μm Kapton insert
Static Data
Avg. Mean G_{Ic} = 1.713
Repeatability S_r = 0.171
Reproducibility S_R = 0.139
Mean
5% G_{Ic},
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
RAE
INTEC
TWI
WSU
IPRI
EPFL
IAI
U.MO.

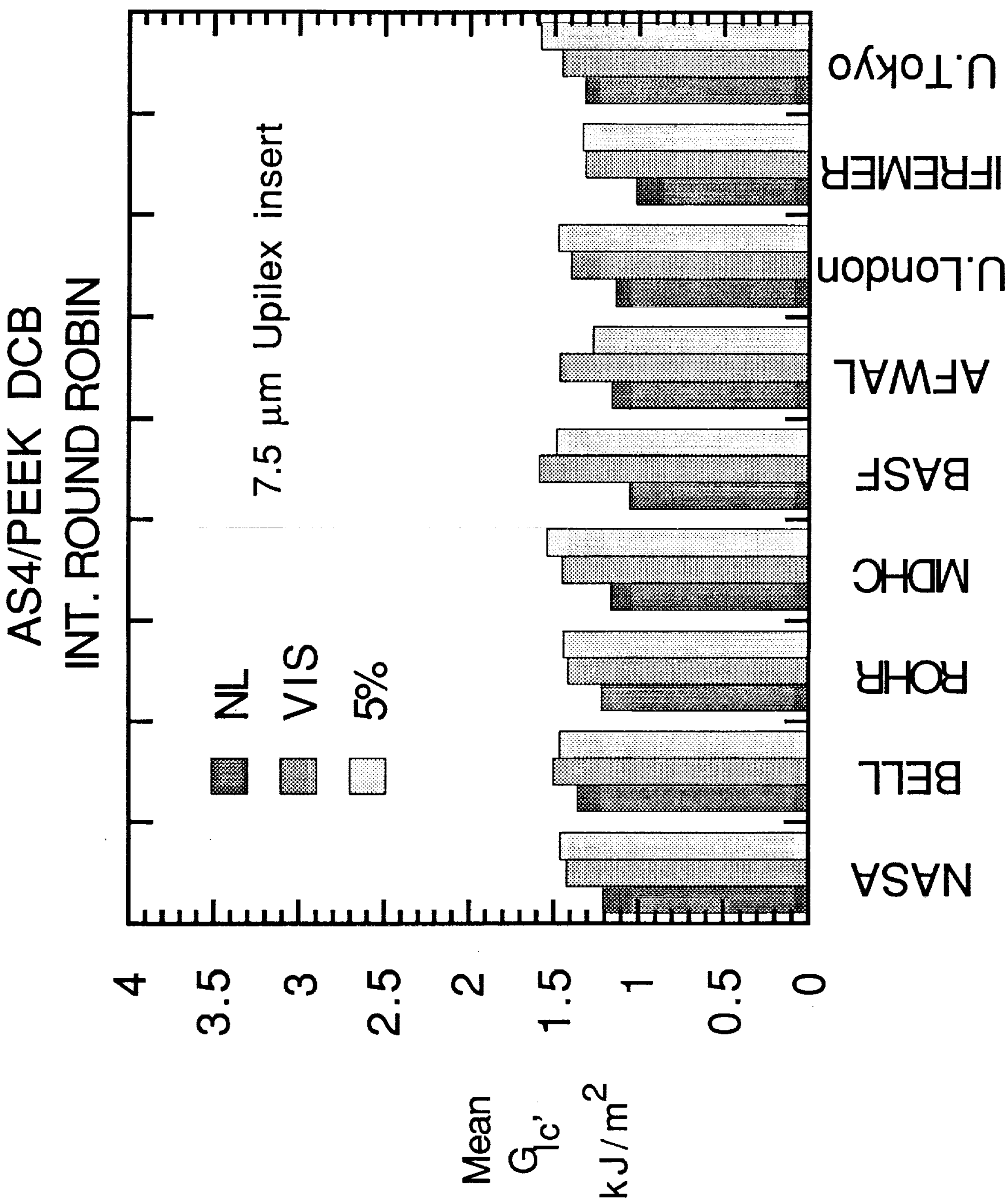

AS4/PEEK DCB INT. ROUND ROBIN
13 MICRON UPILEX INSERT
NL G_{Ic},
kJ/m^2
3
2.5
2
1.5
1
0.5
0
9 LABS
44 TESTS
NL
VIS
5 %

AS4/PEEK DCB INT. ROUND ROBIN
7.5 MICRON UPILEX INSERT

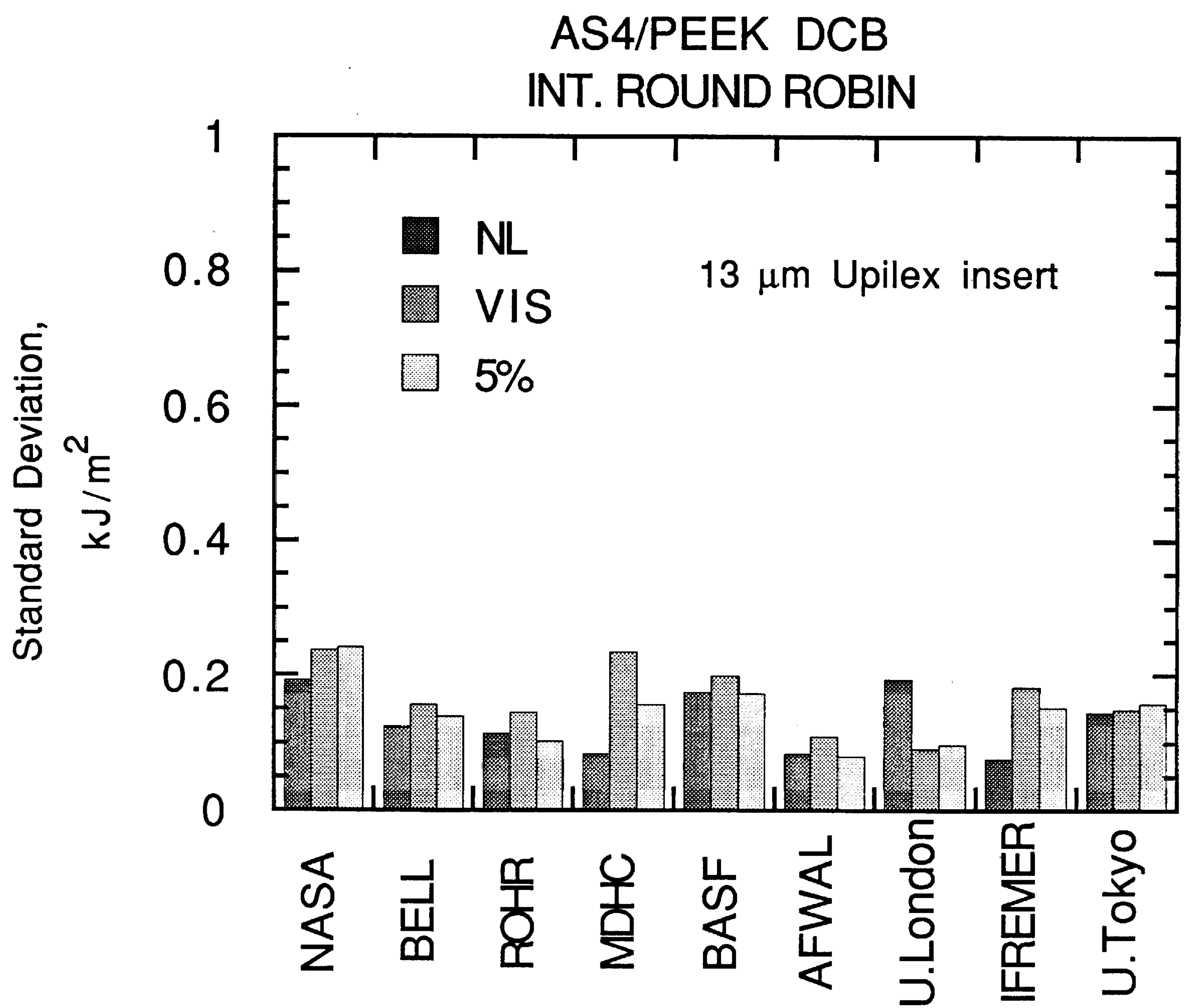


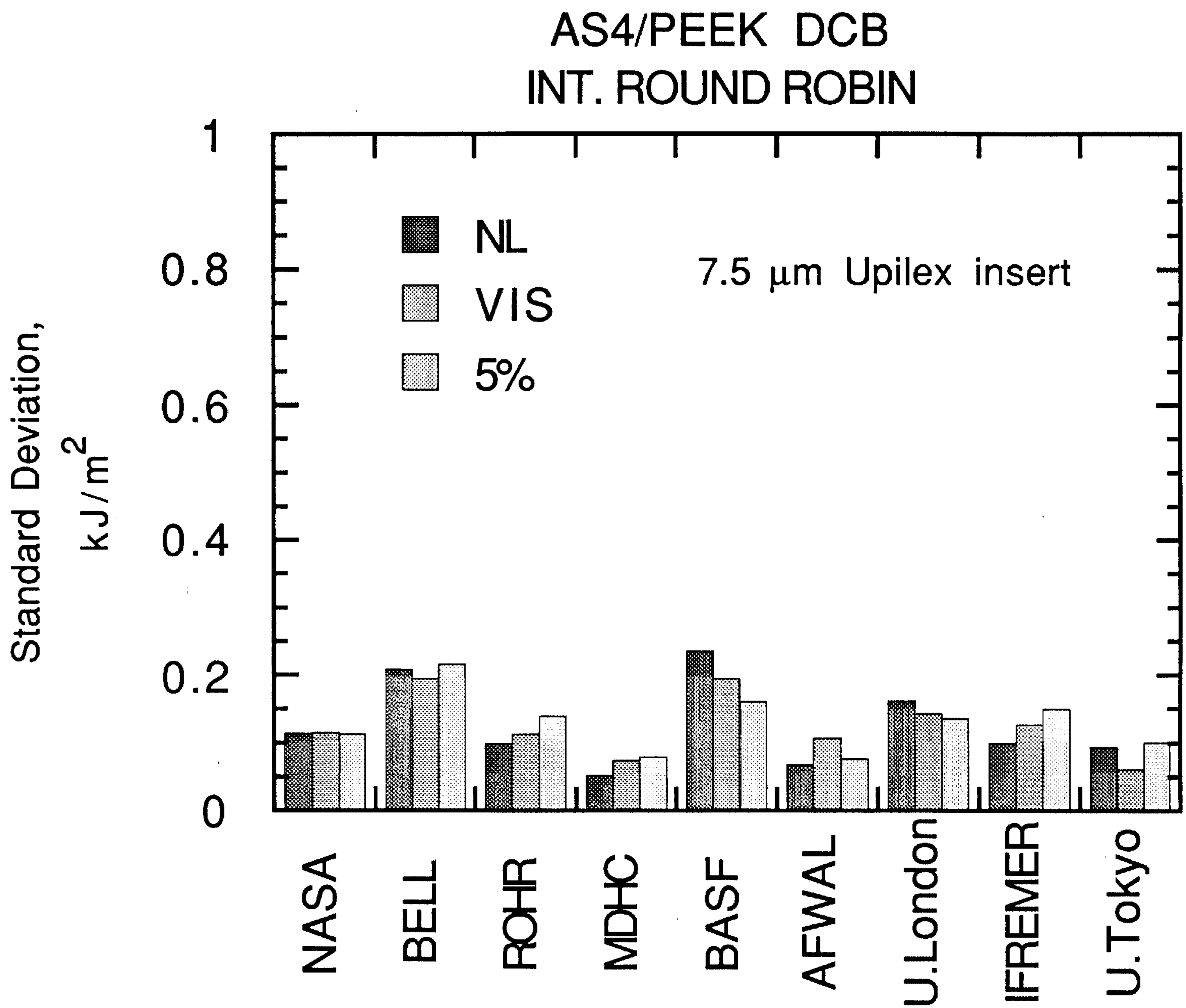


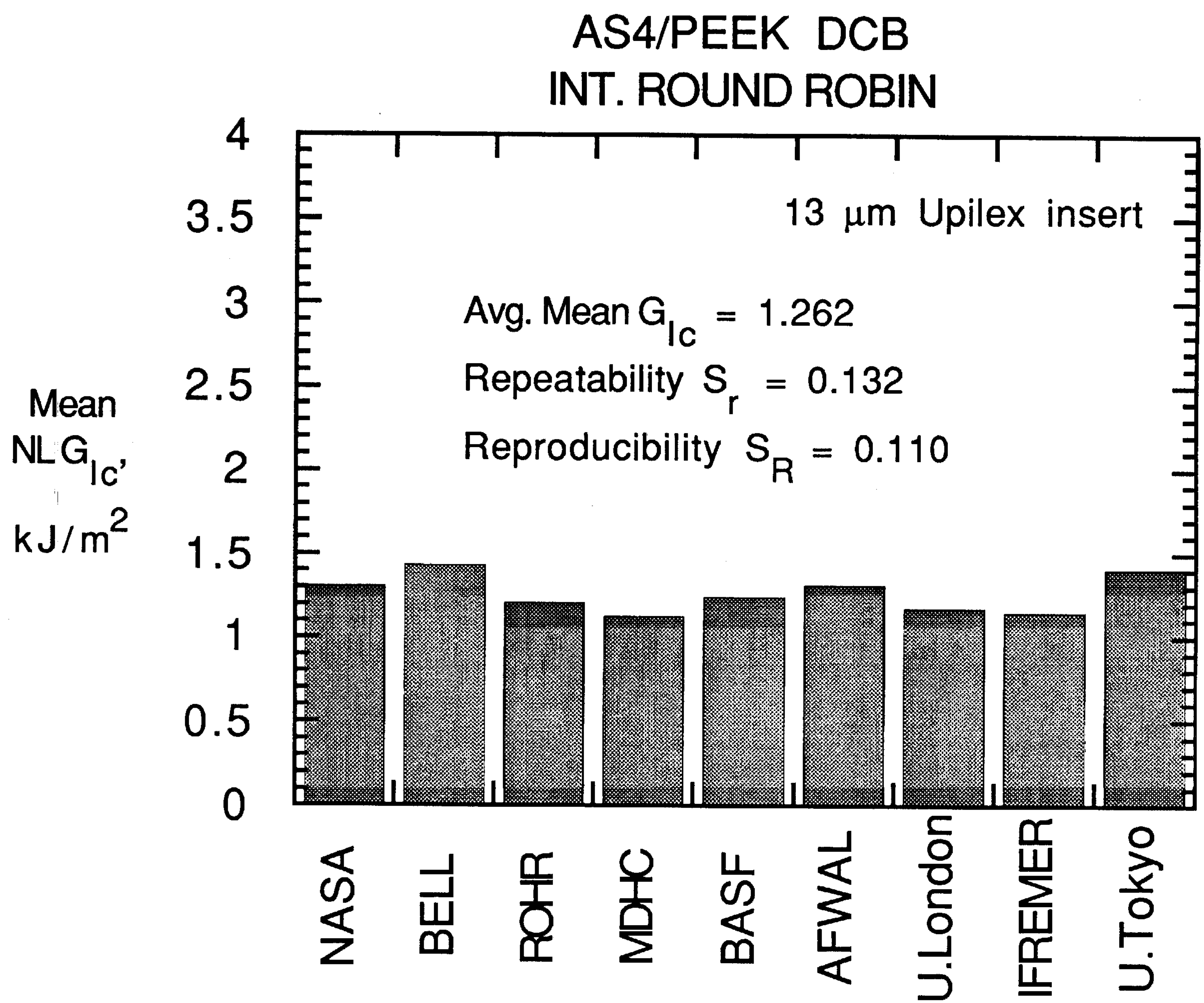
AS4/PEEK DCB
INT. ROUND ROBIN
13 μm Upilex insert
NL
VIS
5%
Mean
G_{Ic}'
kJ/m²
0
0.5
1
1.5
2
2.5
3
3.5
4
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo



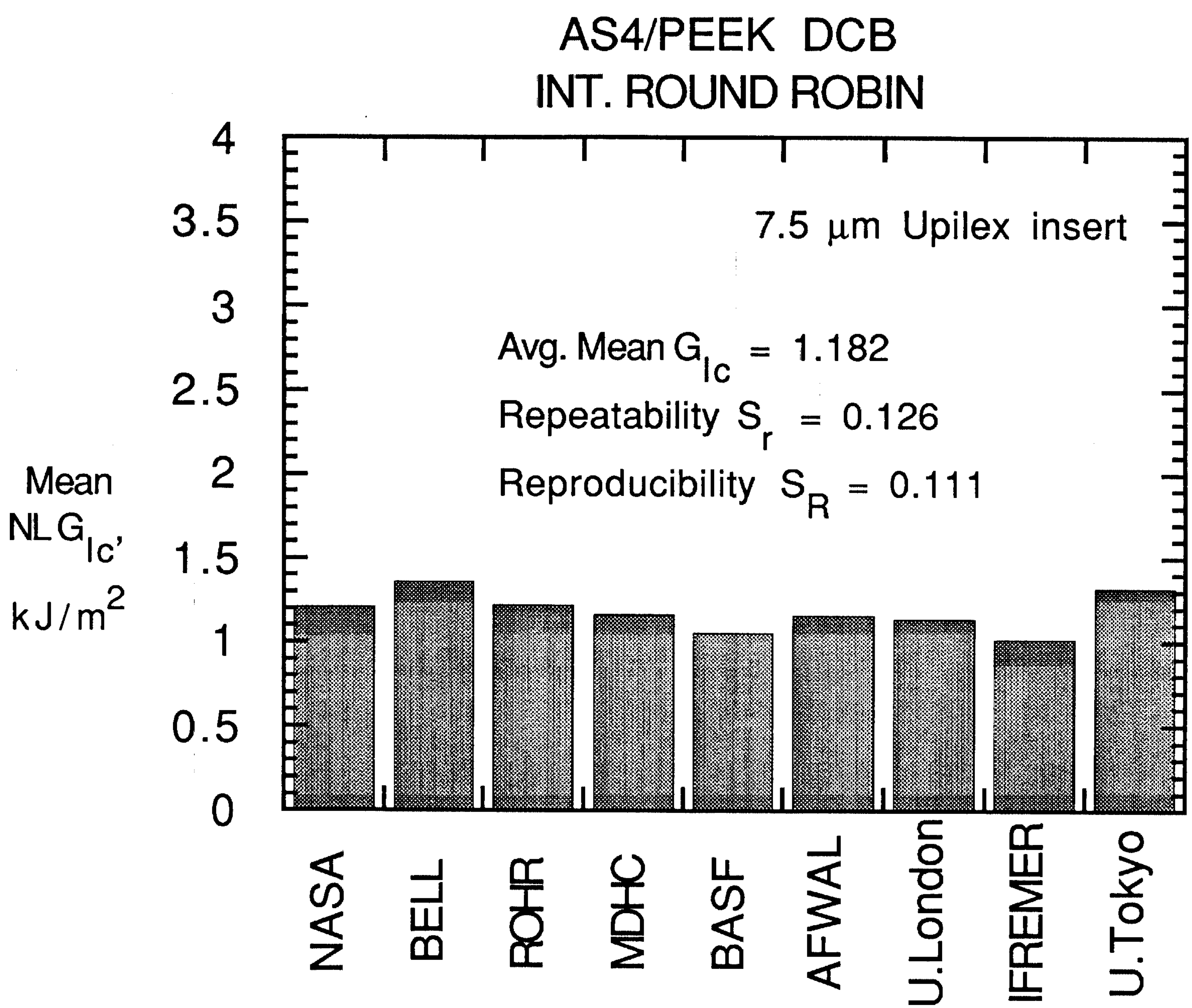
AS4/PEEK DCB
INT. ROUND ROBIN
7.5 µm Upilex insert
NL
VIS
5%
Mean
G_Ic'
kJ/m²
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo

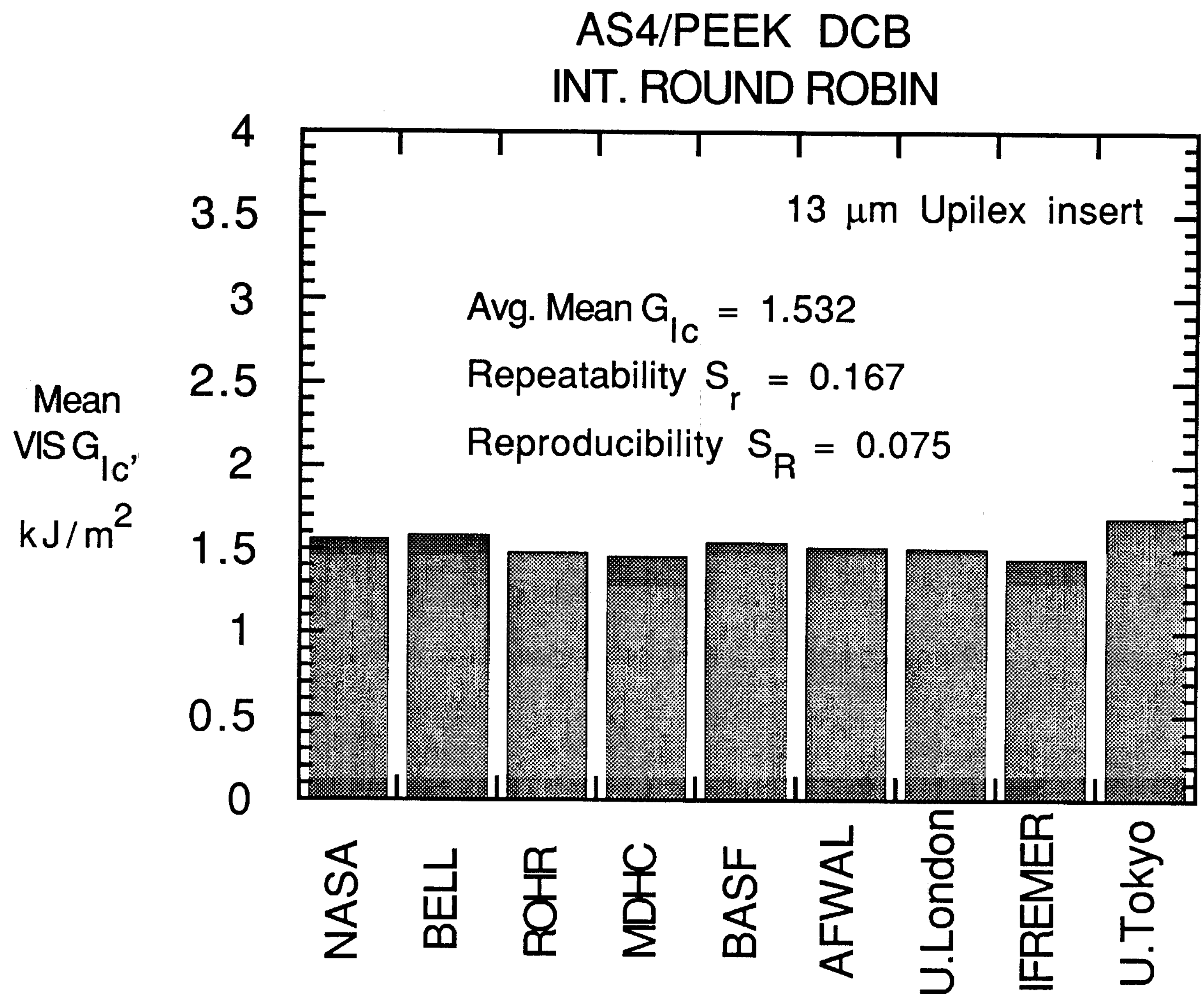

AS4/PEEK DCB
INT. ROUND ROBIN
Standard Deviation,
kJ/m2
1
0.8
0.6
0.4
0.2
0
NL
VIS
5%
13 μm Upilex insert
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo


AS4/PEEK DCB
INT. ROUND ROBIN
NL
VIS
5%
7.5 µm Upilex insert
Standard Deviation,
kJ/m2
1
0.8
0.6
0.4
0.2
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo

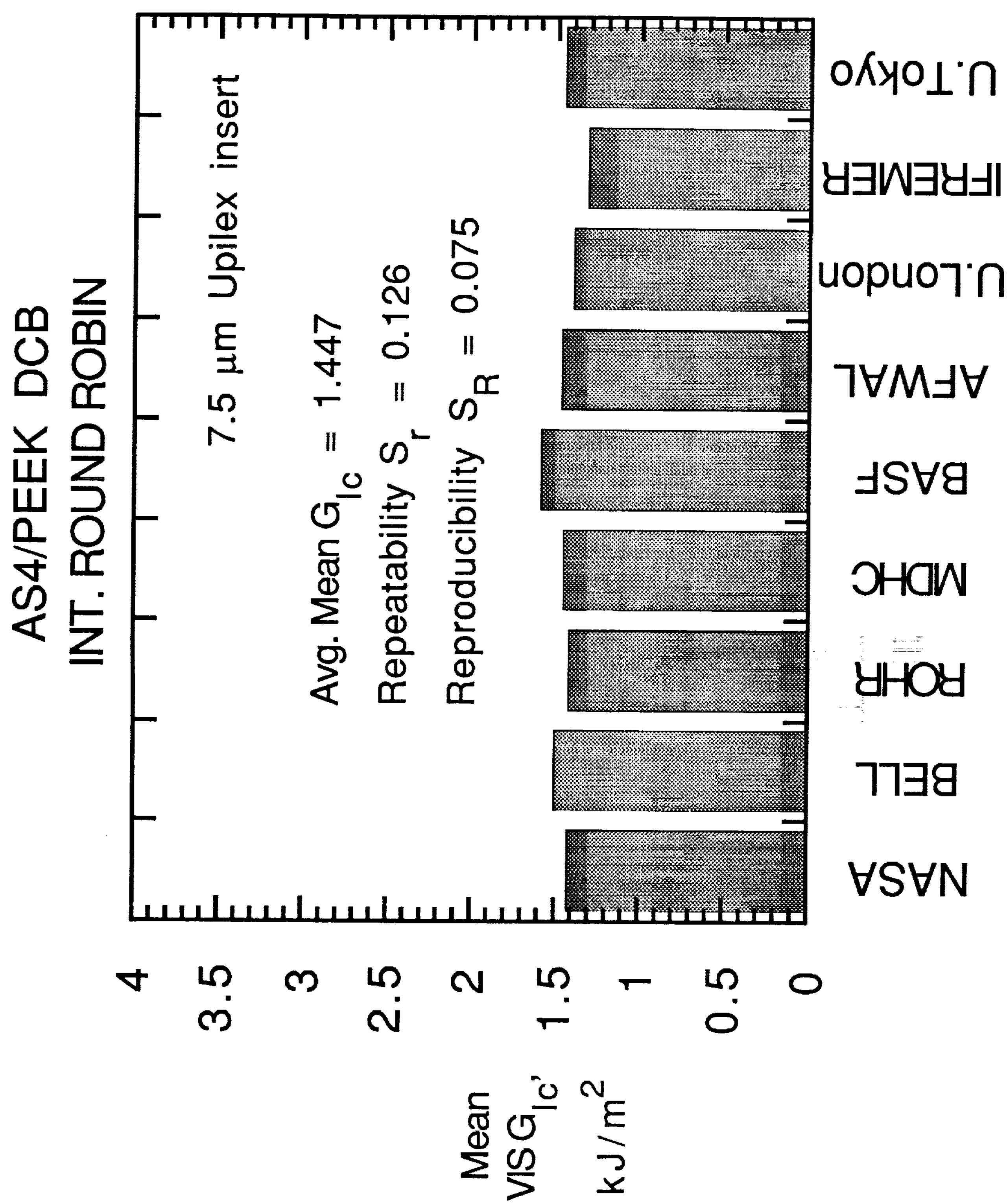


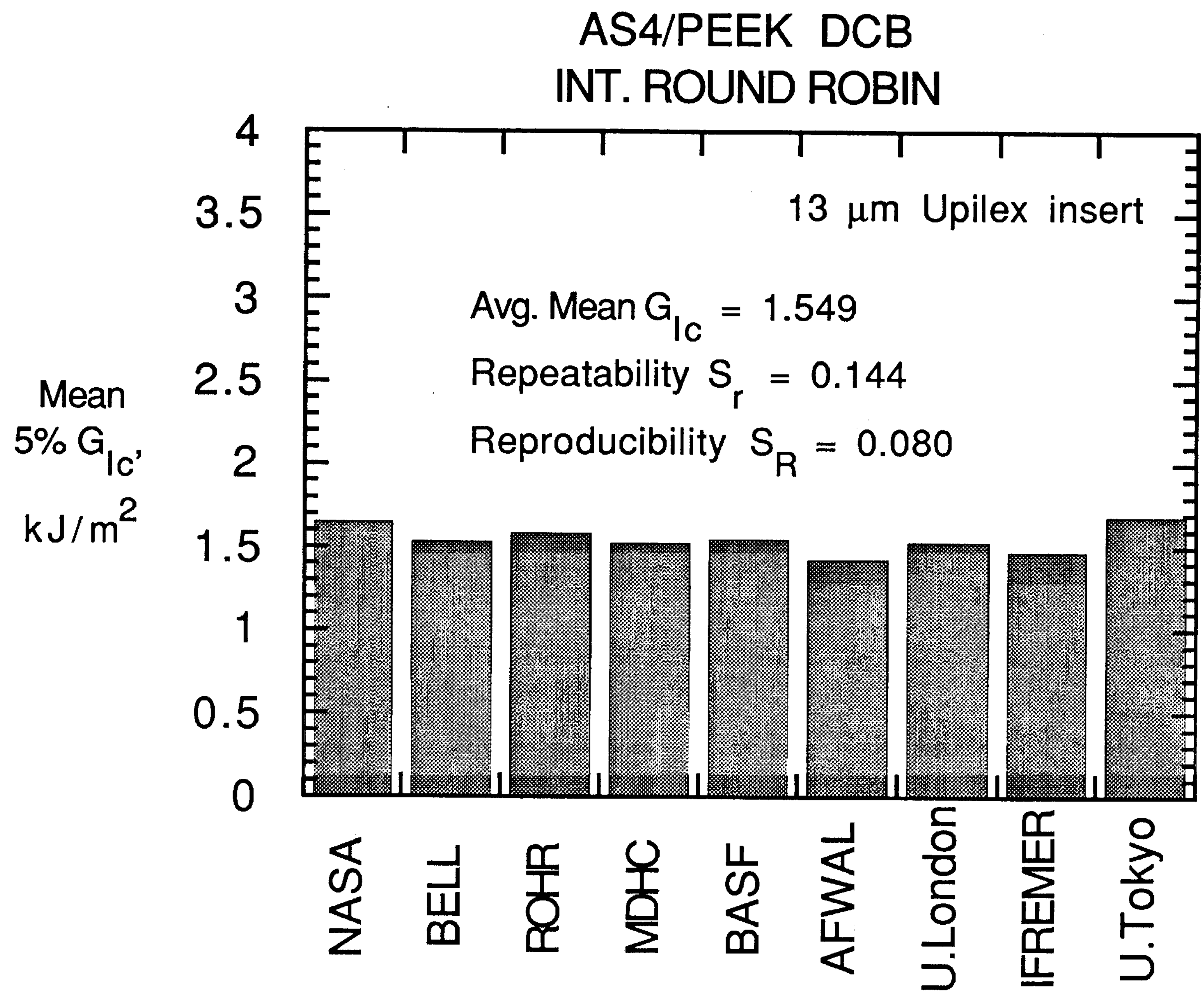
AS4/PEEK DCB
INT. ROUND ROBIN
13 μm Upilex insert
Avg. Mean G_{Ic} = 1.262
Repeatability S_r = 0.132
Reproducibility S_R = 0.110
Mean
NL G_{Ic}',
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo

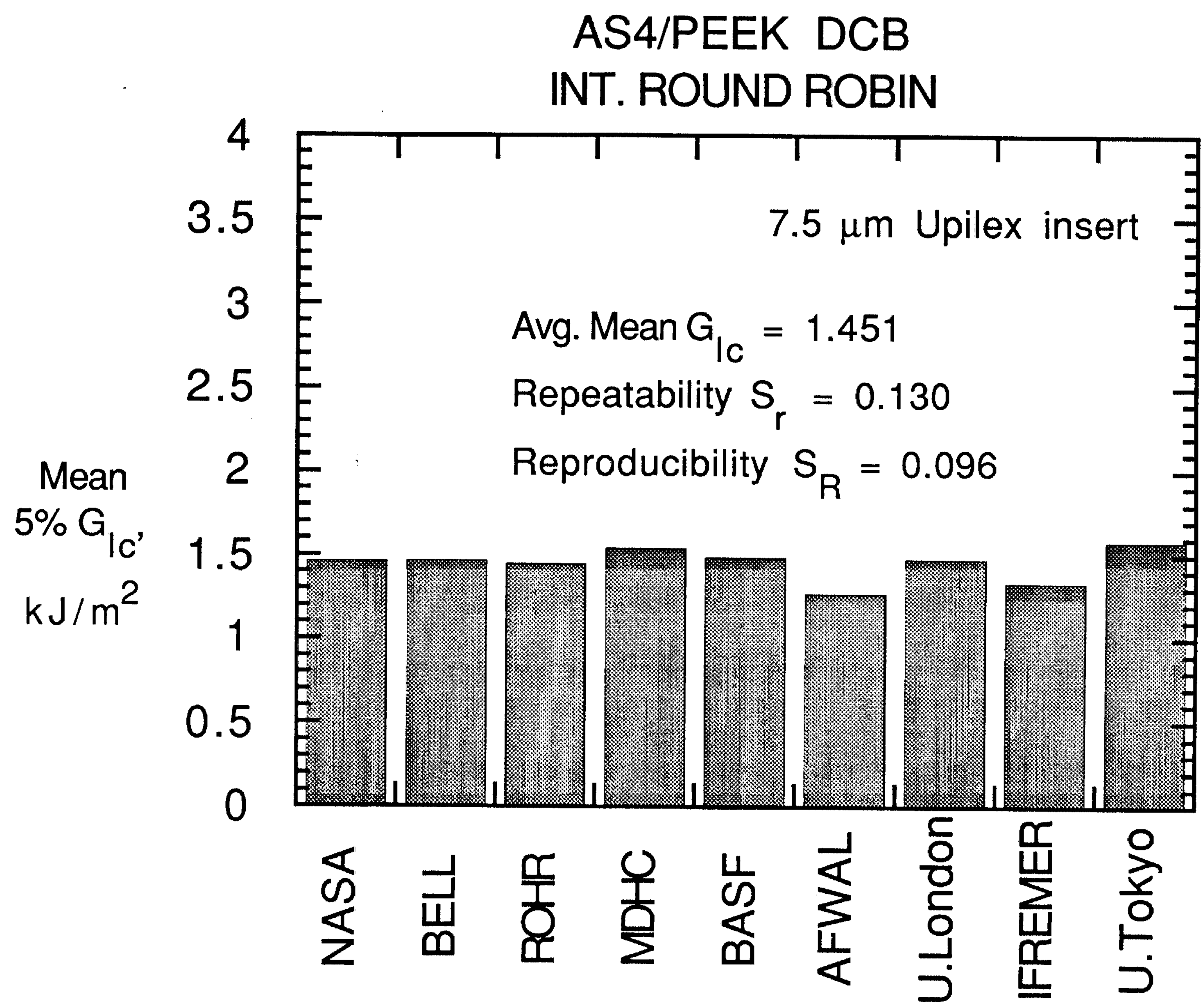

AS4/PEEK DCB
INT. ROUND ROBIN
7.5 μm Upilex insert
Avg. Mean G_{Ic} = 1.182
Repeatability S_r = 0.126
Reproducibility S_R = 0.111
Mean
NL G_{Ic},
kJ / m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo



AS4/PEEK DCB
INT. ROUND ROBIN
13 μm Upilex insert
Avg. Mean G_Ic = 1.532
Repeatability S_r = 0.167
Reproducibility S_R = 0.075
Mean VIS G_Ic, kJ/m²
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo


AS4/PEEK DCB
INT. ROUND ROBIN
7.5 μm Upilex insert
Avg. Mean G_{Ic} = 1.447
Repeatability S_r = 0.126
Reproducibility S_R = 0.075
Mean VIS G_{Ic}, kJ/m^2
0
0.5
1
1.5
2
2.5
3
3.5
4
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo


AS4/PEEK DCB
INT. ROUND ROBIN
13 μm Upilex insert
Avg. Mean G_{Ic} = 1.549
Repeatability S_r = 0.144
Reproducibility S_R = 0.080
Mean
5% G_{Ic},
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo



AS4/PEEK DCB
INT. ROUND ROBIN
7.5 μm Upilex insert
Avg. Mean G_{Ic} = 1.451
Repeatability S_r = 0.130
Reproducibility S_R = 0.096
Mean
5% G_{Ic},
kJ/m^2
4
3.5
3
2.5
2
1.5
1
0.5
0
NASA
BELL
ROHR
MDHC
BASF
AFWAL
U.London
IFREMER
U.Tokyo

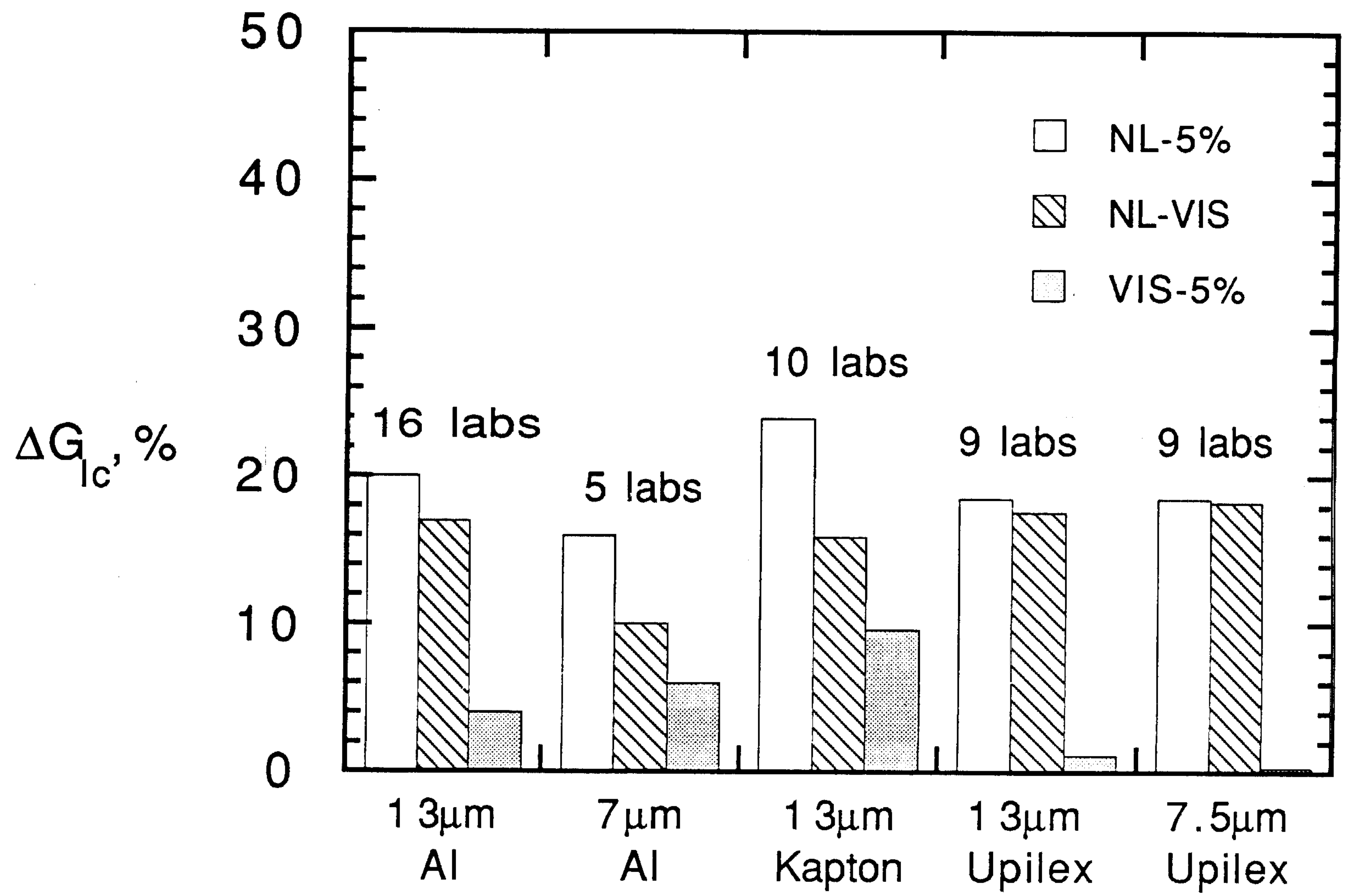


Figure 75. - AS4/PEEK Variation in Delalamination Onset G_{Ic} Measurements

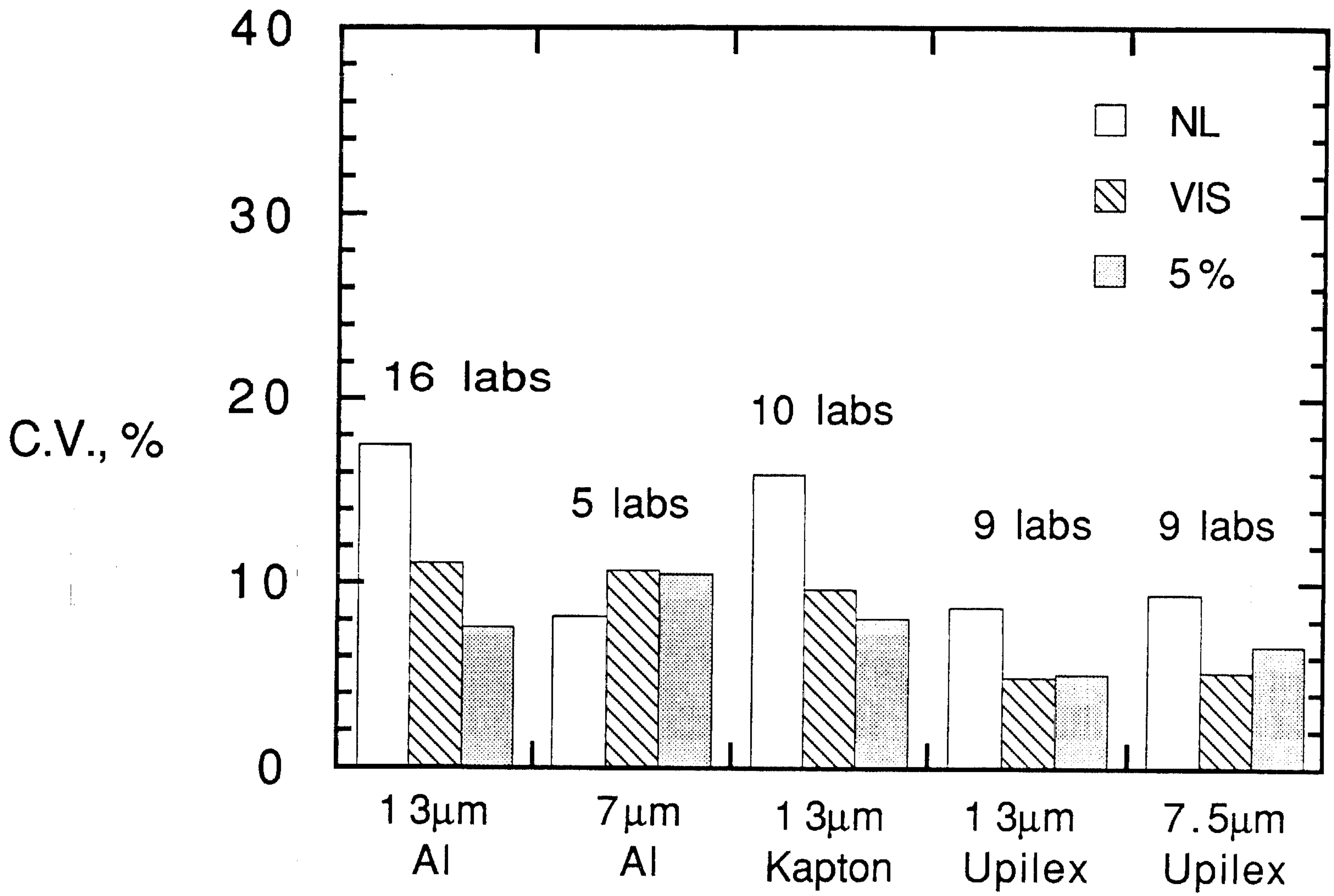


Figure 76. - AS4/PEEK Round Robin Coefficients of Variation

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	November 1992	Technical Memorandum

4. TITLE AND SUBTITLE
Results of ASTM Round Robin Testing for Mode I Interlaminar Fracture Toughness of Composite Materials

5. FUNDING NUMBERS
WU 505-63-50

6. AUTHOR(S)
T. K. O'Brien and R. H. Martin

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
NASA Langley Research Center
Hampton, VA 23681-0001
and
Aerostructures Directorate, U.S. Army-AVSCOM
Langley Research Center, Hampton, VA 23681-0001

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546-0001
and
U.S. Army Aviation Systems Command
St. Louis, MO 63120-1798

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TM-104222
AVSCOM TR-92-B-013

11. SUPPLEMENTARY NOTES
O'Brien: U.S. Army Aerostructures Directorate , NASA Langley Research Center, Hampton, VA; Martin: Analytical Services & Materials, Inc., Hampton, VA

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified - Unlimited

Subject Category 24

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

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 D30 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.

14. SUBJECT TERMS
Composite materials; Double cantilever beam; Interlaminar fracture toughness; Delamination; Standard test method

15. NUMBER OF PAGES
121

16. PRICE CODE
A06

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified		