CURRENT ISSUES IN FRACTURE AND FATIGUE FRACTURE OF FIBER-REINFORCED POLYMER COMPOSITES: AN OVERVIEW

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ABSTRACT

Obtaining sufficiently reproducible quasi-static or fatigue fracture data for fiber-reinforced polymer (FRP) composites is an essential requirement for the design of damage-tolerant FRP components and structures allowing for controlled crack growth in service rather than imposing a "no growth" design requirement. Despite long-term research and development efforts, the different fracture test procedures for FRP composites proposed or standardized remain unsatisfactory. Selected examples will be presented and discussed. Among the issues not yet considered in the standardized testing procedures are fiber-bridging and multiple delaminations, the service environment, possible synergistic effects from material modifications, as well as scatter and sources of scatter in fracture testing and in modelling. Potential approaches to improve testing and reduce scatter are also discussed.

1 INTRODUCTION

In the last few years, an increasing number of publications, e.g., [1-5] have questioned the applicability of using data from standardized and non-standard quasi-static or fatigue fracture test methods for the design of structures comprising fiber-reinforced polymer-matrix (FRP) composites. Some issues relate to upscaling from the laboratory-scale to larger structures. Planar, beam specimens with unidirectional fiber orientation and defined, single delaminations essentially yield one-dimensional delamination propagation. Larger structures, frequently two-dimensional shells, are often curved or with more complex shapes and with thickness variations. These structural features result in two-dimensional delamination propagation- distinctly different from that observed in beam specimens [6]. Components and structures frequently have multidirectional or quasi-isotropic fiber orientations, i.e., they also differ from the unidirectional lay-up of test coupons [7]. Processing and manufacturing can induce multiple defects which act as delamination starters under complex load spectra in variable service environments [8]. Interacting multiple delaminations are likely to yield complex propagation effects that are difficult to characterize and quantify experimentally. Another issue relating to up-scaling is that the modelling and simulation (which have great potential to reduce the test effort in the so-called building block design approach: from test coupons to element and component level and, finally, to full-scale structures [9,10]) require accurate input data. The scatter in fracture test data, amounting to 10-20% in repeatability or reproducibility observed in many round robin tests [11] poses problems for modelling, limiting the accuracy and precision of the simulations. Some approaches for solving these problems are discussed.

2 TESTING PROCEDURES

There are standardized procedures for quasi-static fracture testing of fiber-reinforced polymer-matrix (FRP) composites under mode I, mode II and mixed mode I/II loading [12]. For fracture under cyclic fatigue loading, there have been attempts to develop test procedures, but with the exception of one procedure for the mode I fatigue delamination onset [13], no standards exist yet. The status of standardization procedures (published and under development) has recently been reviewed, e.g., [12,14]. A significant round robin test effort currently addresses the effects from unidirectional fiber lay-up

[15,16] by evaluating a procedure that eliminates the fiber-bridging (Figure 1) typically observed in unidirectional laminates [17]. However, there are applications where unidirectional lay-ups are intentionally used to induce fiber-bridging to increase delamination resistance. One example is in wind-rotor blades [18], another in glass-fiber reinforced rods used as core elements in high-voltage insulator components [19]. In the future, modelling procedures that allow for quantifying the delamination behaviour for arbitrary fiber lay-ups from the standard test data will be required [5].

Figure 1: Mode I tensile opening fracture tests on (left) glass-fiber reinforced rod with unidirectional fiber lay-up yielding significant fiber bridging and delamination resistance (right) carbon-fiber reinforced laminate beam also with large-scale fiber bridging, but to a much lesser extent.

3 ISSUES IN QUASI-STATIC AND FATIGUE FRACTURE

3.1 Fibre-bridging

Fiber-bridging in unidirectional laminates is observed under both quasi-static and cyclic loading. In quasi-static tests, this yields the so-called R-curves, i.e., increasing resistance with increasing delamination length after initiation. In cyclic loading, fiber-bridging tends to shift the data in the so-called Paris graph (double logarithmic graphs of average delamination length increment per cycle as a function of applied energy release rate) towards higher energy release rates (Figure 2).

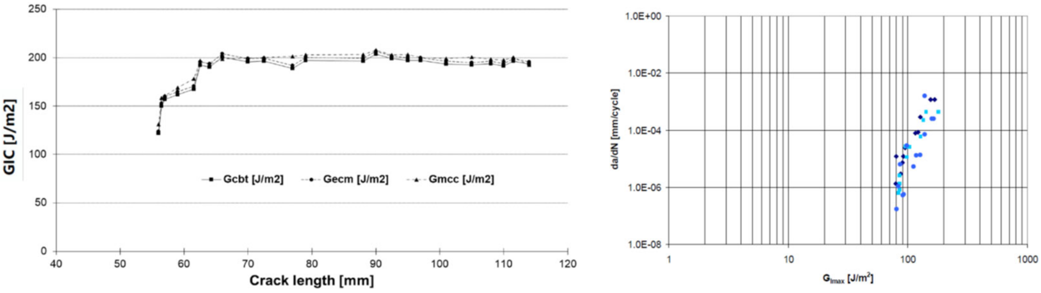

Figure 2: Results of mode I delamination testing of a carbon-fiber epoxy composite (IM7/977-3) under (left) quasi-static loading: G_{IC} versus crack length and (right) cyclic fatigue loading: Log da/dN versus applied maximum G_{IC}.

Figure 2 (left) shows a delamination resistance curve obtained from the quasi-static fracture testing of a CFRP epoxy specimen (IM7/977-3), analysed using three methods (cbt = corrected beam theory, ecm = experimental compliance method, mcc = modified compliance calibration). From the initiation at about 125 J/m^2, the G_{IC} values increase, but within about 10-12 mm of propagation they stabilize at a plateau of about 200 J/m^2. Figure 2 (right) shows delamination propagation curves for three specimens of the same material under cyclic fatigue loading in a double-logarithmic plot of delamination length increment per cycle da/dN versus G_{Imax}, i.e., the so-called Paris graph. Fatigue thresholds below which no delamination propagation is observed (around 80 J/m^2 for the material estimated from Figure 2 right) estimated from the graphs hence tend to overestimate delamination resistance for lay-ups yielding less fiber bridging, e.g., quasi-isotropic lay-ups which would shift curves to the left in Figure 2).

There are currently two methods for experimentally assessing fiber-bridging and determining delamination resistance limit values in cyclic fatigue fracture tests that do not include such effects. The first is to perform tests under constant G control instead of under displacement or load control. Even though this method was proposed in the 1980s [20], it was rarely used, since many test machines at that time could not perform the required G-control sufficiently well. The second method is to perform the cyclic tests in a series of steps with increasing maximum displacement from one step to the next. All steps are performed at constant displacement-ratio, achieved by adjusting both the upper and lower displacement limits. This generates a series of curves that are shifted to higher values of G_{IC} in the Paris plot, until a steady state is reached (Figure 3). The back-extrapolation procedure described in [17] then yields a "fiber-bridging free" curve that can provide safe design limits, once the appropriate safety factors have been included. Both methods, i.e. (constant G [20] and stepwise incremental displacement [16,17]) require a major test effort of several weeks' duration, if a statistically significant sample of at least five specimens are tested. The test duration for both methods is determined by the test frequency applied (between a few and 10 Hz) and the minimum delamination length increment per cycle (da/dN) per specimen [20] and step [16,17], respectively. The latter could be set to a higher value in order to reduce the test duration per specimen. A comparison between the two methods for identical composites would yield information on potential bias [21], i.e., on (cite) *"... the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value"*. Currently, there are no bias statements in any fracture test procedures for polymer composites.

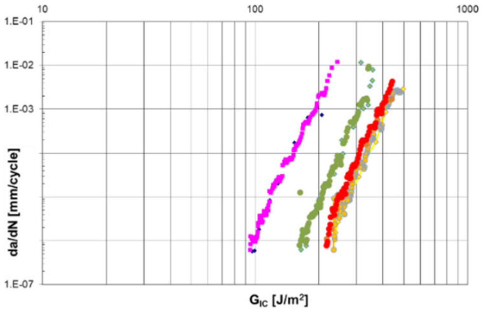

Figure 3: Mode I cyclic fatigue fracture testing, four sequential steps with increasingly higher displacement, yielding overlapping curves for the steady state of fiber bridging, the data can then be back-extrapolated to the "fiber-bridging free" behavior according to [16].

All the fracture data shown in Figures 2 and 3 were obtained from unidirectionally fiber-reinforced laminates. In many structural applications, as noted above, the fiber-lay-up is multidirectional or quasi-isotropic. Such lay-ups tend to yield less fiber-bridging than the unidirectional lay-up. Nevertheless, some fiber-bridging may still occur. However, as more frequently observed in multi-directional lay-ups, the delamination may tend to migrate to another plane or branch into multiple delaminations propagating simultaneously [22-24]. A multidirectional laminate lay-up and its effects on the resulting delamination resistance under quasi-static and cyclic fatigue loading has been investigated by [7]. One aspect discussed was specimen thickness. The first conclusion was that under quasi-static loading, thinner specimens can generate more fiber-bridging and hence there is a thickness effect on the resulting delamination resistance curve. A thickness effect is also observed for cyclic fatigue loads, except for short crack lengths and for the plateau value and this, hence, is of less importance. The authors conclude (cite) *"... that neither thickness nor fibre bridging indeed has obvious effect on fatigue delamination behaviors, if the similarity is appropriately represented."*

Predicting the behavior of multiple delaminations even in a simple beam type specimen can be quite challenging and even more so for composite components or structures as discussed below in section 3.3. Therefore, the development of models predicting the delamination resistance for arbitrary fiber lay-ups (as well as for multiple delaminations) from the standard fracture test data with sufficient precision and accuracy is required [5].

3.2 Two-dimensional delamination propagation

Comparing standard test coupons for fracture testing of FRP with structural parts, coupons essentially differ from components with respect to their length and width, i.e., their planar size. Hence, the question can be asked, how changing the length and width might affect the delamination behavior? As discussed extensively in [25,26], increasing length and/or width to roughly square or rectangular plates yields two-dimensional delamination propagation for sufficiently large plates. The length of the crack tip increases with increasing delamination size, yielding effects not observed in standard test specimens (even though the crack tip in coupons does show some curvature). Hence, it is essential to model two-dimensional delamination behavior for different loading modes (at least mode I and II) and types (quasi-static and cyclic fatigue) to understand the effects. The modelling of mode I is discussed by, e.g., [27,28] and of mode II by, e.g., [29,30]. As expected, experiments indicate that the effects from fiber bridging also come into play, depending, of course, on the type of laminate lay-up.

3.3 Multiple delaminations

Another issue arising when upscaling from test coupons to larger structures is that of multiple delaminations (see Figure 4). These occur either due to processing defects [31] or they can originate from impact damage [32]. Quantifying the effects of interacting multiple delaminations under quasi-static or fatigue loading is challenging, and initial studies clearly indicate that further work is required [8,33], Multiple delaminations have been shown to result either in weakening [34] or toughening of the composite [35,36]. In composite structures or components their presence may reduce the buckling loads [37]. Toughening can be achieved by intentionally employing sacrificial defects, either delaminations formed by thin poly-tetra-fluor-ethylene films or by porosity [36,38]. The toughening by initiation and/or propagation of multiple delaminations requires more energy than for single delaminations.

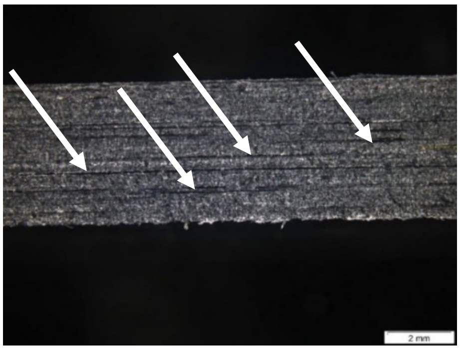

Figure 4: Edge view of a carbon fiber thermoplastic composite beam specimen showing multiple delaminations after manufacturing (examples are indicated by white arrows).

Interactions between multiple delaminations, however, can be quite complex. A Finite Element model assessed the interaction between multiple delaminations with unequal lengths in [39]. The authors concluded that the local values of G at the delamination tip differed due to their unequal lengths and hence yielded apparent scatter in the respective values of the G. These effects would be larger for delaminations present in different planes of the laminate. Compliance, therefore, would not be an appropriate measurement for delamination lengths in such laminates, as confirmed by the delamination length data shown in [8]. Modelling multiple delaminations is discussed in several publications, e.g., [37,40,41]. In structural elements, multiple delaminations under compression have an effect on bending loads and hence buckling failure, as discussed in [37,39]. Ullah et al. [41] started from bending tests on coupons and modeled the high deflection bending behavior in Abaqus with single and multiple layers of bilinear cohesive-zone elements. These authors noted that damage initiation and growth from their model was mesh sensitive, but they found agreement between modelling and experiments. Ma et al. [40] modelled delamination failure in a CFRP T-joint and noted that both methods (extended finite element method X-FEM and augmented finite element method A-FEM) predict load-displacement curves in agreement with experiments, but that failure progression predictions from X-FEM were inaccurate.

3.4 Service environment

A service environment of variable hygro-thermo-mechanical loading affects the delamination initiation and propagation in FRP composite parts and structures in complex ways. Further, stochastic events, e.g., impact by foreign objects, exposure to certain fluids, or improper handling may also induce damage. Often, hygric and thermal effects are correlated, e.g., for aircraft, at low temperatures and dry conditions at high altitudes or elevated temperatures under high humidity conditions on the ground. The time-scales of the variation of mechanical loads and environmental exposure can differ significantly. Even if specific service environments are tested, the full combination of mechanical loads and environment may yield synergistic effects not observed in single or limited parameter tests [42].

However, not all environmental exposures are necessarily detrimental. There are examples of exposures for which the delamination resistance increased compared to the unexposed material. These are discussed in [5]. Improvements in Mode I and Mode II delamination resistance were observed after exposure of an aircraft grade CFRP laminate (AS4/3501-6) to water and jet fuels (Jet-A, JP4) [43]. For Mode I tests that showed higher toughness increase than Mode II, the exposure resulted in a change of the failure location with more cohesive matrix fracture and fiber breaks. For Mode II, only exposure to jet fuel resulted in a change of fracture surface features, namely larger deformation of the hackles.

There are also examples of testing at low temperatures, where contrary to the expectation for decreasing toughness with decreasing temperatures based on published test data for CFRP, e.g., [44] a more complex behavior as a function of temperature was observed (Figure 5). The G_{IC} data are shown for two types of carbon-fiber epoxy [45], one with higher and one with lower toughness. With increasing temperature, both curves show increasing toughness. The tough epoxy possibly indicates a minimum toughness in the temperature range between 100 K and 300 K. However, considering the scatter in the measurements, this is unclear. For the mode II toughness of carbon-fiber epoxy laminates, two of three data sets (one plain woven and one unidirectionally fiber-reinforced laminate [46,47]) show a decreasing toughness with increasing temperature. The values for the unidirectional lay-up are significantly higher than those for the other two lay-ups. The third data set has multi-directional fiber lay-up [48] and shows a somewhat increasing and then decreasing trend with increasing temperature.

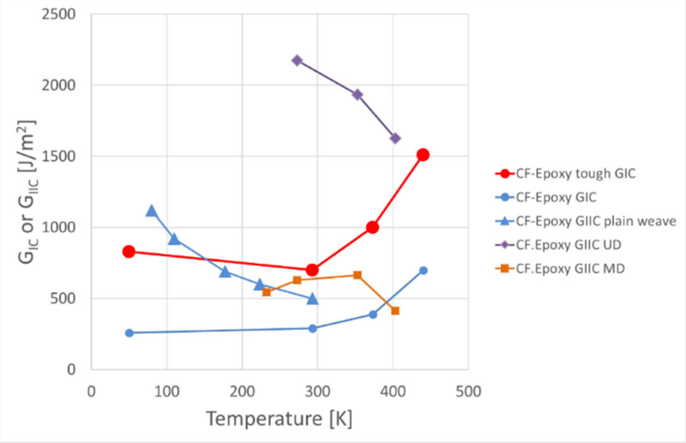

Figure 5: Selected literature data of delamination resistance versus Temperature, G_{IC} for CF-epoxy (toughened) [45], G_{IC} CF-Epoxy [45], G_{IIC} CF-Epoxy plain weave [46], G_{IIC} CF-Epoxy UD = unidirectional [47], G_{IIC} CF-Epoxy MD = multidirectional [48].

Mixed mode I/II tests reported on glass-fiber reinforced polyester laminates [49] indicated increasing toughness with increasing mode II contribution (Figure 6). However, for 77 K (liquid nitrogen temperature) the toughness showed a minimum compared with lower (4 K) and higher (293 K) temperatures. This differs from the approximately linear trend with increasing mode II component observed at 4 K. The data in Figure 5 for toughened epoxy also indicate a potential toughness minimum at a temperature above 4 K, but, considering the scatter in the data, the difference may be small.

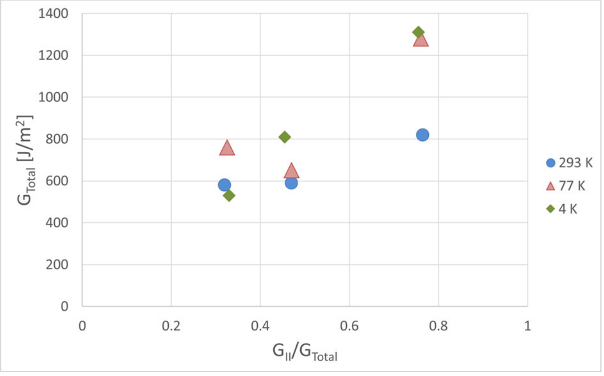

Figure 6: Energy release rate G_{Total} as a function of G_{II} to G_{Total} for a plain woven GFRP laminate for three different temperatures [49].

For unidirectional E-glass/vinyl ester laminated composites under exposure to an acidic environment, an increasing G_{IIC} was observed for short term (up to two weeks) of acidic exposure (distilled water and sulfuric acid with a concentration of 30 wt% and purity of 98 wt% at +25°C) but when the exposure was continued a decreasing G_{IIC} was observed to below the initial value [50]. Exposure to the same medium at +90°C resulted in an approximately linearly decreasing value of G_{IIC} with increasing exposure duration up to twelve days. The short-term increase at +25°C was explained as either being due to filling of the cavities in the material by the acidic solution, or by absorption of water acting as plasticizer for the matrix, or by reactions between hydroxyl groups with the matrix. It is interesting that the decrease observed at +90°C still showed a slight deviation from the essentially linear behavior after a few days (not noted by the authors). It could be speculated that there was a small improvement for a short time, briefly counteracting the overall degradation. However, apparently this was not sufficient to result in a pronounced toughness increase, possibly due to the higher diffusion rate of the acidic fluid and/or increased thermal degradation of the matrix at elevated temperature. Toughness degradation under variable exposure to the acidic medium might yield a complex, time-dependent behavior.

These examples illustrate the complexity of effects that can be induced by different service environments. If variable mechanical loads and environments with different, variable time-scales are combined, it is extremely challenging to make accurate predictions. One reason for this is the potential synergistic effect that may occur between different environmental factors. Appropriate models can only be developed if validated and sufficiently precise experimental data are available. Identifying such synergistic effects in experimental tests is difficult, as discussed in detail in [5]. Synergistic effects are often considered beneficial but, as already shown above, with the example of the exposure of GFRP to acidic environment, may be limited to certain time-scales and specific environmental conditions, e.g., limited temperature ranges. That synergistic effects improving one material property can simultaneously be detrimental to other properties and section 3.5 below briefly illustrates this point.

3.5 Material modifications

Many material modifications have been employed to improve the delamination resistance of FRP composites. Published examples indicate that certain types of nano- or micro-particles or combinations of particles improve the delamination resistance of the polymer and, to some extent at least, also that of the respective composite, see, e.g., [51,52]. Toughening effects in FRP composites can also be realised by interleaving with thin thermoplastic films [53,54]. However, in many cases, toughness improvements by adding certain fillers simultaneously reduce other properties. For thermoset polymer matrices, often, the glass transition temperature Tg is reduced. For GFRP-epoxy pultruded insulator rods, the toughening mechanism employed reduced the Tg as expected and there was a simultaneous trend for a reduction in compressive strength [55]. Delamination resistance and compressive strength could not be improved simultaneously, at least not with combinations of two or three of the fillers ($CaCO_3$, nano-silica, core-shell rubber). Some material modifications may yield additional damage mechanisms that can result in toughness improvements by inducing more damage per unit volume than other material recipes.

3.6 Sources of scatter in fracture testing

Quantitative values of scatter in fracture test data are available, e.g., from published round robin campaigns. Certain test standards nowadays require a so-called precision and bias statement. The latest version of the quasi-static Mode I test [56] states (cite) "*The repeatability standard deviation from a single operator has been determined to be 4.9 % for GIc NPC and 5.0 % for GIc PC.*" NPC is the non-pre-cracked toughness from the insert film and PC the toughness measured from a pre-crack. The quasi-static Mode II test [57] reports NPC and PC toughness data from round robins on two carbon-fiber reinforced and one glass fiber-reinforced laminate with four to nine participating laboratories. Averaging the coefficients of variation from the participating laboratories yields an average value between 3.1% and 6.5% for the NPC data and between 3.8% and 5.9% for the PC data. The repeatability values, r, (precision within a laboratory) and reproducibility values, R, (precision between laboratories) for NPC tests were between 9.2% and 19.0% (for r) and between 9.6% and 19.0% (for R). The respective values for PC were between 11.0 and 17.1% (for r) and between 12.1% and 20.2% (for R). Test data for Mode II from four laboratories testing five specimens each reported in [58] indicate coefficients of variation between 8.5% and 22.2% for the 5% initiation point. Interestingly, this might depend on the method of analysis, with the corrected beam theory with effective crack length (from specimen compliance) yielding the lowest value (8.5%). Visual initiation results in coefficients of variation between 17.6% and 25.8%, are likely to reflect the operator variability in the observation (see discussion below). These values are consistent with the observation that Mode I coefficients of variation tend to be lower than those for Mode II. For Mixed Mode I/II tests, the standard [59] currently does not provide a precision statement. In addition to the precision data in the standards, additional data for Mode I fatigue fracture may be found, e.g., in [60,61].

Considering Figures 2 and 3, it can be asked how much scatter is introduced into the average values or the fitting parameters of the cyclic fatigue fracture curves in the Paris graph by differences in the precrack length before applying the cyclic load. For the measurement of quasi-static Mode II toughness of composites [57] there is an alternative test method [58]. However, round robin data comparing the two standard tests (as well as a further two, namely the four-point end-notched flexure and the stabilized end notched flexure tests) are only available from a preliminary study with limited participation performed during the development of the standards. The results [62] indicate similar scatter between 15-20% in terms of reproducibility (excluding one data set of the end-loaded split test with a coefficient of variation of 73%), but strongly varying repeatability in different laboratories (between 6 and 24%).

Based on the measurement resolution required by the standard fracture test procedures (typically of the order of 1% of the measured quantity) Gaussian error propagation applied to the equations for data analysis suggest that experimental scatter in the calculated toughness values should be about 4-5% [63]. This is comparable to experimental scatter in other mechanical properties. Typical scatter (coefficients of variation) for longitudinal modulus measurements collected from literature are about 10% for E-glass thermoplastic composites, about 2.4% for carbon fiber thermoplastic composites, and about 1.5% for carbon fiber epoxy composites [64]. The observed scatter of 10-20% in toughness values is therefore likely to arise from other sources. Selected examples discussed in [63] indicate that operator-related effects play a significant role. On one hand, manual preparation and processing of laminates contribute to variability in material morphology and hence laminate properties, for this [65] refers to "intrinsic scatter". A knowledge of intrinsic scatter is important for assessing the toughness data of a given material. The other factors contributing to so-called "extrinsic scatter" shall be minimized as far as possible. Among these are decisions about the choice of test set-up and test machine as well as data recording (e.g., play in load-train, load cell range, visual delamination length recording, sampling rates) that determine the measurement resolution. A third and not negligible factor is manual data analysis. A round robin where participants analyzed an identical load-displacement curve yielded scatter (reproducibility) of 4-5% in the determination of the non-linear initiation point [63,66]. Synchronization between the load, displacement and delamination length measurements, essential for data analysis, is probably also operator dependent.

3.7 Possible approaches to reduce scatter

Considering the three main sources of scatter: firstly, the material inhomogeneity and defect concentration from manufacturing and processing; secondly the test apparatus with its specific measurement resolutions; thirdly the manual, operator-specific data analysis (even if semi-automated by the use of spreadsheets for the calculations), the following approaches may contribute to reducing the scatter or at least yielding more consistent data. (i) Process automation in laminate manufacturing and the increasing contribution of artificial intelligence will reduce the number defects and material variability and will hence yield more consistent material properties [67,68] by reducing intrinsic scatter. (ii) Digital tools (e.g., fitting of digital data) and test automation will also improve data acquisition and analysis, i.e., will reduce extrinsic scatter, [69]. (iii) Implementing artificial intelligence approaches [70,71] to composites manufacture and processing, see, e.g., [72,73] as well as to data analysis, see, e.g., [69,74,75] is currently explored in several research projects, so may simultaneously reduce intrinsic and extrinsic scatter. Hence, prospects for reducing scatter in measured toughness values are bright.

4 CONCLUSIONS

Activities to develop standard (quasi-static and fatigue) fracture toughness tests for fiber reinforced polymer composites have identified a number of challenges that require solving before the resulting toughness values are applicable for design purposes. The development of satisfactory approaches to accommodate the phenomena of fiber bridging and multiple cracking are under development as discussed. Additional test standards also have to accommodate two-dimensional delamination and delamination at other than ambient conditions, typically for the different service environments. The accuracy of such standards is critically important for obtaining input values for material simulations. In addition, repeatability and reproducibility of these material parameters characterizing fracture are of great importance. Developments in materials manufacture and processing using automated techniques and artificial intelligence will produce materials with more consistent properties and associated reduction in intrinsic scatter. Further, the use of automated test technique and digital analysis, together with artificial intelligence, will reduce scatter in raw data and calculated parameters, with an associated reduction in extrinsic scatter. Simulations using these parameters will also be essential to reduce the significant test efforts currently required for the design of damage-tolerant composite structures. In combination, the prospects for the development of highly relevant and accurate standards are bright, although challenges remain.

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