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Mode I delamination fatigue crack growth in unidirectional fiber reinforced composites: Development of a standardized test procedure

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Selected mode I fatigue data from five different types of fiber-reinforced, polymer–matrix composites tested in two round robins organized by the American Society for Testing and Materials subcommittee D30.06 and European Structural Integrity Society Technical Committee 4, respectively, are analyzed and discussed. The focus is on experimental scatter (in-laboratory and inter-laboratory) and on schemes for quantitative data analysis. It is shown that in spite of considerable scatter different composites can be distinguished and, under certain assumptions, a relative ranking be established. Further, effects from limited experimental measurement resolution are noted and implications for the test procedure and use of the test data in design of composite structures discussed. For comparative purposes, a rough ranking of different composites is feasible with test data generated within 24 h per specimen in an industrial test environment.

1. Introduction

Characterization of delamination resistance of fiber-reinforced polymer–matrix composites (FR-PMC) under quasi-static and cyclic fatigue loads is important for development of laminates with improved damage tolerance as well as for designing composite structures and, therefore, has been an active area of research for quite a while (see e.g., [1,2] for a summary). While this has resulted in standardized test procedures or at least in standardization activities for quasi-static loading in different modes [3], this is not the case for cyclic fatigue yet. A procedure for mode I tensile opening cyclic fatigue testing of FR-PMC was evaluated in a first round robin test involving three laboratories and was shown to yield sufficiently reproducible results [4] and additional testing showed sufficient discrimination between selected, different types of FR-PMC [5]. Recently, additional round robin testing on mode I cyclic fatigue was performed, both within subcommittee D30.06 on interlaminar properties of composite materials of the American Society for Testing and Materials (ASTM) International and committee TC4 on fracture of polymers, composites and adhesives of the European Structural Integrity Society (ESIS). The present contribution evaluates selected results from these round robins and focuses on approaches for quantitative data analysis on one hand, and on experimental scatter and measurement resolution on the other. Implications for the test procedure under development will be discussed.

2. Experimental

2.1. Materials and specimens

The materials used in the two round robin programmes are listed in Table 1 for ASTM D30.06 and in Table 2 for ESIS TC4. All tests were performed using double cantilever beam (DCB) specimens (according to ASTM D5528 or ISO 15024) with a total length of about 152 mm (ASTM) and of 145 mm (ESIS), a width of 20 mm and thicknesses as listed in Tables 1 and 2. Starter cracks were realized by using a PTFE film insert (about 5 μm for ASTM and about 20 μm thick for ESIS) at the laminate mid-plane. Aluminum load-blocks (about 6 mm thick, 12.8 mm long and 20 mm wide, with a pin-hole with a diameter around 3.8 mm located towards the crack tip for ASTM, and 10 mm thick, 15 mm long and 20 mm wide with centered pin-hole with a diameter of 4 mm for ESIS) were mounted for load introduction.

2.2. Procedure

The ASTM test procedure first asked for drying the specimens according to ASTM D5229/D5229M and then storing them in a

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Table 1
Materials used in the ASTM D30.06 round robin.

	C1	C2	G1
Fiber	Carbon, IM7	Carbon, G40-800 12k	Glass, S2
Matrix resin	Epoxy, 977-3	Epoxy, 5276-1	Epoxy, 5216
Lay-up	$[0]_{26}$	$[0]_{26}$	$[0]_{18}$
Thickness (mm)	3.5 (nominal 3.5)	3.4 (nominal 3.7)	4.1 (nominal 4.0)

Table 2
Materials used in the ESIS TC4 round robin.

	C3	C4
Fiber	Carbon, G30-500 12k	Carbon, AS4
Matrix resin	Epoxy, rigidite 5276	PEEK
Lay-up	$[0]_{24}$	$[0]_{24}$
Thickness (mm)	4.0	3.0

desiccator before testing under standard laboratory conditions of (23 ± 3)°C and (50 ± 10)% relative humidity. The mode I fatigue test shall be performed without precracking at 10 Hz (if possible); at laboratory B tests were run with 3 Hz due to large displacements and with a R-ratio of 0.1. Testing shall be continued until a delamination rate below 10^{-6} mm/cycle is reached in order to determine a threshold value. For determining the maximum cyclic displacement δ_{max}, a series of quasi-static tests shall be performed on a separate set of nominally identical specimens up to a delamination length of 75 mm beyond the initial crack tip. The quasi-static results shall be used to calculate δ_{max} from the following relation (1):

$$\frac{\delta_{max}^2}{[\delta_{cr}]_{av}^2} = \frac{G_{Imax}}{G_{Ic}} = 0.8 \tag{1}$$

$$\text{, i.e., } \delta_{max} = \sqrt{0.8[\delta_{cr}]_{av}^2} \tag{2}$$

where G_{Ic} is the critical strain energy release rate, $[\delta_{cr}]_{av}^2$ is the average of the squared critical displacement values at G_{IC} from the quasi-static tests and G_{Imax} is the maximum cyclic strain energy release rate. Effectively, at laboratory B, a factor of 0.8 proved to be too low to get the delamination propagation started from the initial crack within a sufficiently low number of cycles and a factor around 1.0 was used for most tests. The quasi-static data are summarized in Table 3. One laboratory also tested additional specimens for delamination growth onset according to ASTM D6115 based on developments reported in [6].

The ESIS procedure specified that in order to start the measurements at high crack growth rates just below G_{IC} a quasi-static mode I test for pre-cracking was done as a G_{IC}-test at a cross-head speed between 1 and 5 mm/min. The displacement value at which pre-cracking was stopped was then taken as the δ_{max} value for fatigue loading. The cyclic test was started and continued until a crack growth rate of about 10^{-6} mm/cycle was reached. An R-ratio of 0.1 was used in all the measurements and the tests were done under displacement control. Fatigue loading could be stopped to perform visual observation of delamination lengths with a traveling microscope. In order to avoid errors during the calculation of the results a spreadsheet macrofile was created which was used by all the participants for the calculation of da/dN and G_{Imax}. Data from visual observation of delamination propagation and from recorded machine compliance can be used to evaluate G_{Imax} and the corresponding delamination rate da/dN. G_{Imax} is evaluated using simple beam theory, corrected beam theory and modified compliance calibration and the da/dN-values are calculated in a secant (point-wise) approximation or with a seven point polynomial fit (according to ASTM D647). The number of machine compliance data points can be reduced by specifying a minimum delamination length increment between subsequent data points in the data presentation graphs (typically 50 μm was used). The same spreadsheet was used to analyze the ASTM round robin data from laboratory B. A typical set-up for mode I fatigue testing (laboratory B) is shown in Fig. 1.

Table 3
Quasi-static mode I averages from the ASTM D30.06 round robin (laboratory B, five specimens each).

	C1	C2	G1
Initiation G_{IC} (NL) ± standard deviation (J/m^2)	120 ± 9.7	253 ± 23.8	147 ± 11.8
Initiation G_{IC} (MAX/5%) ± standard deviation (J/m^2)	145 ± 12.1	290 ± 11.8	172 ± 11.9
Average propagation ± standard deviation (J/m^2)	174 ± 16.5	337 ± 27.1	432 ± 27.7
Maximum propagation ± standard deviation (J/m^2)	184 ± 17.2	357 ± 34.2	574 ± 26.9
Back-calculated E-modulus ± standard deviation (GPa)	122 ± 5.8	142 ± 12.5	59 ± 13.3

3. Results and discussion

3.1. ASTM round robin

Only data from laboratory B are available for the analysis and hence, no information on inter-laboratory scatter can be derived. On the other hand, as shown in Fig. 2, the in-laboratory scatter of the data from visually recorded delamination lengths using a traveling microscope is such that it is somewhat difficult to even distinguish trends among the three laminates. This is different from the data presented in [5] where various carbon- and glass FR-PMC could easily be distinguished. It has to be noted that G_{Imax} values are determined from the modified compliance calibration method (MCC, see [7]). As a second step in the analysis, the same data are plotted using delamination lengths calculated from recorded machine compliance data (machine load and displacement, respectively) [4] instead of visually determined delamination lengths (Fig. 3). Since machine data have been recorded every 500 cycles, the number of data points is reduced by requiring a minimum delamination length increment of 50 μm between individual data points in the spreadsheet used for data analysis. A 50 μm delamination length increment roughly corresponds to the resolution of the visual delamination length measurement obtained with the microscope at laboratory B. This allows distinguishing differences in slope of the Paris plots for each group of laminate. In a next step, the data points for each specimen are replaced by a power law fit, $\frac{da}{dN} = A \cdot G_{Imax}^m$, which yields a quantitative result, namely the exponent m, which represents the slope of the linear regime in the double logarithmic diagram. It has to be noted that these fits include all data points and hence may be affected by possible deviations from linear behavior at both, high and low delamination rates. In a further step, the power law fits for each material are presented individually, this time including the non-linear (NL) and maximum or 5% (MAX/5%) initiation values from quasi-static testing of specimens of these laminates (again determined by the MCC method). From the four graphs in Fig. 4, the following conclusions can be drawn: (1) laminates C1 and G1 show a larger scatter in slopes than laminate C2, (2) laminate C2 clearly shows the lowest slope, while C1 and G1 seem to yield similar slopes, (3) laminates C1 and C2 show at least one fit with a distinctly different slope among the five data sets, and (4) for laminate