THE EVOLUTION OF AN ISO STANDARD FOR THE MODE I DELAMINATION TOUGHNESS OF LAMINATES

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This paper reviews the critical stages which have been passed on the road to an ISO standard for mode I delamination toughness testing of fibre reinforced polymer composites. Firstly, the ISO mechanism is reviewed and then the major technical issues relating to mode I delamination toughness testing which were encountered are described. The process by which International Standards are developed by ISO require that international consensus be achieved on the technical content. Over the past four years international consensus on many of the outstanding technical issues has been forged but at the time of writing full consensus has not yet been achieved. The activities and interactions of the three main technical committees involved in this process are appraised.

INTRODUCTION

The double cantilever beam (DCB) test specimen, as shown in Figure 1, has been used for the measurement of the mode I interlaminar fracture toughness, G_{IC}, of composite laminates since the 1960s. Since then there has been much interest in the pursuit of a mode I standard test method and the progress towards standard fracture and fatigue test methods for composite materials was recently reviewed by Davies et al (1). The American Society for Testing and Materials (ASTM) have been investigating the DCB test with a view to standardisation since the 1970s, as reviewed recently by O'Brien (2). The European Structural Integrity Society (ESIS) and the Japanese Industrial Standards (JIS) group have, more recently, joined this endeavour. The ESIS Technical Committee 4 (TC4) on Polymers and Composites started working on the DCB test in 1986, and the three groups, ASTM, ESIS and JIS have each conducted extensive inter-laboratory round robin exercises using this same mode I test. Each group went on to write its own protocol. In 1993, JIS, through the Japanese Standards Association, published a mode I standard based on the DCB test (3). The ASTM balloted and published their version (4) as an ASTM standard in 1994 and in 1995 ESIS TC4 also submitted a DCB protocol (5) to ISO (the International Organisation for Standardisation). The process by which International Standards are developed is now briefly reviewed.

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THE DEVELOPMENT OF ISO STANDARDS

About ISO

ISO is the International Organisation for Standardisation and is a world-wide federation of national standards bodies from some one hundred countries. ISO was established in 1947 to promote the development of standardisation and related activities. The name ISO is not an acronym, but is a word derived from the Greek 'isos', meaning 'equal'. Internationally agreed standards are critical to global industries where technical barriers to trade need to be minimised. The technical work of ISO is carried out in a hierarchy of some 2700 technical committees (TCs), subcommittees (SCs) and working groups (WGs). More information about ISO can be obtained from their Internet site (http://www.iso.ch).

The stages in the development of an ISO standard

The development of an International standard is an evolutionary process, starting from an initial proposal. During this proposal stage, the need for an International standard is identified. A new work item is proposed and is submitted for vote by the members of the relevant TC/SC. The TC/SC then forms a working group consisting of a chairman and a group of experts. During this preparatory stage, successive working drafts are considered until the working group is satisfied that it has developed the best technical solution to the problem. The draft is then forwarded to the working group's parent committee and enters the committee stage. A committee draft is prepared and is registered by the ISO Central Secretariat. At this stage, the committee draft is distributed for comments and voting if required. Successive committee drafts may be considered until consensus has been attained. Once this consensus is attained, a draft International Standard is circulated to all ISO member bodies for voting and comment within five months. This is the enquiry stage. If successfully balloted it becomes a final draft International Standard (FDIS) and goes on to the approval stage. Any further technical comments are now not considered, but are noted for consideration during future revision of the standard. If again successfully balloted, the document goes on to the publication stage and subject to minor editorial changes, becomes an International Standard.

TABLE 1- The stages in the development of an ISO standard

	Stage	Main action	Code used
1	Proposal	New work item is proposed	(NW)
2	Preparatory	Working group develops working draft	(WD)
3	Committee	Consensus on technical content forged	(CD)
4	Enquiry	Draft International Standard is balloted	(DIS)
5	Approval	Text is balloted	(FDIS)
6	Publication	Minor text revision and publication	(IS)

A MODE I DELAMINATION STANDARD FOR LAMINATES

Background

Fracture of composite laminates is covered at ISO by TC61, SC13, WG16. TC61 covers "Plastics", SC13 covers "Composites and reinforcement fibres" and WG16 deals with "Composite materials." TC61 meets annually. A mode I standard for the delamination toughness of composites, G_{IC}, was proposed as a new work item at ISO in 1994. Because the JIS, ASTM and ESIS had all been investigating the test method, effectively three working drafts were prepared (3-5), one by each committee. The technical differences between these drafts reflected partly the differing experiences of the technical committees and partly the different motivating factors which had driven each committee to pursue a standard. The main technical issues and how they were approached by the three groups is reviewed and finally the progress which has been made towards the goal of an International Standard is appraised.

The main technical issues

Limiting the scope to unidirectional $[0°]_n$ reinforcement. Many early studies showed that when multi-directional, cross-ply or woven laminate materials were tested using the DCB geometry, as shown in Figure 1, then multiple cracking and/or crack branching often occurred. This material response clearly precluded a material property from being measured and thus the technical committees focused largely on materials with unidirectional reinforcement, i.e. $[0°]_n$. In addition, this lay-up was expected to yield conservative values of G_{IC} compared to non-unidirectional materials. However, the JIS group included one woven laminate in their pre-standardisation study (6) and ESIS TC4 is currently investigating whether the scope of the current committee draft can be extended to cross-ply laminates (7).

Crack initiation: When does it occur? Detecting the instant of crack initiation in the DCB test was found to be highly elusive. A conservative definition was that initiation occurred at the onset of non-linearity (NL) in the load-displacement trace as shown in Figure 2. Work by Flüeler and Brunner (8) using in-situ microfocus radiography indicated that the NL point coincided with crack initiation at the centre of the specimen. However, use of acoustic emission techniques (8,9) indicated that there were events occurring in the specimen even before the NL point could be detected. The NL definition of initiation has become a common one but is rather subjective: the closer the load-displacement trace is scrutinised, the earlier this point can be detected. An alternative definition of initiation was to quantify a percentage change in compliance which corresponded to a significant but limited crack advance. The JIS group investigated using values from 5% down to 1% for the change in compliance definition and concluded that the 5% definition was the most useful (6). A limiting condition was usually specified that if the intersection of the 5% offset line with the load-displacement trace occurred after the maximum load point, then the maximum load point was taken as the definition of initiation. This definition was thus referred to as the Max/5% point and usually yielded G_{IC} values with less scatter than were obtained from the NL definition however, the values were usually less conservative. A third definition used was to take a value of G_{IC} when the crack was first seen to move with the aid of a travelling microscope or video camera mounted in front of the specimen. However, the degree of scatter in the data obtained from this point was usually larger than from the Max/5% definition, and the point could not be checked after the test.

G_{IC}: Initiation versus the R-curve. Limiting the scope of the working drafts to unidirectional reinforcement did not preclude the occurrence of fibre bridging (fibres bridging the delamination). This is often the result of unidirectional fibres nesting during lay-up and cure. However, fibre bridging results in an increased resistance to delamination as the crack grows and thus a rising resistance curve (R-curve) is often observed, i.e. the value of G_{IC} increases as the delamination grows as shown schematically in Figure 3. As a result of this behaviour it was proposed that minimum or conservative values of G_{IC} would usually be measured at crack initiation. The ASTM technical committee were pursuing minimum values of G_{IC} for structural design and quality control applications and hence they placed most emphasis on initiation values, although their standard (4) did call for the R-curve to be measured. The ESIS and JIS technical committees both considered that the R-curve provided useful additional information on, for example, structural response and both groups considered that the whole R-curve should be measured and that equal emphasis should be given to initiation and propagation values.

Initiation from the insert versus pre-cracking. Perhaps the most insidious of the technical issues, and the one on which consensus has taken the longest to form, was the issue of whether the value of G_{IC} at crack initiation should be measured directly from the film insert, or whether pre-cracking should be used to grow a 'natural' crack ahead of the insert. The initial delamination in the DCB test is achieved by inserting a polymer film at the mid thickness of the laminate and at one end during lay-up. Some early round robin results from the ASTM committee showed that the value of G_{IC} at initiation was dependent upon the thickness of the polymer film insert. Subsequent work by Davies et al (10) using a range of film thicknesses showed that a minimum or limiting value of G_{IC} was attained at a film thickness of 13 microns. One reason for this was shown to be that a resin rich region often forms at the tip of a film insert due to the displacement of the fibres during moulding and cure. A thick film will displace the fibres more than a thinner film and this resulting resin rich region may lead to an artificially high value of G_{IC} being measured. The conclusion was reached therefore by ASTM that initiation from a thin film would yield conservative, lower bound values of G_{IC}. At about the same time ESIS TC4 was running a mode I round robin on a toughened epoxy composite reinforced with carbon fibre. The results obtained were to prove highly significant. Crack initiation from a 13 micron thick film insert failed to produce conservative initiation values of G_{IC}. Instead, unstable crack growth occurred from the insert. However, re-initiating the crack from a mode I pre-crack yielded lower, conservative values. Hence in this case lower bound values were achieved from a mode I pre-crack rather than directly from the insert film. Subsequent round robin testing by ESIS confirmed these earlier findings and the ESIS group concluded that mode I pre-cracking was an essential part of the test. They specified that loading should be continued until the crack was first seen to propagate, then full unloading and re-loading should be performed. The JIS group had also been considering the problem of how to obtain minimum values of G_{IC} at crack initiation. They proposed pre-cracking the samples by clamping close to the tip of the insert and then driving in a wedge and thus growing the crack only a short distance (<5mm). They therefore grew the crack away from the tip of the film insert without introducing significant fibre bridging. ESIS went on to study wedge pre-cracking (11) and found that it was important to ensure that the wedge did not touch the tip of the crack if the creation of additional damage in the zone ahead of the crack tip was to be avoided. Hence the three technical committees each had different views on how crack initiation should be achieved.

PROGRESS TOWARDS AN ISO STANDARD

JIS, through the Japanese Standards Association, published a DCB standard in 1993 utilising wedge pre-cracking (3). The ASTM drafted their test method, D5528-94a, which was balloted within ASTM and passed as an ASTM standard (4) in 1994. In 1995, believing that pre-cracking was important, but that wedge pre-cracking required further study, the ESIS group also submitted a DCB protocol (5). ESIS believed that their document and the ASTM document were not mutually exclusive because the ASTM requirement for initiation from the insert was a necessary step towards creating the ESIS favoured mode I pre-crack. However, the ESIS procedure required full unloading after first initiation which the ASTM did not favour. Both ESIS and JIS favoured pre-cracking. The decision was taken at ISO that the ESIS protocol (5) be accepted as the most promising working document then available, and that this document would be edited by ASTM and submitted through ANSI (the American National Standards Institute) to ISO, first as a working document, and then as a committee draft. A draft was written requiring that initiation first be measured from the insert then, following unloading and reloading, be measured from a mode I pre-crack. Crack propagation points were also to be measured allowing the R-curve to be drawn. Wedge pre-cracking would only be allowed if these two initiation techniques were found to be non-conservative. In January 1996 the new document was written by ASTM. After editing and a further consensus building period, the draft was resubmitted to ISO in June 1996. This document was then accepted as a committee draft and received the ISO designation ISO CD 15024. However, at the ISO TC61 meeting in Montreal in September 1996 it was argued that the draft had become too complex and failed to pass the ballot. A number of editorial changes were then agreed. However, the second ballot in September 1997 did not take place. Consequently, the committee draft has not yet become a draft International Standard. It remains to be seen whether this critical stage will be successfully completed at the TC61 meeting in Whistler, Canada, in September 1998.

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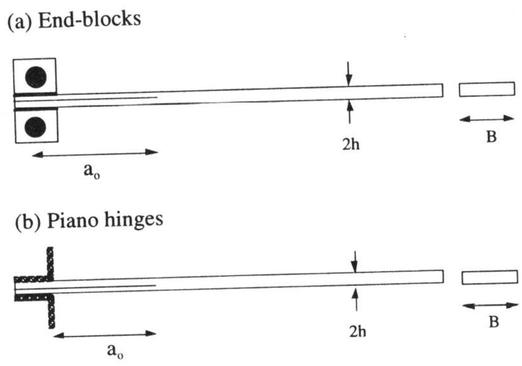

Figure 1 The double cantilever beam (DCB) test specimen with loading via (a) end-blocks and (b) piano hinges. Initial film length is a_o , width B and thickness 2h.

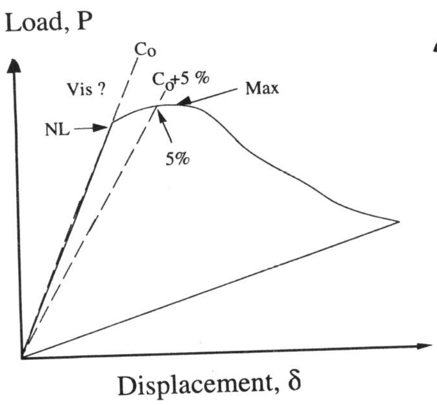

Figure 2 Typical load-displacement trace from a DCB test.

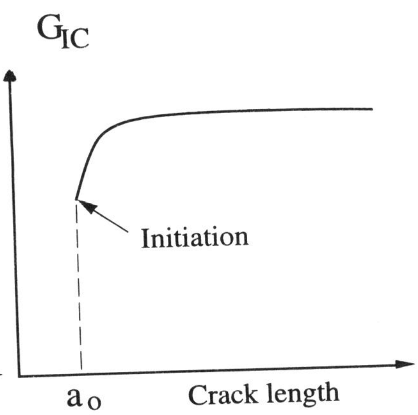

Figure 3 Schematic plot of G_{IC} versus a for a laminate with a rising R-curve.