TOWARDS A STANDARD FOR INTERLAMINAR FRACTURE TESTING OF COMPOSITES.

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This paper describes current efforts to develop a standard test method for the determination of the interlaminar fracture resistance of composite materials. The activities of the EGF Polymers and Composites task group are outlined and the principal problems hindering the drafting of a standard are presented. Finally the status of a joint European/US/Japanese round robin exercise involving some fifty participants is described.

INTRODUCTION.

At the present time a large number of test laboratories, both industrial and academic, perform tests to determine the interlaminar fracture resistance of fibre reinforced composite materials. The most common tests are carried out on the mode I double cantilever beam (DCB) and on the mode II end notch flexure (ENF) specimens (1). Data from such tests are used for a number of purposes ; e.g. screening of new materials, studies of fibre and matrix behaviour and prediction of damage tolerance. In spite of the popularity of such tests, no widely accepted standard exists. However, this lack of standardization has not been due to neglect but more to certain fundamental problems which will be discussed later.

For the determination of basic mechanical properties such as moduli and strengths the composites industry suffers from too many rather than too few standard test methods.

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These methods originate from a number of sources. For example :

National organizations (BS, DIN, AFNOR, ASTM etc.)
Trade organizations (SACMA, ETAC)
Aerospace groups, (CRAG, ACOTEG, EFA)
Individual companies (Boeing, Northrop etc.)

While many of the basic standards are very similar there are strong economic reasons for their rationalization within a single framework. The initiative of the European Community (EN, European Standards) thus offers a welcome opportunity to establish a single set of standards for composite materials.

As far as interlaminar fracture testing is concerned a number of groups are active. An ASTM task group has been studying interlaminar fracture tests since 1981, and has gathered a certain amount of data from round robin activities. A NASA document published in 1982 included a mode I test based on values measured during propagation of a delamination (2). More recently first drafts of standards have been proposed for DCB and ENF tests. In Europe, a group of aerospace companies assembled the CRAG (Composites Research Advisory Group) test methods in 1985, and these included a mode I DCB test (Curtis (3)). This method was also based on values measured during propagation, as was a recent DIN proposal. Unfortunately however, a number of published results have indicated that propagation values may be dependent on specimen stiffness e.g.(4) and this has been one of the major factors which have hindered the development of a standard. The ASTM group is therefore now tending towards the use of initiation values for mode I characterization, but this has not been without problems and is the subject of a current round robin exercise described below.

Two other groups have examined interlaminar fracture tests recently. The first, VAMAS (Versailles Agreement on Materials and Standards) is studying a number of different aspects of composite testing, having started with mode I and mode II tests and currently concentrating on creep and fatigue. The second is the Polymers and Composites group of the European Group on Fracture and their activities will now be described.

EGF POLYMERS &COMPOSITES TASK GROUP

This group was set up in 1985 and first developed a draft standard for polymer Kc testing. However a very active composite activity was soon established and some 30 laboratories from 12 countries have participated in round robin tests to date (see Acknowledgements).

Round robin tests. The mode of operation of the task group has been to perform tests on a common material and then to meet twice a year to discuss the results. Three round robin exercises have been completed, on the following unidirectional materials, kindly supplied by Ciba-Geigy and ICI :

Carbon / Epoxy (each time)
Carbon / PES
Carbon / PEEK
Glass / Polyamide
Glass / PU

The first two round robin exercises were described previously, (5). From the experience gained a protocol was drawn up, which was used for the third round of tests.

Third round robin. All groups tested 3 mm thick mode I and mode II carbon/epoxy and carbon/PEEK specimens, and selected groups also tested two other specimen thicknesses, (1.5 and 5.2mm). Initiation and propagation were examined. During the 2nd round robin a modified beam theory approach for data analysis was developed at Imperial College, (6), and this was also studied.

Mode I. Examples of results for the 3mm thick carbon/PEEK specimens are shown in Figure 1. Initiation values show considerable scatter, even within results from a single group, and this is believed to be due to the thickness of the starter film (between 20 and 30 microns). Previous work has indicated that as thin a film as possible should be used to avoid blunting effects, and preferably less than 13 microns (7). It was therefore not possible to judge whether initiation values could be reproducibly measured. The use of initiation values has been advocated as they are seen to represent a conservative lower bound below which no delamination damage is measured. However, a number of points must be clarified if these values are to be adopted to characterise a material.

First, the definition of initiation must be shown to be valid. The results in Figure 1 were determined by noting the point of deviation from linearity of the load- displacement curve. An alternative definition suggested by the ASTM group is the point at which the crack is first seen to move. These procedures are extremely subjective. The point of non-linearity has been correlated with acoustic emission recordings for some materials (8), but its exact position clearly depends on how closely the load-displacement curve is followed. Visual observation is hampered by the curved crack front and by stopping tests it has been shown that initiation occurs at the specimen centre well before a crack appears on the edges. A compromise may be to use a 5% offset value as was adopted in the unreinforced polymers protocol, in spite of the rather arbitrary nature of such a criterion. A second problem with these measurements is their dependence on manufacturing methods. A single foil of 13 microns or less must be placed at specimen mid-thickness during moulding. The waviness and straightness of these films and the potentially harmful effect of a release agent must all be taken into account. The third disadvantage of the initiation value is that the data analyses are least reliable for the first few points of crack propagation where the influence of transverse shear and the stiffening effect of hinges are most important.

Propagation values on the other hand, such as those in Figure 1, are each the mean of 9 or 10 values, and appear quite reproducible for both materials. They are easily measured and avoid all the uncertainties associated with initiation values. However, in this round robin as in previous work a specimen thickness (or stiffness) dependence was noted in the carbon/PEEK composites, mean propagation values rising by about 25% as specimen thickness was increased from 1.5 to 5.2mm (Figure 2). This difference is due to fibre bridging and multiple cracking in the thicker specimens of the tougher material. The relatively brittle epoxy composite gives values independent of thickness.

The two data analyses gave very similar results. Both require curve fitting of compliance (C) as a function of crack length (a). Berry's method uses the slope of a log C-log a plot (8) whereas the corrected beam theory uses an intercept of a $(C)^{1/3}$ v a plot. The advantages of the modified beam theory are its physical basis (the use of an effective crack length), and the possibility of checking the modulus of the material.

Mode II tests were performed on both ENF and ELS (End Loaded Split) specimens (1). There was considerable scatter between results from different groups, Figure 3 and part of this is certainly due to the test procedure. As is common practice mode II delamination was started from a mode I precrack. This is clearly unsatisfactory, as all the uncertainties associated with fibre bridging and multiple cracking in mode I propagation are transferred to the mode II values. Alternatives are to use either thin inserts, with the same inherent problems as described above for mode I, or to introduce a "natural" precrack. The current ASTM preference is for a shear precrack, the main disadvantage of which is the necessity for a separate compliance calibration to determine the initial crack length. (The advantage of the mode I precrack was that its length could be clearly measured on the fracture surface after the test.) The pre-occupation with initiation values is due to the unstable propagation in the ENF specimen whereas the ELS geometry promotes stable propagation and allows a complete R curve to be determined.

Current activities. In November 1989 representatives of the EGF Task Group met ASTM, VAMAS and Japanese Industrial Standards group members. After exchanging data from previous tests it was agreed to conduct a series of tests in common with the aim of developing a single standard acceptable to all parties. At the time of writing (April 1990) materials supplied by ICI and BASF have been sent to some 50 groups, together with a jointly-drafted protocol, (available from the author). Tests on specimens with the the following defects will be conducted :

Mode I (DCB) 7 µm and13 µm films

Mode II (ENF) 7 µm and13 µm films, shear precracks.

It is hoped that these tests will resolve the questions remaining concerning the reproducibility of mode I initiation and mode II measurements. Representatives of the different groups are due to meet shortly before the ECF8 conference, when the results of this joint round robin exercise will be presented.

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Groups marked with an asterix are involved in current testing.

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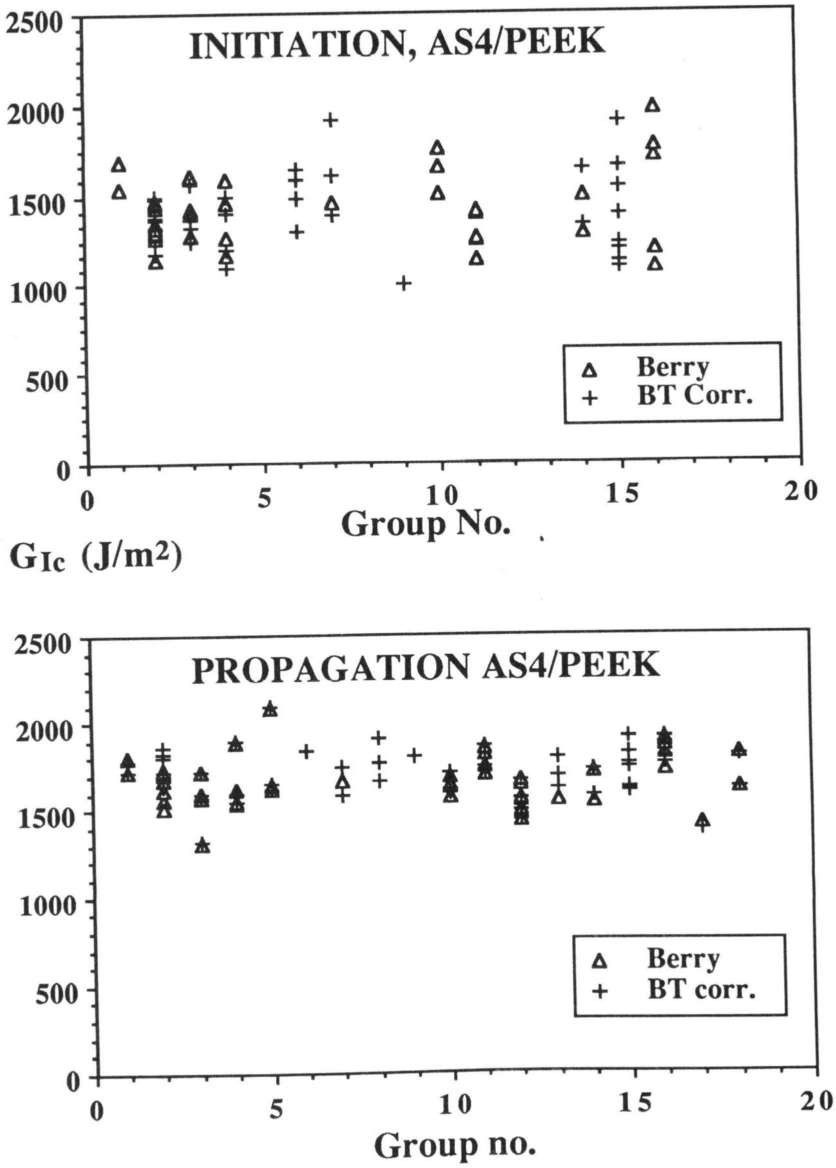

Figure 1. 3rd round robin results, mode I.
3mm C/PEEK specimens.

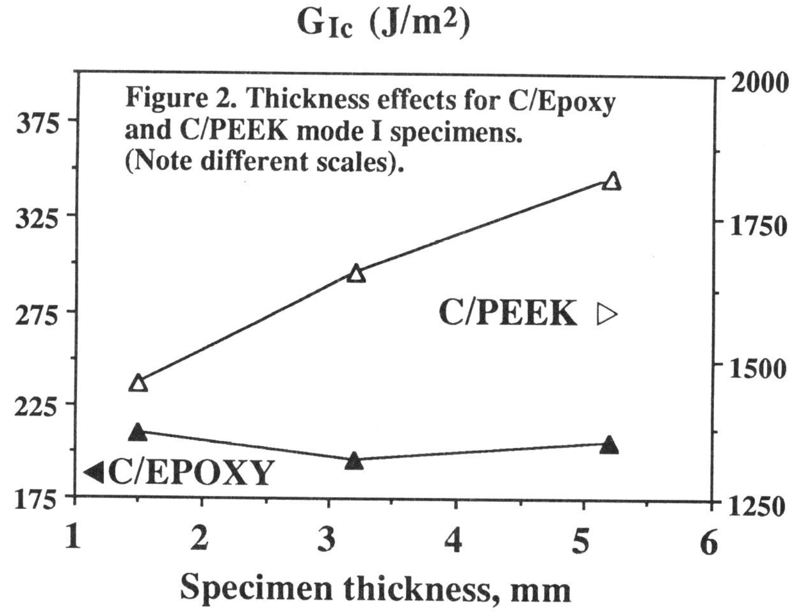

Figure 2. Thickness effects for C/Epoxy and C/PEEK mode I specimens. (Note different scales).

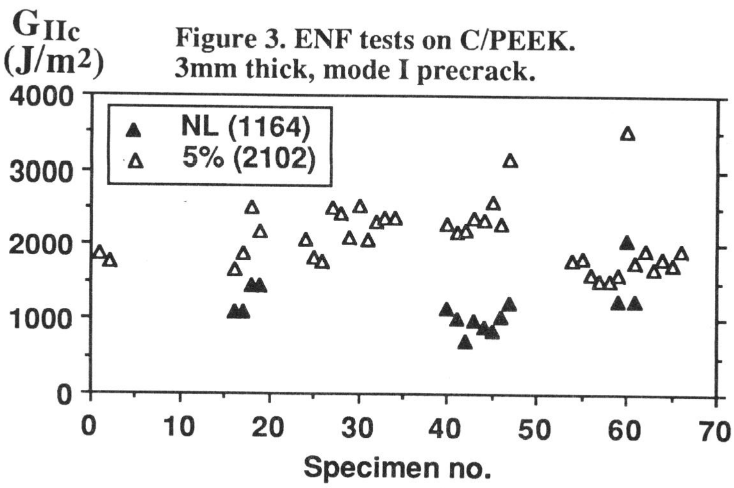

Figure 3. ENF tests on C/PEEK. 3mm thick, mode I precrack.