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Review of delimitation resistance testing of polymer-matrix composites

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Abstract: *The development of fracture mechanics test methods for the determination of delimitation resistance or fracture toughness of fiber-reinforced, polymer-matrix composites is an active area of research. The emphasis in this review is on standardization of test methods. Recent developments leading towards new standardized test procedures will be presented, complementing and updating earlier reviews.*

Keywords: *fracture toughness, delimitation resistance, polymer-matrix composites, test procedure, standards, review.*

1. INTRODUCTION

The status of the development of test methods for fracture mechanics properties of fiber-reinforced, polymer-matrix composites (FRPC) around the millennium has been reviewed by Davies et al. [1] and Tay [2]. The present contribution will focus on more recent developments and provide an up-date on these earlier reviews. For the ease of comparison, it will be organized similar to the review by Davies et al. [1]. The emphasis will be on experimental work, i.e., the development of test methods that have been recently standardized, are currently being considered for standardization, or are under development with the aim of proposing them as future standard test methods. There is also extensive research on theoretical developments and modeling in fracture mechanics of FRPC, but a review of these aspects is beyond the scope of the present paper. Modeling aspects are discussed extensively by Tay [2].

It is worthwhile to briefly consider for which purposes delimitation resistance data from various laminates can be used. As outlined in [1], comparative assessment of different formulations of matrix materials with respect to improved toughness for materials development, the determination of damage tolerance of composite structures that was perceived to correlate with delimitation resistance, and the need to establish critical energy release rates for structural design and calculations are major reasons for the development of standardized test methods. The latter is still limited to a few applications, even though the principal methodology has been established [3]. Quality control in manufacturing or failure analysis of composite parts and structures, in principle, do constitute other areas of application of the standards. While the former may be implemented at some manufacturers (e.g., those publishing data sheets with fracture mechanics values), the latter is rarely used and, to the best of our knowledge, not documented in the literature.

In the area of delimitation resistance testing, the concurrent development of test methods for composites and adhesives by the Technical Committee 4 (TC4) Polymers & Composites of the European Structural Integrity Society (ESIS) is fairly recent. This recognizes the fact that the major problems are the same or analogous in various polymer-based materials. In this respect, it will also be worthwhile to follow the development of test methods for “natural” composite materials, such as wood (see, e.g., [4]). A detailed description of the test procedures developed for FRPC within ESIS TC4 and data determined with these procedures up to 2001 are available in [5].

In the following, the status of delimitation resistance test method development is briefly reviewed and recent developments are discussed. The sequence of sections will follow the mode of loading (mode I = opening, mode II = shear, mode III = twisting, various mixed modes) and each section be organized according to the loading-rate (quasi-static, fatigue, high-rate).

2. MODE I TESTING

A mode I (opening) test method for delimitation resistance has finally been accepted as an international standard by ISO in 2001 [6]. The test method published by the American Society for Testing and Materials (ASTM) in 1994 [7] is, in a revised version issued in 2001, technically equivalent to the ISO test. The mode I standard test method issued in 1993 together with a mode II procedure by the Japanese Standards Association [8] is similar, but not identical to the ISO and ASTM procedures. For details on the earlier approaches, the reader is referred to [1].

The preferred specimen type in most mode I tests is the so-called Double Cantilever Beam (DCB), schematically shown in Fig. 1. A non-sticking, thin film insert acts as starter crack (recommended to be less than 13 µm thick). Tensile load to open the starter crack and to promote delimitation through the mid-beam interlaminar layer is introduced via two load-blocks at quasi-static cross-head displacements between 1 and 5 mm/min. Delimitation lengths are determined visually during the test, the use of a travelling microscope for more accurate delimitation length readings is optional, but recommended. The data analysis is either based on beam theory (with corrections for load-blocks and large displacements) or on experimental compliance calibration [6].

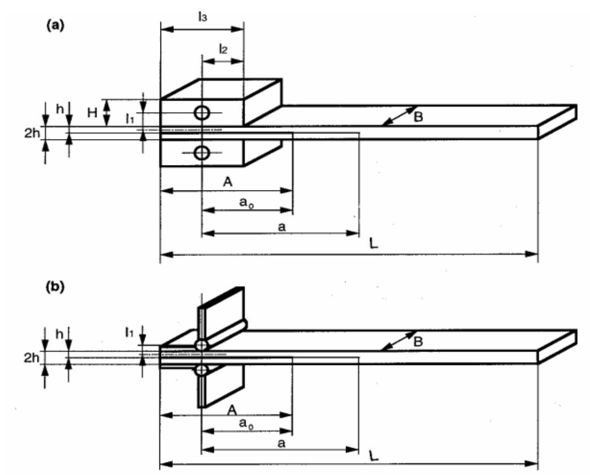

Figure 1: Schematic of the Double Cantilever Beam specimen used for Mode I testing, load blocks (a) or piano hinges (b) can be used for the load introduction [6].

Delimitation resistance testing of unidirectional fiber-reinforced composites from man-made fibers has, with the exception of glass- and carbon-fibers, not yet been investigated in round robin studies and hence not been standardized. The same holds for composites made from natural fibers. A priori, there is no obvious reason why composite laminates with other fiber types could not be tested according to the mode I ISO procedure, as long as size and stiffness criteria [6] are fulfilled. Even though most literature on fracture toughness or delimitation resistance testing of laminates with other types of fibers [12-16], i.e., not carbon or glass, does not reference any of the standard test methods, the methodology in many cases is equivalent. It can hence be concluded that the standard test method [6] is applicable to composites reinforced with other, unidirectional aligned fibers.

The applicability of the standard DCB specimen for delimitation resistance testing of laminates with multidirectional lay-up has been assessed by Choi et al. [20], and more recently by Morais et al. for carbon- and glass-fiber laminates [21, 22]. Multidirectional lay-ups frequently pose problems because of crack branching and/or deviations of the delimitation from the central plane. Both effects invalidate the analysis according to the ISO standard [6]. Delimitation resistance from DCB-test on multidirectional laminates can probably be quantified for initiation only. No significant dependence on the delaminating interface (fiber orientation) was observed [22].

Even for cross-ply composites (alternating 0° and 90° orientations stacked on top of each other), extensive round robin testing yielded about 50% of invalid tests due to deviation from the mid-plane [23]. Typically, the delimitation path oscillates between adjacent 0° plies (Figure 2). These specimens yielded initiation values similar to those observed in the corresponding unidirectional laminate and much steeper resistance curves [23], but higher initiation values have been reported also [24]. This increase in apparent delimitation resistance does not scale with the increase in delimitation area and the mechanisms are not fully clear yet [25]. The observations that initiation values for cross-ply and multidirectional laminates are comparable to those in unidirectional laminates and apparently do not depend on the type of delaminating interface could be due to the observation (made in the case of cross-ply laminates) that the delimitation has to propagate a certain distance (around 0.5 to 1 mm, Fig. 2 edge view), before the oscillating pattern forms [25].

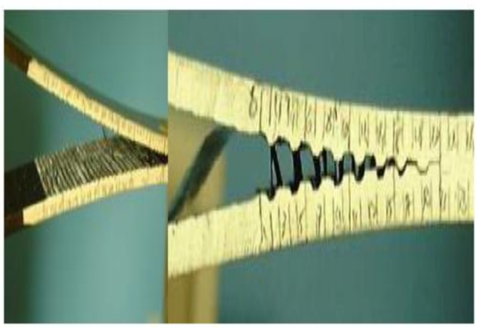

Figure 2: Typical delimitation path observed in cross-ply (alternating 0°/90° layers) laminates, oscillating between adjacent 0° plies. (Left): view onto fracture surface. (Right): edge view. If delimitation paths deviate into the 0° plies tests are considered invalid.

Clearly, lay-ups with different fiber orientations of the laminate are preferred over unidirectional orientation in most applications and determining experimental data, e.g., for design, still seems to be problematic. Frequently, unidirectional laminates are considered to represent a lower bound but this assumption may discard a considerable part of the design potential of specific multidirectional laminates.

The ASTM D6115 procedure constitutes a first step towards standardization of other types of loading, namely mode I fatigue [26]. Constant amplitude tension-tension fatigue loading at various G-levels is used to determine delimitation growth onset in unidirectional fiber-reinforced laminates. The procedure is based on a limited data base from unidirectional carbon fiber tape laminates with single-phase polymer matrix, as stated in the scope [26]. Fatigue delimitation propagation, however, is not considered in this procedure. Within ESIS TC4, there are plans for round robin testing towards a standard test procedure for fatigue delimitation propagation with parameters based on published research [27, 28]. A model for the effect of stress ratio on fatigue delimitation under mode I loading has been developed by Andersons et al. [29]. Fatigue life models based on experimental data have also been investigated (e.g., [30, 31]).

Ballistic impact loading is performed at even higher rates (10 m/s and higher) but typically on plate specimens or parts without starter defects. Damage mechanisms are quite complex. Delimitation initiation and growth do occur [34] but quantitative assessment of delimitation resistance does not seem feasible from such experiments.

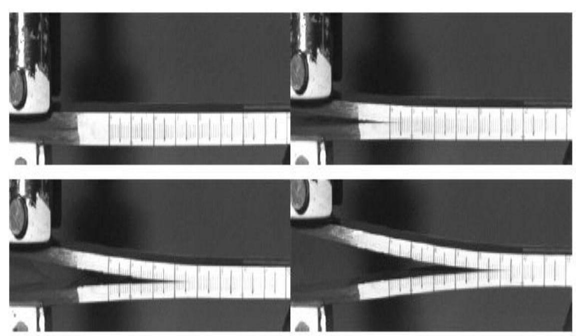

Figure 3: A series of frames selected from a high speed video sequence recorded at 4000 frames per second. The time interval between each frame shown here is 5 ms (in the film sequence, the frame interval was 250μs). The photographs are from a DCB-test at 1 m/s and even at this relatively moderate loading-rate, an asymmetry is introduced.

3. MODE II TESTING

Currently, there exists one Mode II standard test [8] based on the three-point bending end notch flexure (ENF) specimen, since the European Aerospace procedure (prEN6034) is still at the draft stage. Early round robin work on mode II ENF had been conducted jointly by JIS, ASTM and ESIS but, contrary to the situation in mode I, had not resulted in international consensus [1]. Several factors contributed to that. First, the ENF-test is essentially unstable and thus allows only determination of initiation values but not of resistance curves. Second, the question of friction contributions was raised and this resulted in the question whether mode II data were to be regarded as apparent values with no significance as materials data [35].

The four point end notch flexure (4ENF) test was proposed by Martin and Davidson in 1997 [16] and appeared to resolve many of the mode II testing problems. It offered three significant advantages, stable crack propagation, a simple test fixture and a straightforward data analysis. It was evaluated in two round robins organized by VAMAS, involving ASTM, ESIS and JIS participants [17, 18]. In the first, values of GIIc were compared to values from other mode II tests. In the second a more detailed study of the 4ENF specimen was carried out. There were some anomalous results recorded in these two exercises, notably an apparent decrease in GIIc with increasing span length, Figure 4.

Another observation from early results was that the 4ENF tended to give values of GIIc significantly higher than those from 3ENF specimens [19].

Nevertheless, overall, and compared to the three other mode II tests examined (ENF, ELS and SENF), the 4ENF specimen appeared satisfactory. Stable propagation was observed, allowing compliance calibration to be made during the test, and results from different laboratories were reasonably consistent (Figure 5).

The test was subsequently used in a number of studies. For example, data analyses were discussed in more detail by Schuecker and Davidson [20]. Results from tests on glass/epoxy specimens were also published [21].

The situation in 2003, therefore, seemed clear and it appeared to only be a matter of time before the 4ENF specimen would become the standard mode II test method. However, Davidson and colleagues continued to work on the test, in particular trying to understand and explain differences between 3ENF and 4ENF test results. They examined in more detail, for both test configurations, the influence of four factors:

- loading roller diameter
- specimen geometry
- friction, and
- fixture compliance.

Their conclusions were presented to the ASTM D30 committee in 2004 [22] and are currently being published. First, the roller diameter will contribute to non-linearity as the contact point's move and there is a specimen shortening effect. This will be more pronounced in the 4ENF than in the ENF test as the shortening effect comes from both inner and outer rollers. A second related effect is the change in vertical moment arm. Davidson and Sun [23] showed that these effects will cause the ratio of non-linear to linear beam theory values of GII to decrease below one at low loads then to increase above one as loads increase. The specimen geometry (span length) will also affect these non-linearity's, but for typical dimensions and roller diameters with carbon/epoxy materials these effects are likely to affect the measured results by less than 5%.

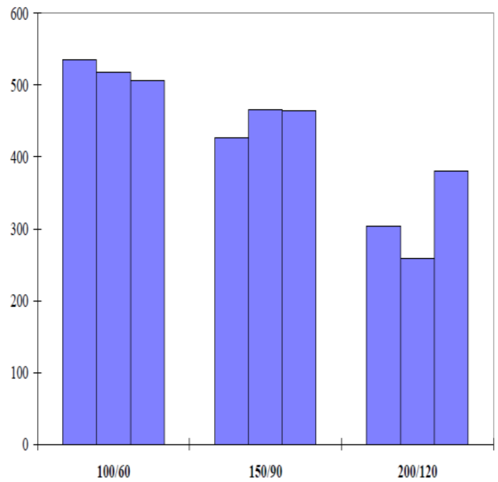

Figure 4: Influence of specimen geometry (lower span/upper span) on initiation values of GIIc (in J/m²) from mode I precrack, brittle carbon/epoxy specimens 3 mm thick, results from K. Tanaka, University of Toyama, 2nd VAMAS Mode II round robin tests.

The influence of friction in the 3ENF test had been examined numerically previously by Carlsson et al. [24] who concluded that for values of friction coefficient between 0 and 0.5 the influence on GIIc values would be less than 5% for most cases. For the 4ENF specimen Davidson et al. measured friction coefficient values using a variable wedge fixture which allowed them to increase the wedge angle until samples of half specimens started to slide. They obtained values of 0.374 and 0.345 for two carbon/epoxy materials. When these values were included in numerical analyses it was observed that friction would have a slightly larger influence in the 4ENF specimen than in the 3ENF, but its influence was still minor. It was therefore apparent from these analyses that while all these factors influence the accuracy of results they were not sufficient to explain some of the large differences reported between GIIc values from 3ENF and 4ENF tests.

The final aspect examined was fixture compliance. Tests were performed on aluminum bars of different geometries to calibrate the two fixtures. FE analyses then enabled the influence of the fixture compliance to be determined and it was clearly shown that fixture compliance played a very significant role in GIIc determination. It was also shown that the 4ENF specimen is more sensitive to this, due to the need to include an upper roller bearing. A fixture compliance calibration will be essential if this test is to be used in a standard, but such calibrations are already standard for other tests.

Mode II 4ENF propagation

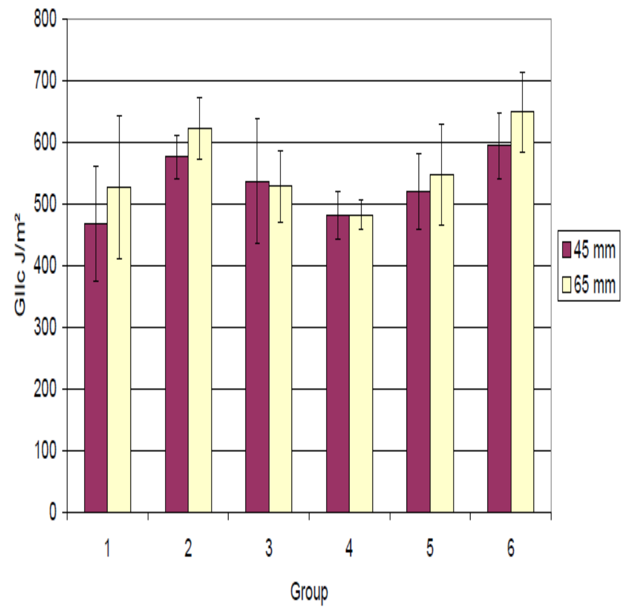

Figure 5: Propagation values measured by 6 groups at two crack lengths for carbon/epoxy,

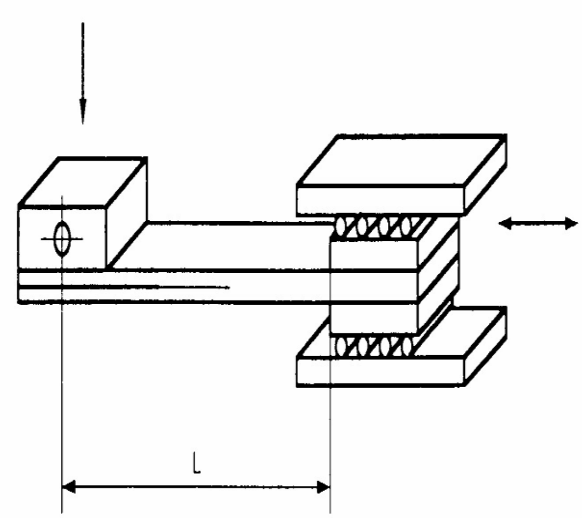

Figure 6: Schematic of the End Loaded Split test set-up for mode II delimitation resistance testing.

Mode II tests of multidirectional glass- and carbon-fiber laminates and their analysis, complemented by extensive modeling are reported in the literature [27-29]. A dependence on the angle of the delaminating interface was observed, with increasing values of GIIC for increasing angle of fiber orientation. Also, a comparison between initiation values from a starter film and from mode II pre-crack showed significant resistance curve effects, i.e., higher GIIC values from the pre-crack.

4. SUMMARY AND OUTLOOK

Summarizing the current status of standardized tests for determination of the delimitation resistance of fiber-reinforced polymer-matrix composites (FRPC), significant progress within the last five to eight years can be noted. First there are now standardized test methods published by recognized organizations for a number of different cases. Considerable research has been performed investigating issues related to either different type of lay-up or to different types of loading. Specifically, the important questions of rate-dependent and fatigue behavior of laminates are currently being addressed by committees dealing with the development of standardized test methods.

Progress has also been made with respect to analysis and interpretation of the data. Several test methods now offer spreadsheets for data analysis, including various correction factors and offering the opportunity to compare different approaches for analyses (e.g., beam theory based versus experimental compliance). Currently, the exact measurement of the delimitation length has become a focus of attention and recent models may provide operator-independent determination of effective crack lengths. Modeling efforts may help to further improve understanding of various factors affecting the measurements. While not currently being considered for standardization, development of procedures for determining the fracture toughness of parts or elements made of FRPC has also advanced. On the other hand, few examples of the application of fracture mechanics data in design are available in the published literature.

Developing suitable testing and analysis procedures for the determination of the delimitation resistance of multi-directional laminates under quasi-static and fatigue loading in the different modes remains a challenge.

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