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35 years of standardization and research on fracture of polymers, polymer composites and adhesives in ESIS TC4: Past achievements and future directions

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Abstract

Since its first meeting in 1985, ESIS TC4 has held regular semiannual meetings with between 15 and 35 participants, has organized a series of conferences (the first in 1994, then triennial since 1999) and has developed six ISO test standards on the fracture of polymers, polymer composites and adhesives with another two currently going through ISO standardization and ballots, and several more under development. The activities have also resulted in publications, including two books and two review papers. Initial activities focused on round robins providing test methods for determination of fracture properties for, e.g., technical data sheets, quality assurance, materials selection, or materials development and optimization and materials modelling. These procedures defined standard specimens, test rigs and test conditions. For polymers, standards for specific ranges of loading rate and for composites and adhesively bonded joints, procedures for different loading modes and mode mixes were developed. Recently, standard composite specimens with unidirectional fiber orientation were shown to overestimate the delamination resistance of multidirectional laminates under cyclic fatigue loading. First round robin data from the environmental stress cracking tests show the potential for discriminating between the different susceptibilities of polymers to environmentally induced fracture. Future activities will include elastomeric materials, simulation and modelling in combination with experiments or prediction of fracture behavior. Another topic of recent interest concerns digital tools, e.g., image analysis, automated data acquisition, data fitting and analysis. Guidelines on how to best reduce extrinsic scatter and eliminate human errors will improve the data quality.

Keywords: Fracture testing of polymers, polymer composites and adhesives; standardizaton; fracture mechanics design; digital tools;

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1. Introduction

This contribution presents the past, current, and envisaged future activities of ESIS TC4 on Fracture of Polymers, Polymer Composites and Adhesives; originally founded as "Task Group on Polymers and Composites" within the European Group of Fracture (EGF). The activities of the committee include the development of fracture test procedures for submission as international standards, and the related fracture research and dissemination of results. The present paper firstly summarizes the achievements of ESIS TC4over the past 35 years, then secondly it focusses on open issues in fracture testing that currently are or will soon become activities within the committee. Some of these questions are for example how fracture data from material tests on composites can be applied to structural design (Jones et al. 2017); how the effects from environmental exposure can be assessed (Kamaludin et al. 2017, Bredács et al. 2018, Contino et al. 2021); and how recent developments in digital technology may contribute to minimizing scatter in the experimental data (Brunner 2020) or speed up design and development of components and structures (Chisholm et al. 2019). Another issue relates to the development of new types of engineering materials based on polymers. Materials for which the applicability of the standard tests has to be assessed are, e.g., foams or aerogels (see, e.g., Banerjee and Sinha Ray 2020, Liu et al. 2020, Lee and Park 2020), self-healing polymers or composites (see, e.g., Wang and Urban 2020), nano-modified polymers (see, e.g., Sankasubramanian et al. 2019), or polymer-based metamaterials (see, e.g., Askari et al. 2020). This question also has relevance for new processing and production methods, such as the additive manufacturing of polymers or of polymer composites, see, e.g., Dev Nath and Nilufar (2020) and Jafferson et al. (2021) that may exhibit new types of defects or defect distributions differing from those from established methods. However, there are also "conventional" engineering materials, such as elastomers, for which no standard fracture test methods yet exist.

Nomenclature

AM	Additive Manufacturing
ASTM	American Society for Testing and Materials, International
BS	British Standard
CD	Committee Draft (*standard)*
CEN	Comité Européen de Normalisation
C-ELS	Calibrated End-Loaded Split (*test-rig*)
DCB	Double Cantilever Beam (*specimen*)
G_{IC}	Critical Mode I Energy Release Rate [*or*] Mode I Interlaminar Fracture Toughness
ECF	European Conference on Fracture
EGF	European Group on Fracture (*renamed ESIS in 1990*)
ENF	End-Notched Flexure (*specimen*)
ESIS	European Structural Integrity Society
ISO	International Organisation for Standardisation
J_C	Critical Fracture Energy [*or*] Critical Fracture Toughness (*from path-independent contour integral*)
JSA	Japanese Standards Association
JIS	Japanese Industrial Standard
K_{IC}	Critical Mode I Stress Intensity
LEFM	Linear Elastic Fracture Mechanics
NWI	New Work Item (*for standardisation*)
PVC	Poly-Vinyl-Chloride
RR	Round Robin (*test involving several participating laboratories*)
TC	Technical Committee
3-ENF	three-point-ENF (*test or specimen*)

Standardization of fracture tests for polymers, polymer composites and polymer-based adhesives requires development of test procedures and their validation in RRs for assessing in-laboratory and inter-laboratory scatter and their respective accuracy and precision data. Such activities often take place in technical committees, e.g., ASTM D20 or D30, ESIS TC4, or JSA. Test procedures are then submitted to standards organizations, e.g., ISO, JSA, ASTM, or CEN. These documents are typically handled in a step-wise balloting process with the aim of them becoming accepted by majority votes both in respect of technical content in addition to formal requirements and language.

2. Overview of ESIS TC4 activities to date

2.1. Development of ISO standards within ESIS TC4

Since 1985, ESIS TC4 has developed six ISO standards (three on plastics, two on fiber-reinforced composites, and one on adhesives), another two standards are currently in the ISO balloting process (one is a peel test for flexible laminates, e.g., used in packaging, and the other a plane stress fracture resistance test for thin plastic films and sheets), see Table 1 for details. Several test procedures are in preparation (see Table 2), and proposals for new test developments are discussed or under consideration.

Table 1. ISO test standards developed by ESIS TC4 (active documents and in ballot).

Standard title	Designation	Active since
Plastics — Determination of fracture toughness (G_{IC} and K_{IC}) — Linear elastic fracture mechanics (LEFM) approach	ISO 13586	Issued 2000; revised 2018
Fibre-reinforced plastic composites - Determination of mode I interlaminar fracture toughness, G_{IC}, for unidirectionally reinforced materials	ISO 15024	Issued 2001
Plastics - Test method for Tension-Tension Fatigue Crack Propagation	ISO 15850	Issued 2002; revised 2016
Plastics — Determination of fracture toughness (G_{IC} and K_{IC}) at moderately high loading rates (1 m/s)	ISO 17281	Issued 2002; revised 2018
Plastics — Determination of fracture toughness (G_{IC} and K_{IC}) — Linear elastic fracture mechanics (LEFM) approach — Amendment 1: Guidelines for the testing of injection-moulded plastics containing discontinuous reinforcing fibres	ISO 13586/Amd 1	Amendment issued 2003, withdrawn 2018, replaced by "Testing of plastics containing short fibres" as Informative Annex B in ISO 13856:2018 and Informative Annex C ISO 17281:2018
Adhesives — Determination of the mode 1 adhesive fracture energy of structural adhesive joints using double cantilever beam and tapered double cantilever beam specimens	ISO 25217	Issued 2009; based on BS 7991 (issued 2001)
Fibre-reinforced plastic composites — Determination of the mode II fracture resistance for unidirectionally reinforced materials using the calibrated end-loaded split (C-ELS) test and an effective crack length approach	ISO 15114	Issued 2014
Peel test for the determination of interlaminar fracture energy of flexible packaging laminates	ISO/CD 18485	Standardization presently on hold, pending RR for precision statement
Plastics — Determination of fracture toughness of films and thin sheets: the essential work of fracture	ISO/CD 23524.2	In ballot (Spring 2021)

Table 2. Fracture test procedures under development by ESIS TC4.

Document working title	Development started in	Current status and remarks
Plastics - High loading rates >1 m/s	1992	One RR completed, not submitted to ISO, data published by Leevers et al. (2014)
Plastics – Determination of fracture initiation by J-testing of ductile polymers at slow speeds using the load separation criterion	1992/2008	Several RR on J testing multi-specimen procedure since 1988, data published by Hale and Ramsteiner (2001), three RR on J testing with load separation criterion since 2008, not submitted to ISO yet, RR data published by Agnelli et al. (2015)
Plastics - Impact J testing	1993	RR completed, summarized in TC4 Annex to J testing protocol, not submitted to ISO, pending completion of J testing protocol, data published by MacGillivray (2001)
Fibre-reinforced plastic composites – Determination of mode I interlaminar fracture toughness, GIC, for unidirectionally reinforced materials at moderately high loading rates (~1 m/s)	2004	One RR completed, a second RR is in preparation, no RR data published yet
Adhesives — Determination of the mode II adhesive fracture energy of structural adhesive joints using the calibrated end-loaded split (C-ELS) test and an effective crack length approach	2005	RR still in progress, the procedure is based on ISO 15114 for fiber reinforced composites, no RR data published yet
Polymer films and coatings - Scratching	2006	Preliminary tests, no RR yet, data published by Blackman et al. (2016)
Polymers - Cutting	2006	Preliminary tests, no RR yet, test details are published by Williams & Patel (2016) and data by Patel et al. (2009)
Fibre-reinforced plastic composites - Determination of mode I interlaminar fracture toughness, G_{IC}, for unidirectionally reinforced materials under cyclic fatigue fracture loading	2008	Two RR completed, third RR on alternative procedure and data analysis taking fiber bridging into account is in progress, see Yao et al. (2017) for details
Environmental Stress Cracking	2008	Preliminary tests performed at selected laboratories, RR in preparation
Fibre-reinforced plastic composites — Determination of fixed-ratio mixed mode I/II fracture resistance for unidirectionally reinforced materials using the inverted calibrated end-loaded split (C-ELS) test and an effective crack length approach	2011	One RR completed, ISO draft document in preparation, fixed mode ratio I:II is 4:3
Fibre-reinforced plastic composites — Determination of mode II interlaminar fracture toughness, G_{IIC}, for unidirectionally reinforced materials under cyclic fatigue fracture loading	2011	One RR completed (C-ELS and 3-ENF), draft in preparation, not submitted to ISO yet, selected data published by Brunner (2015)
Plastics - Notching of polymers	2013	Three RR completed, intended informative annex to ISO 13856 and ISO 17281
Fibre-reinforced plastic composites - Laminate mandrel peel test for thin composites	2015	First RR with two materials in progress, test based on ISO/CD 18485 mandrel peel test for flexible laminates
Fibre-reinforced plastic composites - Laminate mandrel peel test for thin composites	2015	First RR with two materials in progress, test based on ISO/CD 18485 mandrel peel test for flexible laminates

Several procedures "under development" listed in Table 2 indicate rather long time-periods since the development started. There are various reasons for this, e.g., the limited availability of suitable materials to perform RR, the limited availability of participating laboratories with suitable test equipment and operators (for some activities involving novel apparatus, e.g., the laminate peel test, the limited availability of the apparatus has been overcome by creating a “travelling rig” whereby the test fixture is sent from one RR participant to the next). Frequently the data analysis for a test has posed problems, e.g., the need to obtain acceptable in-laboratory and inter-laboratory scatter has been challenging with sometimes for former achieved only. The standardization process, at least in ISO, now follows a rather strict timeline that allows for the development of a new standard in about three years from the time of acceptance as a NWI to an active standard.

ASTM subcommittee D30.06 also organized a RR on mode I fatigue fracture of polymer composites with two carbon fiber epoxy composites (IM7/977-3, and G40-800/5267-1), and one glass fiber epoxy composite (S2/5216) in 2009-2010 with one laboratory from ESIS TC4 participating. Partial results were published by Stelzer et al. (2012) and by Murri (2014) and Brunner (2015). However, there is no active ASTM standard published and the topic is not included among the current ASTM work items. In April 2021, the ASTM D30.06 website lists two work items related to activities of ESIS TC4. The first is WK67477 "Standard Test Method for Determination of the Mode II Interlaminar Fatigue Crack Growth Rate and Onset of Unidirectional Fiber Reinforced Polymer Matrix Composites Using the End-Notched Flexure (ENF) Test" and the second is WK74182 "Characterizing Mode-I Interfacial Fracture Toughness of Adhesives with Composite Adherends". ESIS TC4 had performed preliminary tests with a few selected laboratories on mode II interlaminar fatigue fracture comparing the C-ELS with a 3-ENF test-rig, as well as fatigue fracture under fixed ratio mixed mode I/II with the inverted C-ELS test-rig in one laboratory in 2012. These tests indicated the basic feasibility; selected data of both RR have been published by Brunner (2015). Development of standard test procedures will continue within ESIS TC4, and potential topics for new activities are discussed below.

Table 3. Fracture Mechanics Test Methods for Polymers, Composites and Adhesives, ESIS Publication 28 (2001).

Chapter title	Pages	Author(s)
Introduction to linear elastic fracture mechanics	3-10	J.G. Williams
K_c and G_c at slow speeds for polymers	11-26	J.G. Williams
Determination of fracture toughness (G_{IC} and K_{IC}) at moderately high loading rates	27-58	A. Pavan
The measurement of Kc and Gc at slow speeds for discontinuous fibre composites	59-72	D.R. Moore
Determination of the impact fracture toughness K_{Id} of plastics at high rates of loading “>1m/s”	73-89	W. Böhme
Fatigue crack growth of polymers	91-116	L. Castellani, M. Rink
Introduction to elastic-plastic fracture mechanics	119-122	J.G. Williams
J-Fracture toughness of polymers at slow speed	123-157	G.E. Hale, F. Ramsteiner
J-Fracture toughness of polymers at impact speed	159-175	H. MacGillivray
Essential Work of Fracture	177-195	E.Q. Clutton
Introduction to adhesion and adhesives	199-202	A. Kinloch
Peel testing of flexible laminates	203-223	D.R. Moore, J.G. Williams
Fracture tests on structural adhesive joints	225-267	B. Blackman, A. Kinloch
Introduction to delamination fracture of continuous fibre composites	271-275	P. Davies
Mode I delamination	277-305	A.J. Brunner, B.R.K. Blackman, P. Davies
Mode II delamination	307-333	P. Davies, B.R.K. Blackman, A.J. Brunner
Delamination fracture of continuous fibre composites: Mixed-mode fracture	335-359	B.R.K. Blackman, A.J. Brunner, P. Davies

2.2. Dissemination of fracture test development and research

Standards or guidelines have not been the only product or output of ESIS TC4. The committee has organized a series of eight conferences on the fracture of polymers, composites and adhesives to date (the first in 1994, then tri-annual from 1999 on) and currently is preparing two more (a virtual conference replacing that planned for 2020 in September 2021 and a regular conference in September 2023). In addition to these, ESIS TC4 in 2016 also organized a symposium on "Advanced Fracture Mechanics Testing of Polymers, Adhesives and Composites" within ECF-21. Elsevier (2001) has published selected ESIS TC4 test procedures for polymers, polymer composites and adhesives in a book as ESIS Publication 28. Table 3 shows the titles of the papers and the authors. A second book, ESIS Publication 33 by Elsevier (2004), presented applications of fracture mechanics to polymers composites and adhesives. Selected contributions from the first three conferences have been published as books, namely ESIS Publication 19 by Mechanical Engineering Publications, Ltd. (1995) and as ESIS Publication 27 (2002) and ESIS Publication 32 (2003) by Elsevier. Since 2006, conference contributions are published in special issues of Engineering Fracture Mechanics. Selected papers from the TC4 symposium at ECF-21 in 2016 have been published in Volume 2 of Procedia Structural Integrity in 2016. Two ESIS TC4 status reviews for delamination resistance of fiber-reinforced polymer-matrix composites have been published by Davies et al. (1998) and Brunner et al. (2008).

Recent meetings of ESIS TC4 since 2018, in addition to discussing the test procedures under development and the associated RR data review, have also included technology discussions on state-of-the-art issues in fracture and open questions. The topics covered so far have included "Composites fatigue fracture" (by Prof. René Alderliesten, TU Delft), the "Status of modelling and simulation of fracture mechanics" (by Prof. Jordi Renart, Universitat Girona), "Environmental effects on fracture" (by Prof. Jörg Fischer, Johannes Kepler Universität Linz) and on "Smarter testing" (by Prof. John-Alan Pascoe, TU Delft). It is likely that future fracture testing and test developments will benefit from suitable modeling or simulations. On one hand, this will assist the optimization of test set-ups and identify critical parts as well as support RR data analysis and interpretation, and on the other, it will advance the use of fracture mechanics based design of components or structures by implementing so-called "smarter" testing techniques. This term refers to a combination of composite material testing with extensive modelling and simulation, replacing some of the testing in the so-called building block approach for validating structural designs, see, e.g., Chisholm (2019) for details.

2.3. Fracture test data publications by ESIS TC4

Members of ESIS TC4 have also published several research papers reporting and discussing RR data and analysis, and selected examples are noted here. The precision statement in ISO 13586:2018 contains RR results on a polyamide material and that in ISO 17281:2018 results on a PVC material from the RR performed by ESIS TC4. The RR data for the fracture of polymers at loading rates around 1 m/s were published by Pavan and Williams (2000). ISO 25217 on quasi-static mode I fracture of structural adhesives does not provide a precision statement, but the RR data from the development of the test procedure were published by Blackman et al. (2003). ISO/CD 23524.2 on essential work of fracture of thin polymer films has a precision statement with RR data from five laboratories, and data have also been published by Williams and Rink (2007).

For composites, selected RR data of quasi-static delamination resistance tests were published by Davies et al. (1990) for an unspecified unidirectional carbon fiber epoxy laminate, for a CF/epoxy and a CF/PEEK by Davies et al. (1992), for quasi-static mode II of a CF/epoxy (HTA-12000 carbon fibres in Toho 113 epoxy resin, produced from Toho Q-1113-1450 prepreg) again by Davies et al. (1999), for quasi-static mode I testing of unidirectional and cross-ply laminates made of T300 fibers and 970 epoxy and IM7 fibers and 977-2 epoxy in a book chapter by Brunner (2008) and a research paper by de Morais et al. (2002). RR data from quasi-static and cyclic fatigue fracture RR under mode I and mode II loading with unidirectional CF/epoxy and a CF/PEEK (type IM7/977-1, IM7/977-2, IM7/977-3, G30-500 12k/Rigidite 5276 and AS4/PEEK, respectively) were published by Brunner et al. (2009), Brunner et al. (2013), Stelzer et al. (2014), and in a book chapter by Brunner (2015). Selected ESIS TC4 members also participated in the joint RR on mode I delamination resistance of polymer composites organized by ASTM, EGF and JIS in the early 1990ies. The data from this joint RR have been published by O'Brien and Martin (1993). JSA performed a RR on mode I delamination resistance without international participation, the results were published by Hojo et al. (1995).

3. Discussion of open issues in fracture test standardization and approaches

3.1. Fiber bridging in polymer composites

The so-called delamination resistance curve from quasi-static mode I tests (G_{IC} plotted versus delamination length) of polymer composites reflects fiber bridging between the two beams of the standard unidirectionally fiber-reinforced DCB specimens. The amount of large-scale fiber bridging and of delamination resistance typically increases with increasing delamination length after delamination initiation, until reaching saturation at which the delamination resistance remains roughly constant (with some variation about an average propagation value). The difference between initiation and average propagation values depends on the type of composite and may range from about 100 J/m^2 to several hundred J/m^2 or more, see, e.g., Sørensen and Jacobsen (2000) or Brunner (2015). For cyclic fatigue fracture, fiber bridging also affects the data, in that case, the curves describing average delamination rate per load cycle for a range of G_{IC} values are shifted to higher values of G_{IC} for higher amounts of fiber bridging. An important issue is the use of the materials' fracture test data in structural design with polymer composites. In composite parts or structures, the fiber orientation is often not unidirectional. Hence, delamination resistance in most composite structures does not show significant fiber bridging effects and is lower than the standard test values. One exception to that, however, are wind rotor blades were the fiber-bridging is considered in the design, see, e.g., Sørensen (2020). Of course, delamination propagation in components and structures is affected by more than just the fiber orientation and the resulting fiber bridging, Other factors include, e.g., shape of the part and ply drop-offs, residual stresses from manufacturing, or defects from processing, see, e.g., Sørensen (2020). Hojo and Aoki (2015) had proposed a procedure for the determination of Mode I fatigue fracture yielding data that allow extrapolation to a curve without fiber bridging. The procedure was a so-called "constant-G" test, but this was rarely used, due to the required machine control that was not available on all test machines at that time. Recently, Yao et al. (2017) developed a multi-step Mode I fatigue fracture test with alternating quasi-static and cyclic fatigue loads applied with increasing load and displacement levels, respectively. This yielded a set of curves that eventually produced a steady state for which subsequent curves overlapped. A back-extrapolation procedure then resulted in a curve without fiber-bridging effects that proved more conservative than the data from fatigue fracture cycles run at a given load or displacement level that were dominated by fiber-bridging. Using a modified Hartman-Schijve equation to plot the data as discussed by Jones et al. (2012, 2014) provides explicit values of delamination thresholds as well as quantitative scatter estimates for that, see, e.g., Mujtaba et al. (2017) or Jones and Kinloch (2020).

Depending on the laminate lay-up and the fiber orientation in the different plies, a propagating delamination may branch into two or more delaminations, e.g., in multidirectional laminates as observed by Choi et al. (1999). This may be beneficial for increasing the delamination resistance of the material by the different plies or fiber bundles bridging the delaminations, but it is difficult to quantify the delamination resistance in terms of critical energy release rate G and R-curves (G versus delamination length a). Khudiakova et al. (2021a, 2021b) discuss approaches of how laminates with multiple delaminations might quantitatively be characterized for delamination resistance under quasi-static and cyclic mode I fatigue fracture loadings, respectively. Since only a limited amount of data has been analyzed this approach will require additional investigations for validation.

3.2. Environmental effects on fracture and fatigue fracture of polymers, polymer composites and adhesives

Environmental exposure may come from many different sources and may induce a wide range of degradation mechanisms in polymers, including several that affect their fracture toughness as discussed by, e.g., Hinkley and Connell (2012). For assessing the long-term durability of a polymer or polymer composite part or structure, these effects require quantification. ESIS TC4 is preparing a RR on environmental stress cracking of polymers based on the preliminary investigations by, e.g., Kamaludin et al. (2017), Bredács et al. (2019) and Contino et al. (2021). For polymer composites, effects of environmental exposure have been discussed by, e.g., Broughton (2012) or Davies et al. (2012), the latter specifically focusing on marine environment. Environmental effects on adhesives joints are discussed by, e.g., Dillard (2010) or Costa et al. (2017). Of course, the variety of environmental conditions in the different service environments, typically comprising an ambient medium (e.g., air, humidity, service fluids) often combined with temperature variations requires an extensive experimental effort. Such effects will have to be considered, among others, in aircraft operation, see, e.g., Brunner (2019) for references with test conditions and data. Ideally, the exposures defined in the test methods should accelerate the environmentally induced degradation of the test specimens beyond that under service conditions to provide data for prediction of the behavior in-service in a reasonable time frame. The combination of different types of exposure or simultaneous exposure of specimens to several specific fluids may result in synergistic effects that cannot be reliably predicted from single exposure tests. The damage mechanisms may change with time or variation of exposure, as discussed by Bank et al. (1995), Stewart and Douglas (2012), or Qin et al. (2021) for the example of polymer composite structures in civil engineering applications.

3.3. Scatter sources in fracture and fatigue fracture data of polymer composites

The sources of scatter in fracture testing data comprise two classes: (1) extrinsic, e.g., from test set-up, measurement resolution, operator dependent effects, and (2) intrinsic, essentially from material property variation due to manufacturing and processing, as discussed for the case of polymer composites by Alderliesten et al. (2018). Extrinsic scatter cannot be completely eliminated, but a well-designed test protocol should minimize this, while intrinsic scatter, (if representative of the manufacturing and processing used to form the composite structures or components) shall be preserved. If load cells and displacement transducers with appropriate measurement resolution are used and play and compliance of the test-rig are accounted for the operator-dependent effects remain the largest source of extrinsic scatter. This holds true not only for conducting the tests (where the visual observation of delamination propagation for determination of the crack length is the major component, as discussed in more detail below), but also for the data analysis, if performed manually. Examples of operator-induced scatter in data analysis include, e.g., variation in the determination of the non-linear and 5% compliance increase load points from the load-displacement record, as discussed by Davies (1996). As discussed below in more detail, digital data fitting routines discussed by Clerc et al. (2019) may eliminate these operator dependent effects and reduce the respective scatter, while simultaneously providing a consistent analysis approach.

Agnelli and Horsfall (2013) discussed scatter for high-rate fracture tests on polymers for which RR data had shown large scatter in the time-to-fracture measurements from which the toughness was calculated. Notching quality was identified as a major source of scatter. Notching techniques for fracture tests on polymer specimens are under investigation in an ESIS TC4 RR (see Table 2). Even though razor-blade tapping by hand has been found to yield lower toughness values for brittle epoxies than most other approaches, see e.g. Guild et al. (2018), the comparatively poor repeatability and the dependence on operator skills are considered a disadvantage of this technique. An ablation process with a femto-laser equipment yielded sharp notches in polymer films without significant damage around the crack tip as reported by Martinez et al. (2013). However, a comparison by Salazar et al. (2015) between several techniques indicted that for high-density polyethylene specimens fatigue pre-cracking proved to yield the lowest values. ESIS TC4 has a RR activity comparing notching methods for different polymers with the aim of providing guidelines intended for submission as an informative annex to the ISO standards on toughness of polymers.

Reasonably safe design limits can only be determined based on the data from the standard fracture tests, if the extrinsic scatter is minimized, an if for fiber-reinforced polymer composites with non-unidirectional lay-up the fiber-bridging effects (which in principle are intrinsic for unidirectional composite laminates) are properly accounted for. Jones et al. (2017) and Mujtaba et al. (2017), for example, discuss the scatter in the test data from polymer composites and determination of design limits from such data in detail.

3.4. Digital tools in fracture and fatigue fracture testing

Use of digital "tools" in fracture testing dates back to the last century, when first test machines with digital data acquisition and digital control became available. Within the activities of ESIS TC4, programmed spreadsheets for data analysis were the first digital tool for data analysis. These proved essential for making RR data from different laboratories comparable as discussed by Brunner (2020). Digital imaging and image analysis is now available at affordable cost, e.g., with Digital Image Correlation for operator-independent determination of delamination lengths, see, e.g., Khudiakova et al. (2020). Digital cameras for still images or video recording of delamination tests even work for high-rate tests with loading rates up to several tens of m/s, where visual observation is not feasible, as shown by

Thorsson et al. (2018), Isakov et al. (2019) or Ekhtiyari et al. (2020). Interfaced with the test machines these tools automatically yield the raw data required for determination of toughness (corresponding loads, displacements and delamination lengths) under quasi-static loading, see, e.g., Chocron and Banks-Sills (2019). Digital tools clearly show potential for improving data acquisition and analysis by reducing scatter from human effects, e.g., in visual observation for quantification of cracks or delaminations, and in manual determination of non-linear load points indicating fracture initiation as shown for one example by Clerc et al. (2019). A promising perspective, especially for large numbers of standard fracture tests, are fully automated test set-ups operated by robots, simultaneously eliminating human errors, allowing for continuous operation, and, finally, saving cost. Sun et al (2020, 2021) have investigated the use of DIC to simultaneously measure crack length, crack tip opening velocity, G_C and J_C in mode I fracture tests on structural adhesive joints at various test rates. The techniques offer significant future potential for the automation of fracture testing.

Modelling and simulation approaches for fracture and fatigue fracture issues also benefit from advances in digital technology, specifically from higher computational power to handle large amounts of data and/or to perform the calculations faster. A recent development are "digital twins" that allow for simulating essential aspects of the long-term behavior of composite parts or structures rather than having to perform complex structural tests. One example of such a digital twin is a composite wind rotor blade presented and discussed by Sayer et al. (2020).

3.5. Fracture testing of "new" polymeric materials and polymeric materials manufactured with "new" processes

Several recently developed new polymeric or polymer-based materials have been noted in the Introduction. With respect to determination of their fracture properties, the question is whether standard procedures are applicable or whether they have to be modified, or else whether new test methods or new analysis approaches have to be developed and validated. For nano-modified polymers and adhesives or for composites with nano-modified matrix, the applicability of standard procedures has been shown, e.g., by Brunner et al. (2006) for epoxy with layered nano-silicates, Srivastava et al. (2018) for graphene platelets and carbon black filled epoxy adhesives, and by Domun et al. (2020) or Burda et al. (2020, 2021) for composites with nano-modified epoxy matrix. One limitation may be imposed by the use of highly toughened adhesives where the adherends may fail before the adhesive as discussed by, e.g., Blackman et al. (2012) and Jojibabu et al. (2020). Soft materials, such as gels, viscoelastic polymers and soft elastomers, may also pose problems in measuring toughness, often due to their viscoelastic behavior, see, e.g., Kwon et al. (2011), Shen and Vernerey (2020), Guo et al. (2020) for a discussion of the relevant issues. Kwon et al. (2011) found reasonable agreement between essential work of fracture and J-integral tests on soft biogels made from agarose powders. Silica aerogels, on the other hand, may require new or adapted test methods as discussed, e.g., by Haj-Ali et al. (2016). Fracture of soft fiber-reinforced polymer composites with stiffness ratios between fiber and matrix up to 10^7, i.e., much higher than the typical 10^2-ratio for conventional fiber-reinforced polymer-matrix composites with thermoplastic or thermoset matrices, is discussed by Hui et al. (2020). ESIS TC4 is planning the development of a fracture test procedure for elastomers and that may be considered for other soft polymeric materials in the future.

Among new processing and manufacturing methods, AM techniques are explored for various materials, see, e.g., Bhuvanesh Kumar and Sathiya (2021), and specifically for polymers, see, e.g., Das et al. (2021) or Sharafi et al. (2021), but also for fiber-reinforced composites with thermoplastic, see, e.g., Blok et al. (2018) and thermoset matrix materials, see, e.g., Hao et al. (2018) or Ming et al. (2019). One issue in polymer materials and parts produced with AM is the defect distribution from manufacturing that may differ significantly from that resulting from other processes, see, e.g., Regalla et al. (2020) and Penumakala et al. (2020). The quantification approaches for mode I delamination resistance of laminates with multiple delaminations discussed by Khudiakova et al. (2021a, 2021b) effectively deal with a thermoplastic CFRP composite that has been manufactured by an AM technology, specifically the automated tape placement with in-situ consolidation.

3. Summary and Outlook

Development and validation of test procedures for measuring the fracture mechanics parameters (G_c, K_c and J_c) for polymers, polymer composites and polymer-based adhesives are the core activity of ESIS TC4 and this will continue into the future. However, there are important issues that require research for future developments. Even though ISO test standards are not application-specific, the potential applications of the test data require consideration in drafting new procedures. One example of this are the cyclic fatigue fracture tests for composites in which fiber-bridging has to be accounted for if the data are to be used in fracture mechanics based structural design with non-unidirectional laminate lay-up. For unidirectionally reinforced composites, fiber-bridging mainly affects the Mode I tensile opening loads, but may also play a role in other loading Modes or Mode mixes. Understanding the effects of environmental exposure on the fracture behavior of polymer-based materials and components is essential for long-term service and, in ESIS TC4, this is about to become a RR activity for fracture of polymers. For new polymer-based materials developed by industry, or such materials manufactured with new processes, the applicability of the existing standards and of the test procedures under development has to be assessed. If necessary, these procedures should be suitably adapted. Examples of the former are "soft" materials, and examples of the latter are polymers and polymer composites produced by AM processes. In the case of significant changes to the properties compared with currently available materials, which were investigated in the RR, procedures may have to be adapted and re-validated. Integration of digital tools into the test procedures is another aspect that deserves attention. It is expected that, first and foremost, operator-dependent effects and resulting scatter can be reduced by digital tools. Further, there is some potential for simultaneously also reducing test cost, e.g., by implementing partially or fully automated test set-ups and data analysis routines. Beyond the determination of the fundamental fracture properties of materials and their intrinsic scatter, simulation and modelling are expected to become increasingly important, for example in the "smarter" testing of components and structures.

Finally, ESIS TC4 is always open for collaboration with research laboratories and industry, and interested researchers are welcome to attend the semiannual meetings (usually one in Spring, i.e., March to April, and one in Fall, i.e., September to early October) and the conferences. Information on ESIS TC4, its activities, the meeting schedule and the contact details are available under the following link:

https://sites.google.com/structuralintegrity.eu/tc04

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