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Juan Pablo Márquez Costa. Review of fracture characterization by delamination and debonding of polymer composite materials and bonded interfaces subjected to high temperature. *Mechanics of Advanced Materials and Structures*, In press, <10.1080/15376494.2025.2466075>. <hal-04973238>

HAL Id: hal-04973238

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Submitted on 2 Mar 2025

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Review of fracture characterization by delamination and debonding of polymer composite materials and bonded interfaces subjected to high temperature

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Preprint submitted to Mech. Adv. Mater. Struc.

Review of fracture characterization by delamination and debonding of polymer composite materials and bonded interfaces subjected to high temperature

Abstract: Polymer composite materials and bonded composites are now widely used in the design of major aircraft components due to their high specific properties and feasibility for joint assembly. Nevertheless, there are fire-related risks, which can significantly alter their thermal behaviour. High temperatures directly influence the thermochemical and mechanical degradation of composites and bonded layers, leading to the apparition of damage phenomena such as delamination or interfacial cracking at the interface, and debonding at the bonded layer. The characterisation of delamination and debonding in temperature remains critical to ensuring the integrity of aeronautical structures. This review aims to describe the mechanics leading to delamination and debonding as a consequence of thermomechanical loading. A recompilation of experimental tests used to characterise the mechanical properties of composite interfaces and bonded layers is provided. The adaptation of standard tests to high-temperature conditions is discussed, along with key findings from the literature. Limited research has focused on the characterisation of fracture properties at elevated temperature due to the challenges associated with conducting experimental tests and analysing the resulting data. Finally, a discussion about the main findings and the need for further advancements in delamination and debonding characterisation at high temperatures is presented.

Keywords: composite; bonded composite; delamination; debonding; high temperature; thermomechanical loading; experimental characterisation

1. Introduction

The study of polymer composites and bonded composites exposed to high temperatures is critical to ensuring the safety of structures and preventing their failure in extreme environments **Erreur ! Source du renvoi introuvable.** Figure 1 illustrates some examples of components of structures in the aeronautical sector presenting a fire risk [2],[3]. Fire risks must be anticipated in order to adapt the stages of design, prevention and certification of structures and composite components subjected to high-temperature conditions. This approach aims to reduce the current safety margins, which are necessitated due to limited experience and feedback, and to address new challenges associated with the use of these materials.

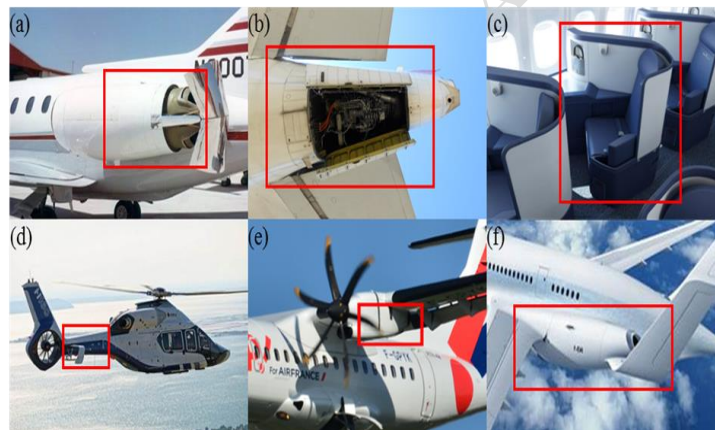


Figure 1. Fire risk zone in components of aeronautical structures [2],[3]. From left to right and top to bottom: (a) Nacelle of a turbofan engine, (b) APU system cover, (c) cabin seat, (d) helicopter anti-torque rotor support structure, (e) embedding the engine in the aircraft wing and (f) embedding the engine in the aircraft fuselage.

To ensure the integrity of structures, certification and regulation aim to establish standard tests and criteria for the design and validation processes of aeronautical and aerospace structures made from materials that may be exposed to fire or high temperatures [3]. The regulations of fire safety in civil aviation are administered by the

Federal Aviation Administration (FAA) [4] in the United States and by the European Union Aviation Safety Agency (EASA) [5] in Europe. These international standards define requirements for various potential fire scenarios, including those occurring during the parking, the cruise and the post-impact phases. The most critical scenario arises when multiple events interact, particularly structural degradation combined with the spread of flames and smoke into the passenger cabin [6]. The certification of structures containing non-metallic materials (such as composite materials or bonded joints) is governed by various aeronautical standards established by the FAA and the EASA. Nevertheless, for specific applications (*e.g.*, thermal isolation), certification standards remain highly generic and apply equally to metallic and non-metallic structures, including composites [3]. That means that standard certification tests do not differentiate between metallic and composite materials, focusing instead on fire reaction and the structural strength [7].

In this context, previous studies have shown that composites structures, including bonded composites, exhibit better fire resistance than metallic ones [3]. However, their degradation process is more complex due to the thermo-physio-chemical interactions of polymer composites and the phenomena involved in a fire scenario [1], which can significantly impact the mechanical performance of the final structure. As a result, certification standards should be reviewed to incorporate a more accurate description of material degradation. It is therefore essential to consider fire risks in the behaviour of particularly complex composite materials or bonded composites at high temperatures. This will aid in standardizing safety standards and adapting certification processes accordingly.

Numerous researchers have studied the thermal degradation of composite materials subjected to high thermal fluxes [8]-[13], and its effects on mechanical strength [15]-[19]. Among these, the work of Mouritz et al. [15],[16],[20],[21] have made

significant contributions to understanding the degradation mechanisms involved. Their research encompasses both experimental methods for characterisation and models for simulation. Mouritz et al. developed techniques for characterising composite materials exposed to fire and identified a complex degradation and damage scenario, as illustrated in Figure 2 [1].

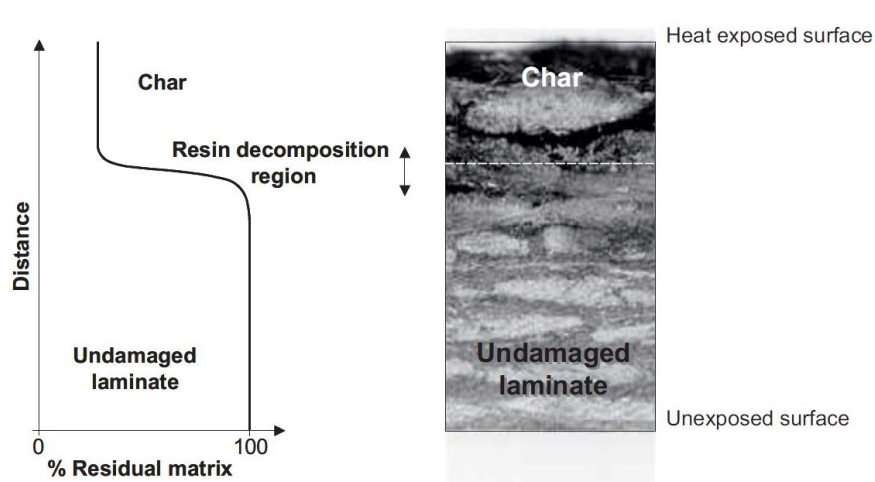


Figure 2. Scenario of the various damage zones caused by a fire aggression and diagram of the residual mass through the thickness of the composite material [1].

Regarding the damage scenario, these authors have demonstrated that damage initiation occurs at the interface layer, leading to catastrophic delamination. However, previous characterisations are often performed at the macroscopic scale rather than at the material scale. This makes it challenging to accurately characterise delamination by phenomenological approaches based on thermal degradation tests, such as the calorimetric cone or the heat flow radiant panel [3],[22]. In the case of bonded composites, both the bonded layer and the composite interfaces are susceptible to failure [23]. The complexity of a bonded assembly makes it difficult to accurately explain its failure based on macroscopic tests, particularly under high temperature conditions [23],[24].

For these reasons, a deeper understanding of fracture characterisation testing is necessary to overcome the lack of knowledge regarding degradation and damage mechanisms at elevated temperatures, which can lead to delamination at the interfaces between composite plies or in the bonded layer on bonded composites. This paper reviews experimental tests for characterising delamination and debonding, as well as the mechanical properties of interfaces. The review focuses on: (i) the mechanisms involved in interface degradation as a result of thermomechanical material degradation and (ii) the experimental tests and devices used for characterising delamination and debonding in under elevated temperature. Thus, the proposed structure is as follows:

- Section 2 describes the mechanisms that occur during the scenario of material degradation induced by its exposition to fire or high temperatures.
- Section 3 emphasises on fracture mechanisms for both delamination and debonding, explaining lastly the failure scenario in temperature from a multi-scale point of view.
- Section 4 presents studies focused on material characterisation by experimental tests conducted at room temperature with the aim to overcome the complexity of fracture mechanics at the material scale.
- Section 5 details the adaptation of existing tests to elevated temperature conditions and highlights the need for a more in-depth analysis of experimental results than currently proposed in existing standards, which are not designed for high-temperature applications.
- Section 6 discusses the main advantages and limitations of standard tests currently available in the state of the art, with particular emphasis on the maturity of experimental setups for addressing nonlinear effects involved in the fracture characterisation of composites in high-temperature environments.

- Section 7 provides conclusions on the relevance of experimental testing.
- Section 8 outlines perspectives for innovative test setups and data analysis.

2. Mechanisms in composites subjected to fire or high temperatures

The fire resistance of composites and bonded composites is strongly influenced by the thermal and mechanical properties of the materials. Moreover, the fracture mechanisms leading to failure evolves differently depending on the operating temperature and the level of applied loading [23]-[25]. This dependency is a consequence of the time- and temperature-dependent behaviour of composites and bonded layers. To better understand this thermomechanical coupling, this section aims to synthesize the physical phenomena involved in the thermomechanical degradation of composites.

The complexity of studying the thermomechanical degradation of composites exposed to a flame arises from the variety of thermally, chemically and mechanically induced phenomena, as well as their strong interactions [3]. Figure 3 presents a diagram which illustrates the most significant phenomena, along with examples of induced mechanical damage, resulting from the exposure of a polymer matrix composite to a flame [21].

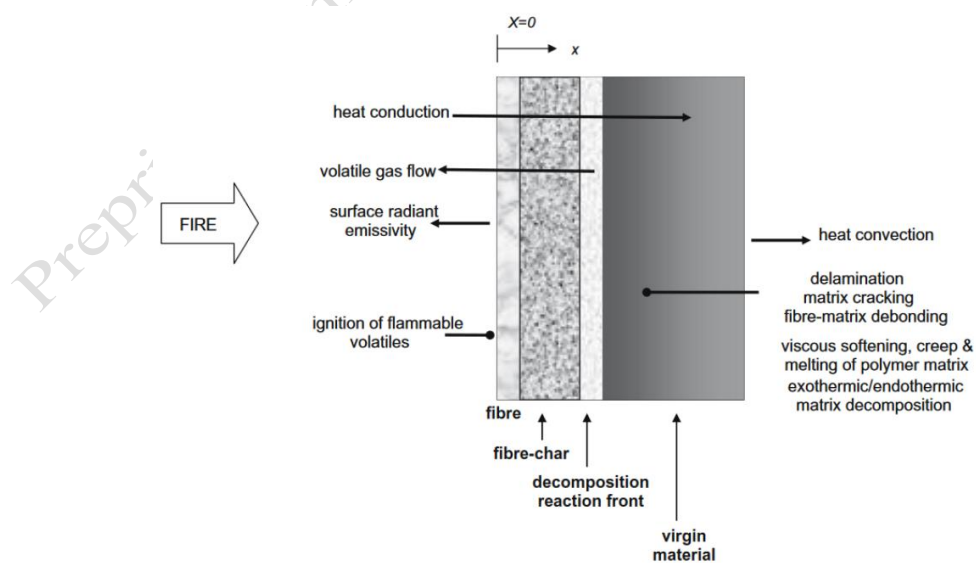


Figure 3. Diagram of thermomechanical phenomena associated with thermal degradation of a polymer composite subjected to fire [21].

These couplings complicate both the characterisation of the phenomena and their modelling, and consequently the sizing of the structure, as noted by Mouritz et al. [21]. The main phenomena that occur when a composite or a bonded composite is exposed to fire include: heat conduction; heat exchanges by convection and radiation; thermal expansion or contraction, decomposition of the polymer matrix and/or the bonded layer; production of gases and carbonaceous residues (commonly known as char) through pyrolysis (Figure 4a [21]), which increases the porosity and internal pressure of the material (Figure 4b [21]); combustion feeding (Figure 4c [3]); delamination (Figure 4d [21]); fibre and carbon decomposition (Figure 4e [21]); blistering (Figure 4f [3]); and ablation.

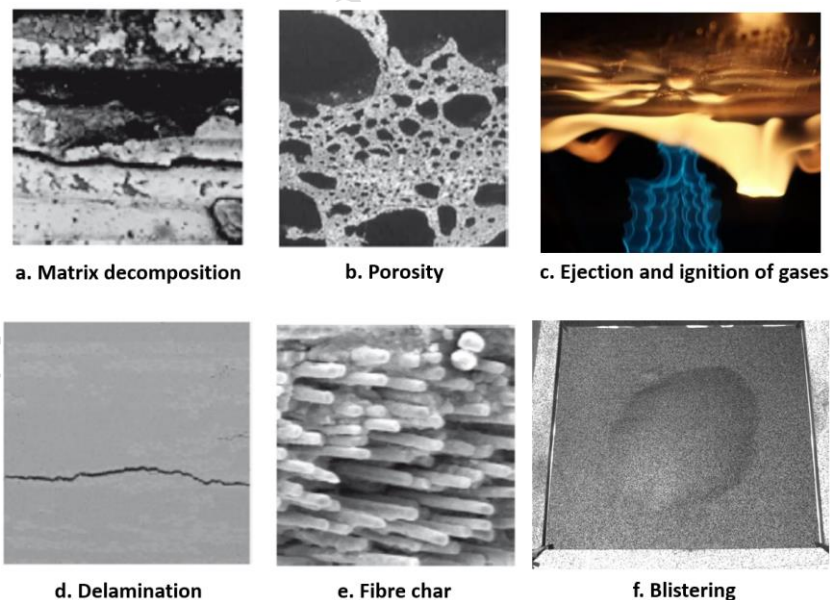


Figure 4. Views of thermomechanical phenomena induced by thermal degradation of a composite: (a) Micrograph of polymer matrix decomposition [21], (b) Micrograph of polymer matrix porosities [21], (c) Ignition of ejected gases as a consequence of the

surface degradation of a polymer composite [3], (d) micrography of delamination [21], (e) micrography of oxidation of fibres [21] and (f) photography of blistering on the back surface of a polymer composite [3].

These phenomena do not have uniform consequences on the thermal and mechanical responses of the material, and by extension, on the structure. The thermal degradation of the material leads to several processes: (i) thermochemical decomposition of the matrix through pyrolysis and (ii) production of volatile gases, which are expelled towards the exposed surface of the structure and subsequently feed the flame. Consequently, the thermal loading experienced by the composite structure may be altered due to the interactions between the heterogenous flame and the structure, as well as the anisotropic behaviour of the material. It is important to note that this complexity at a macroscopic scale results in catastrophic failure mechanisms at both the structural and the material scales.

On the one hand, at the macroscopic scale of the laminate, the most significant mechanical phenomena observed are ablation and blistering [3],[26]. Ablation of the composite surface occurs at very high temperatures (above 900°C) as a result of chemical reactions and mechanical erosion caused by the flow of reaction gases along the surface [8],[27]. Blistering (Figure 4f [26]) is primarily induced by two main factors: (i) the increase in internal pressure within the material due to thermal degradation and (ii) mechanical loading generated by the thermal gradient, particularly as a result of thermal expansion at various stages of material degradation.

On the other hand, when the interaction between the flame and the laminate are neglected, mechanisms such as delamination (Figure 4d [21]) can be observed, as well as complex loadings at the material scale, which can nevertheless induce significant consequences on the macroscopic response of the structure [1],[15],[17]-[20]. In the case

of bonded composites, layer separation occurs at the bonded interface and is referred to as debonding [23]. These mechanical phenomena at the material scale are also the result of various interacting physical mechanisms. At this scale, three primary mechanisms associated with thermomechanical loading can be distinguished: thermal expansion, thermal degradation and internal pressurization [3].

Thermal expansion at the material scale is a mechanical phenomenon associated with temperature variations [3],[10]. It results from the thermal behaviour of the material, which undergoes expansion as the temperature increases at a given state of degradation. For anisotropic materials, it is necessary to identify different coefficients of thermal expansion for the fibres and the matrix, or directly for the laminate, depending on the fibre orientation [21]. In the case of bonded composites, separate coefficients must be identified for the plies and the adhesive layer [23],[24].

The thermal degradation of the polymer matrix and/or the adhesive layer can be divided into two successive stages: (i) decomposition of the matrix by pyrolysis (Figure 4a [21]), accompanied by the production of porosity (Figure 4b [21]), and (ii) decomposition of carbonaceous species (char and fibres) by oxidation (Figure 4e [21]). The production of gases and char by pyrolysis is a direct consequence of this decomposition process. Organic matter degrades with increasing temperature, yielding two primary reaction products: carbonaceous residues (char) and decomposition gases. Subsequently, the decomposition of carbonaceous species (fibres and char) occurs due to oxidation reactions between oxygen and the charred residues formed during matrix pyrolysis, as well as with organic fibres. As highlighted in the literature [21], thermal degradation induces several significant effects at the material scale: (i) alteration of thermal properties [16], (ii) degradation of mechanical properties [17]-[19], (iii) internal pressurisation [3],[10],[26], and (iv) disruption of the thermal behaviour due to heat

conduction, heat exchange by convection and radiation, and gas transport within the material [9],[11].

The mass loss resulting from thermal degradation by pyrolysis also leads to the formation of pores in the matrix [1],[10],[28]-[30]. These pores facilitate the accumulation of decomposition gases, with internal pressure depending on the quantity of accumulated gas, the temperature of the medium and the pore volume. This internal pressure represents a source of damage in the material volume which can induce either expansion or contraction [1],[3].

The mechanisms described above are significantly influenced by the anisotropy of the material. Thermal conduction is anisotropic, resulting in varying temperature gradients depending on the fibre orientation. Strong temperature gradients through the thickness, combined with the thermal degradation of mechanical properties, thermal expansion and internal pressure, can lead to cracking and delamination in composites, or cracking and debonding in bonded composites [20],[23],[24]. Such damage typically occurs between plies in composites or at the bonded layer when two composite adherends are joined. In both cases, delamination and debonding are generally catastrophic, often leading to the final failure of the material.

3. Fracture mechanisms induced by thermomechanical loading

This section firstly describes delamination and debonding of composites. Subsequently, a detailed description of the failure scenario induced by elevated temperatures is provided.

3.1. Delamination of composites

Delamination occurs between the plies, as illustrated in Figure 5. It is a mechanical phenomenon driven by the internal stress field at the interfaces between the plies.

According to Mouritz and Gibson [25], delamination at high temperature is primarily caused by the pressure exerted by gases accumulated in the pores. These gases promote matrix cracking and its propagation along the interfaces leading to the initiation of delamination. Delamination can occur both along the degradation front and in non-degraded regions [20],[25]. Several authors [3],[20],[25],[26] have associated delamination with stress and strain gradients induced by strong thermal gradient through the thickness, material anisotropy, and the resulting thermal expansion. This underscores the importance of mechanisms at the material scale which are directly linked to the thermomechanical degradation of the material and its mechanical response, particularly in relation to the initiation of delamination.

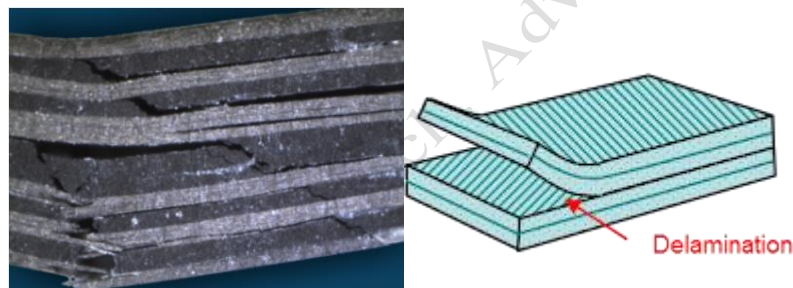


Figure 5. Delamination at the interfaces (left; courtesy of LAMIH laboratory) between two adjacent plies of a composite and its schematic representation (right).

Beyond the issue of fire resistance, delamination represents a major challenge in thin structures commonly used in the aeronautics sector due to the loss of stiffness it induces, particularly under compression [31],[32]. In addition, this mechanism has a notable impact on the apparent thermal properties of the material. The thermal conductivity of the material can drop substantially depending on the extent of the delaminated surface and its through-thickness position. Delamination acts as a thermal barrier upstream of the delaminated surface. As a result, the upstream region may experience accelerated thermal degradation, potentially increasing the gas flow towards the edges without delamination. This interaction can influence the flame dynamics and

consequently, alter the distribution of the incident heat flow on the exposed surface [25],[26].

3.2. Debonding of bonded composites

Debonding occurs when the adhesive bonded layer between two composite adherents fails to adhere. In the context of material fracture, debonding is considered a rupture mechanism and can manifest in three different modes [23]: (i) adhesive debonding, (ii) cohesive debonding and (iii) mixed debonding. Adhesive debonding occurs at the interface between the adherent and the adhesive bonded layer, while cohesive debonding takes place within the adhesive bonded layer itself. Mixed debonding is a combination of both adhesive and cohesive modes. These cases are illustrated in Figure 6 **Erreur ! Source du renvoi introuvable.** When bonded composites are exposed to high temperature, the type of debonding depends on both the degradation of the surface preparation and the strain and stress field at the interfaces, including the bonded layer and the adherent/adhesive interface [23],[24].

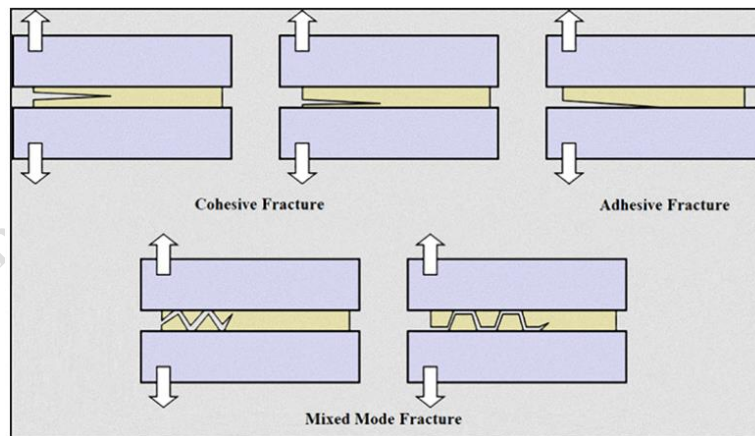


Figure 6. Different kinds of debonding at the interface between two adjacent composite adherents and its schematic representation [33].

As explained by Blackman, the failure of bonded composite joints may also result from delamination of adjacent plies near the bonded interface [34]. This occurs because

the adhesive interface is typically more toughened than the adherents. In more complex cases, debonding may initiate within the bonded layer and subsequently migrate into the composite adherent. For thin aeronautical structures, when the surface preparation of composites adherents is sufficiently effective to prevent adhesive failure and precedes cohesive failure, debonding tests can resemble delamination tests.

3.3. Fracture mechanisms in temperature

Temperature accentuates various mechanisms (*e.g.*, viscous effects, plasticity) at the plies, the interface and/or the bonded layer, which can significantly influence the material behaviour. Consequently, the damage evolution leading to delamination and debonding may be strongly affected by temperature. The following paragraphs describe the primary causes of delamination and debonding in composites and bonded composites to enhance the understanding of these phenomena under high-temperature conditions.

3.3.1. From material to fibre/matrix scales

For composite materials subjected to thermal attack, delamination is often observed throughout the thickness of the composite, occurring in either thermally degraded or virgin regions. This type of damage has been reported under exposure to flame [3],[15],[28],[35]-[39] as well as laser impact [3],[26]. According to Mouritz [1] delamination may be confined to the pyrolysis front (*i.e.*, the transient degradation zone) or may also occur in regions of the underlying undamaged material. Delamination resulting from thermomechanical loading can originate from intraply matrix cracking at the fibre/matrix scale or from decohesion of adjacent plies at the interply interface [23], as illustrated in Figure 8.

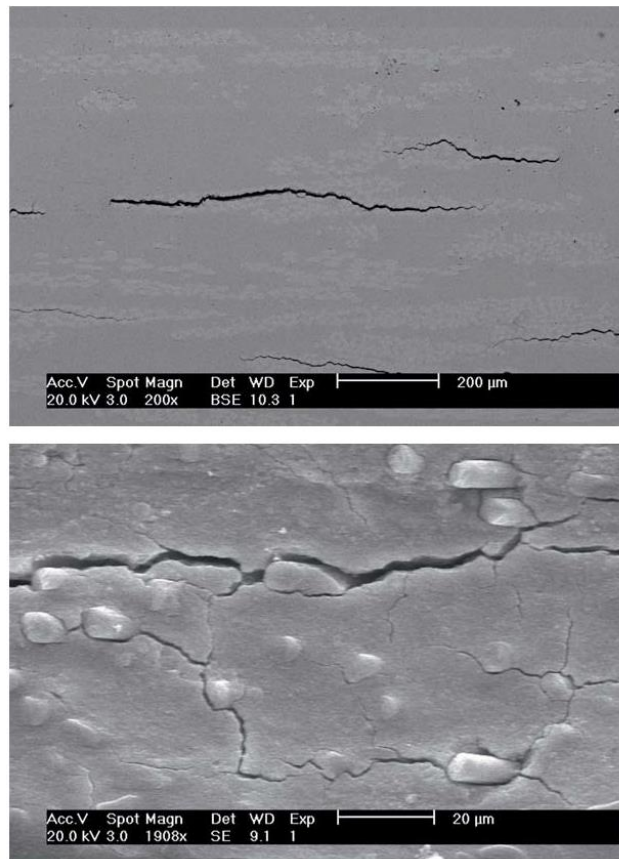


Figure 8. Interply delamination (at top) and matrix cracking (at bottom) for a composite exposed to fire [1].

Mouritz [1] states that the temperature in the delamination cracking region is significantly higher than the glass transition temperature of most polymer systems used in composites. As a result, delamination can be explained from a thermal point of view, as a combination of increased pore pressure and matrix softening. Some authors [1],[15],[28],[35]-[37],[40] partially attribute these cracking mechanisms to the rise in internal pressure within the material caused by the accumulation of volatile gases and, in certain cases, the vaporisation of trapped moisture [13],[14]. In addition to this internal loading, the pronounced temperature gradient through the material thickness induces a thermomechanical loading due to the differences in mechanical behaviour between adjacent plies [1],[3].

3.3.2. *The ply/interface scale*

The consequences of delamination and cracking within the material are diverse and can be categorised into two main families, both of which significantly influence the thermal response of the material. The first category pertains to the retardation of thermal decomposition due to delamination at the interfaces. Delamination leads to the increasing of the thermal resistance at the interfaces, thereby reducing thermal conduction within the material. The second category relates to changes in the material permeability due to the presence of cracks. These cracks alter the interaction between the material and the heat flow, resulting in a modification of the thermal stress experienced by the material. This accelerates thermal degradation in the surface exposed to thermal loading. Moreover, cracking provides pathways for the rapid release of pyrolysis gases [41] which can feed the flame as discussed in the Introduction. Additionally, when laminates are positioned over a flame source or oriented vertically, delamination may cause plies to eject, exposing the virgin material to fire [1]. However, the authors agree that the magnitude of these effects on the thermal response, and consequently on the mechanical response, remains poorly quantified due to the complex coupling of the involved physical mechanisms.

To address the complexity of this problem and better control the occurrence and effects of delamination, Biasi and Leplat et al. [8],[26] employed a laser flux as a substitute for a flame to thermomechanically stress composite laminates. This approach eliminates the coupling between the flame and the material, allowing a focused study on the thermomechanical degradation of the material. Figure 9 shows the damage scenario for a laminated composite used in aeronautical applications, composed of T700GC carbon fibres and M21 epoxy thermosetting matrix. It is reinforced with thermoplastic nodules to improve compressive strength after impact [3]. The stratification under consideration is quasi-isotropic (QI) with 16 plies and a total thickness of 4.16 mm. In the red frame of Figure 9, the first delamination is observed near the char region, located

at the interface within a 90° delaminated ply. This intraply interface is attributed to the high grammage of the ply and the presence of thermoplastics nodules [3],[26]. A close-up view of the upstream region of the delamination reveals diffuse damage within this interface. This damage is characterised by matrix cracks approximately one hundred microns in size. Similar to delamination caused by low-speed and/or low-energy impact, the most important delamination is highlighted in the blue frame. It occurs near the rear surface at the interply interface between the 45° and 90° plies. This delamination develops at a temperature below the glass transition temperature (T_g) which is approximately 573 K for the M21 epoxy resin.

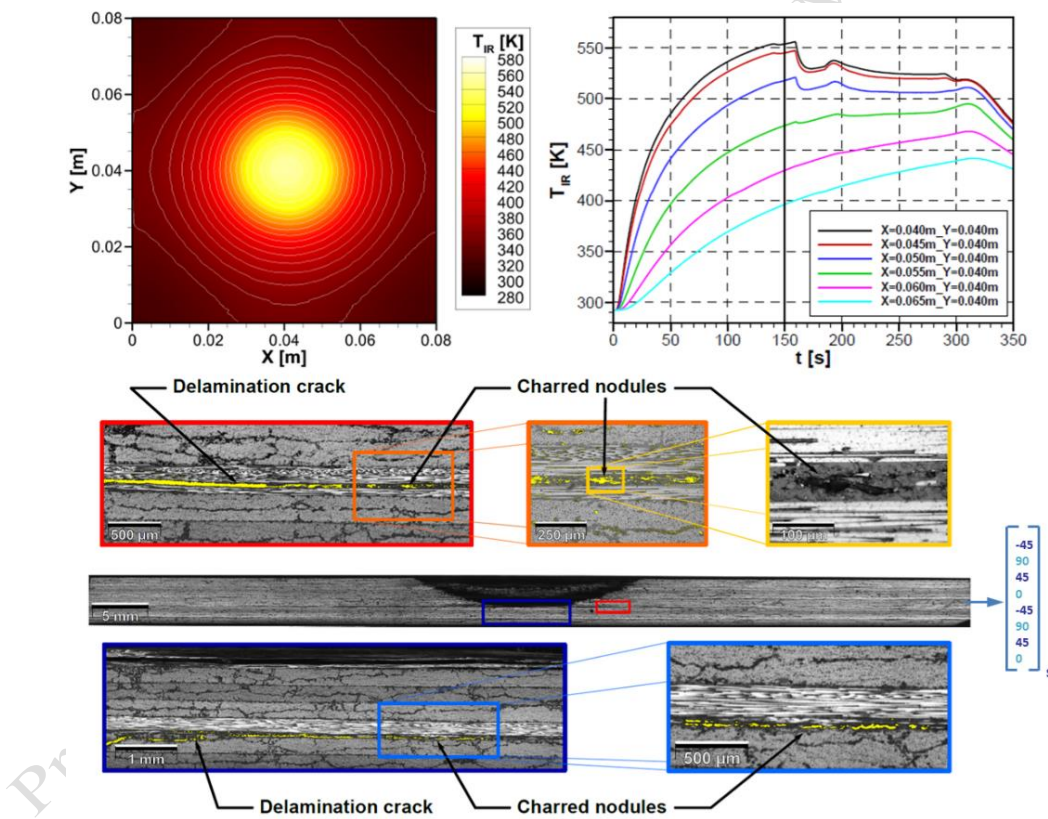


Figure 9. Thermal response on unexposed surface measured by infrared thermography. At left: instantaneous temperature maps at $t = 150$ s. At right: temperature extracted over time at different locations from the centre of the coupon. Influence of charred nodules on the crack onset of delamination. [3],[26].

Regarding the propagation of delamination at elevated temperatures, it is predominantly observed in mode II, even though the apparent crack opening may seem to correspond to mode I. The mechanisms of mode II are inherently more complex to analyse than those of mode I. This is primarily due to the apparent crack closure that occurs during mode II crack propagation [3].

For thin bonded composites, the interface layer acts as a bonding element between the composite adherents [23]. Consequently, the composite arms respond similarly to thermal and thermomechanical loads. The primary distinction lies in the fact that the bonded layer exhibits a nature distinct from that of a typical interply interface [24]. Therefore, its local behaviour is expected to significantly influence the interface damage mechanisms leading to debonding and fracture [23],[24],[42],[43]. This underscores the necessity of studying the effects of temperature and the mechanical loads induced by thermal exposure on delamination and debonding.

The strong interactions between delamination or debonding and the mechanisms discussed in the previous section make the analysis of the fire resistance of composites highly complex. This complexity emphasises the critical importance of conducting appropriate tests to ensure the characterisation of the influence of temperature on delamination and debonding. It is therefore essential to assess the interface properties, as presented in the following section.

4. Experimental characterisation of delamination and debonding

Delamination and debonding leads to a consequent degradation of the residual mechanical strength of the laminate or the bonded composite respectively. These mechanisms are a consequence of a set of thermophysical mechanisms, nonlinear mechanical behaviours and matrix cracks at the microscopic scale which will favour the appearance of delamination at interfaces [44]-[46] or debonding at the bonded layer

[23],[24],[34],[47],[48]. Therefore, it is very important to choose the relevant tests to characterize the desired properties and mechanisms, notably: the crack onset strength and the energy release rate. This is crucial in order to define failure criteria for the interfaces of both bonded composites and composites. In this Section, a review of available experimental standardised tests applicable for characterising both delamination and debonding at room temperature is exposed. It aims to highlight the difficulty to separate each contribution of mechanisms because of the material behaviour, even when temperature influence is neglected.

4.1. Typical failure scenario: Crack initiation and propagation

Both delamination and debonding are damage and fracture mechanisms that involve the loss of strength at the interface between two parts: adjacent plies of a laminated composite in the case of delamination [35],[49]-[52] and composite adherents in the case of debonding [23],[24],[33],[34],[47],[48]. These mechanisms can occur due to stress fields acting normal to the interface (causing tensile stresses), tangential to the interface (causing shear stresses) or a combination of both. Apparition of delamination or debonding can arise from various factors [44]-[46],[50]-[52], including material heterogeneities, internal residual stresses [53], porosity and defects introduced during manufacturing, free edges effects or impact loading.

Delamination can be divided into two main stages:

- Crack onset. This refers to the initiation of the crack. Crack initiation is characterised by an unstable dynamic phenomenon that results in the formation of a crack of a finite length [44];
- Crack propagation. This refers to the progression of the crack along the laminate interface or the bonded layer. Propagation can be either stable or unstable depending on the applied loading or the geometry of the test specimen.

The crack onset and propagation phases are illustrated in Figure 7 [54]. These phases can occur in a pure fracture mode or a combination of multiple modes. The pure modes of fracture are defined as follows:

- Mode I: opening mode induced by tensile loads perpendicular to the interface.
- Mode II: Sliding mode caused by in-plane shear loads parallel to the interface.
- Mode III: Tearing mode resulting from an anti-plane shear loads perpendicular to the plane of the interface.

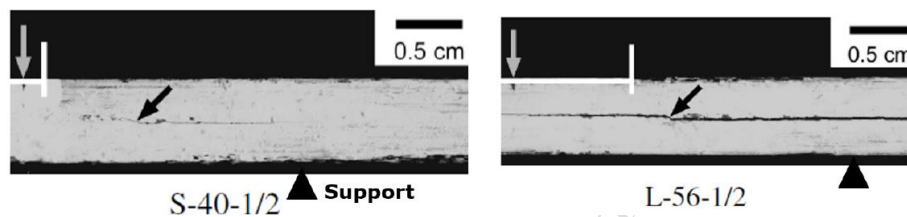


Figure 7. Representation of crack onset and crack propagation in a composite subjected to bending load [54].

Each pure mode is typically characterised by the resistance to crack initiation and the critical energy release rate or tenacity leading to crack propagation [35],[44],[55]. Since delamination and debonding are damage and fracture mechanisms that regularly lead to structural failure, numerous researchers have focused on the analysis of these phenomena, particularly under purely mechanical loading conditions. This highlights the critical importance of characterising the initiation and propagation of delamination and debonding to incorporate their effects during the design stages of composite structures.

4.1. Crack onset characterisation

The initiation of delamination and debonding at a non-singular interface or bonded layer is associated with interlaminar resistance of the interface [3],[44] or the adhesive bonded layer [34]. This resistance is characterised by either tensile (opening) strength (S_{33}), out-

of-plane or longitudinal shear strength (S_{13}), or in-plane transverse shear strength (S_{23}), corresponding to failure modes I, II or III respectively.

4.1.1. Out-of-plane tensile strength

The Interlaminar Tensile Strength (ILTS) characterises crack initiation in pure mode I. This mechanical property can be determined through a 4-point bending test on an angle specimen [56],[57], also known as the Curved Beam Strength (CBS) test as described in the standard ASTM D6415 [58]. Figure 10 shows the application of this test on a composite angle specimen. The identification of S_{33} is computed using either finite element analysis or analytical calculation.

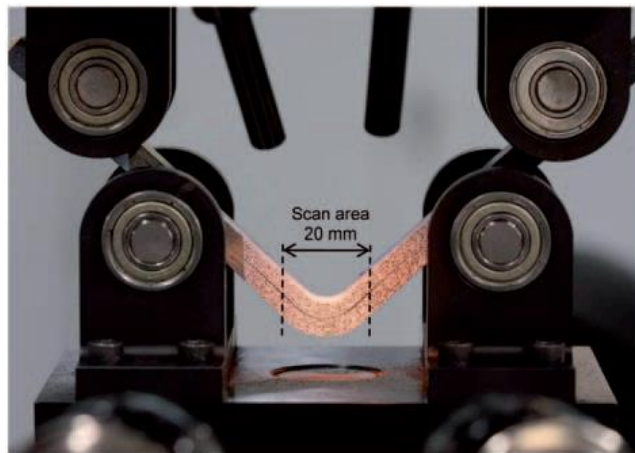


Figure 10. Application of standard ASTM D6415 for the 4-point bending test on a composite angle specimen to identify the ILTS [57], [58].

Makeev et al. [57] highlighted the efficacy of the 4-points bending test in obtaining the ILTS for unidirectional IM7/8552 carbon fibre reinforced plastic (CFRP) composite with epoxy matrix. Their results revealed that the ILTS is sensitive to individual voids at critical locations, which leads to lower ILTS values. Yavuz et al. [59] compared ILTS values for CFRP laminates with $0^{\circ}/0^{\circ}$ and $45^{\circ}/-45^{\circ}$ ply orientations. They observed lower ILTS values for the $45^{\circ}/-45^{\circ}$ configuration due to its higher resin content in the interlaminar region.

4.1.2. Out-of-plane shear strength

The Interlaminar Shear Strength (ILSS) characterises crack initiation in shear modes. These mechanical shear properties (S_{13} in mode II and S_{23} in mode III) can be achieved using several tests:

- The Iosipescu test: this test involves asymmetric 4-point bending on a V-notched specimen to promote out-of-plane shear failure [60]-[62]. It is detailed in the standard ASTM D5379 [63]. The test setup is shown in Figure 11;
- The Short Beam Shear (SBS) test: a 3-point bending test is conducted on a short specimen relative to its thickness and clamped between supports [64],[147]. It is defined in the standard ASTM D2344 [65] and its setup is shown in Figure 12;
- The angle iron folding and unfolding test [66]: this test determines the ILSS in mode II (S_{13}) for unidirectional laminates, and the ILSS in mode III (S_{23}) for laminates with different stacking sequences including $90^\circ/90^\circ$ plies in the failure region. However, no standard exists FEA is required to The test setup is shown in Figure 13 [66];
- The Double Notch Shear (DNS) test [66],[67]: this test uses a double-notched specimen to measure the in-plane shear strength. It is described in the standard ASTM D3846. This test presents two variants depending on the kind of applied load: the Double Notch Compression (DNC) test and the Inclined Double Notch shear (IDNS) test, which allow to identify the out-of-plane ILSS. The test setup is shown in Figure 14.

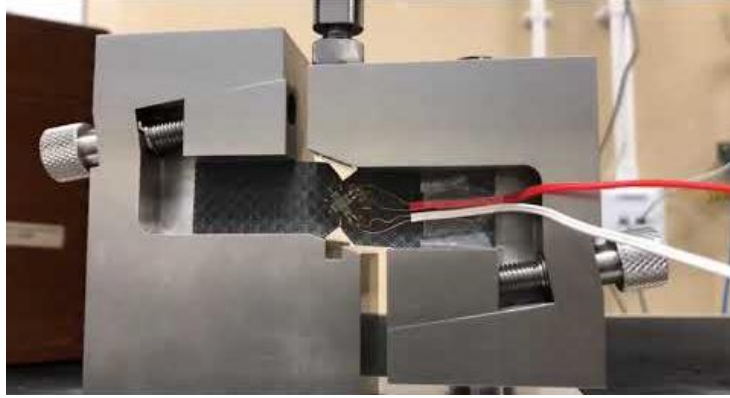


Figure 11. Application of standard ASTM D5379 for the Iosipescu test on a composite to identify the ILSS [63].

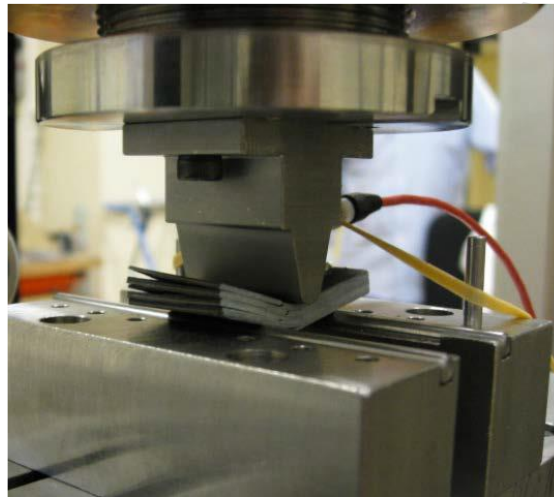


Figure 12. Application of standard ASTM D2344 for the Short Beam Shear test on a composite to identify the ILSS [65],[66].



Figure 13. Application of standard ASTM D3045 for the Tensile test on a composite to identify the ILSS [67].

Figure 13. Application of the angle iron folding and unfolding test on a composite to identify the ILSS [66],[67].

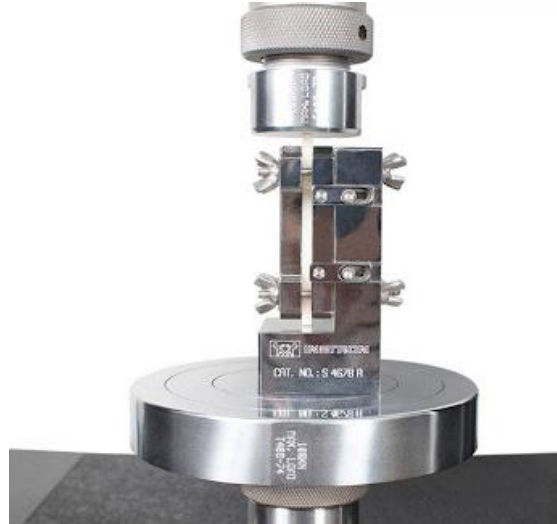


Figure 14. Application of standard ASTM D3846 for the Double Notch Shear test on a composite to identify the in-plane shear strength (or the ILSS using the DNC or the IDNS variants) **Erreur ! Source du renvoi introuvable.**,[67].

Each standard specifies a unique test protocol along with a method for calculating interlaminar strength. The Iosipescu test [63] does not guarantee pure shear at the interface and requires a sufficiently thick specimen, which may limit the representativeness of the measured strength. The SBS test provides a strength value calculated through an analytical function [65]. However, this function is based on the beam theory and assumes a uniform stress distribution, which is valid only for isotropic materials. The DNS test **Erreur ! Source du renvoi introuvable.**,[67] presents challenges due to the complexity of the double-notch setup. Conversely, the angle iron bending and unfolding test is relatively straightforward in terms of both experimental setup and the analytical or finite element analysis required for interpreting the results [66].

Allott et al. [68] measured the ILSS of the IM7/8552 composite and evaluated the influence of specimen thickness to assess the validity of small-scale SBS tests. They observed a little deviation between ILSS values obtained from 1/6-scale tests and the full-scale tests, as presented in Figure 15. They attributed this difference to a change in the failure mode associated with the size effect.

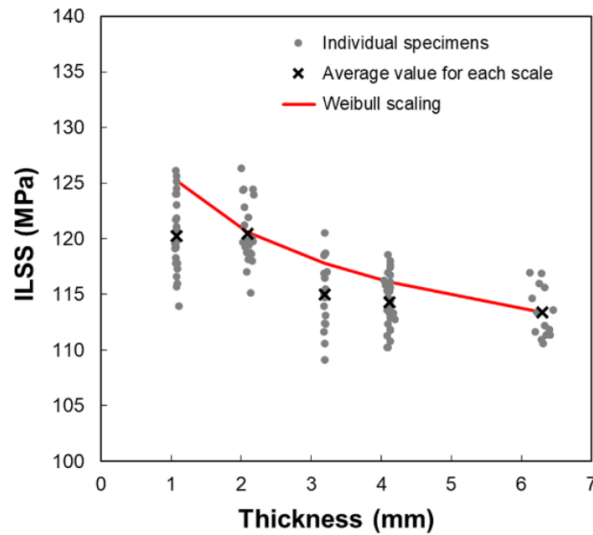


Figure 15. Influence of the size effect on the ILSS obtained by SBS tests according to ASTM D2344 for an IM7/8552 composite [68]

4.1.3. Out-of-plane strengths of bonded composites

The previous tests are developed specifically for composite materials, even if so. However, specific tests have been developed when bonded interfaces are involved to characterise the mechanical strengths σ^u and τ^u of bonded joints under each loading mode (*i.e.*, mode I, mode II).

The flatwise tensile test consists on applying tensile load on a square specimen as outlined in the standard ASTM D7291 [69] to induce failure in the out-of-plane normal direction at the bonded interface. It is also applicable for ILTS of composites.

The Single Lap Shear (SLS) requires to use a single-lap specimen with an overlap area, as described in the standard ASTM D5868 [70]. The ASTM D3165 [71] propose a

similar shear test using a notched specimen. This test consists on applying tensile load until failure to identify shear strength at crack onset. This mechanical property can be also identified by the Scarf specimen, but no standard is currently available. However, it also exists the TAST test [72], whose standard is preconised for non-rigid adhesives, but can be adapted for rigid ones or even for composites [73].

Another test that allows the characterisation of crack onset strengths for both delamination and debonding is the Arcan test [74]. Even if it does not exist a recognised standard for this test, it is widely used to characterise the interface under biaxial loading conditions. Its setup is represented in Figure 16. This test utilises a specific device that enables the sample to be installed at different loading angles, allowing it to be subjected to various loadings (tensile/shear and compression/shear). Thus, this test enables the characterisation of interlaminar strengths under mixed-mode loading.

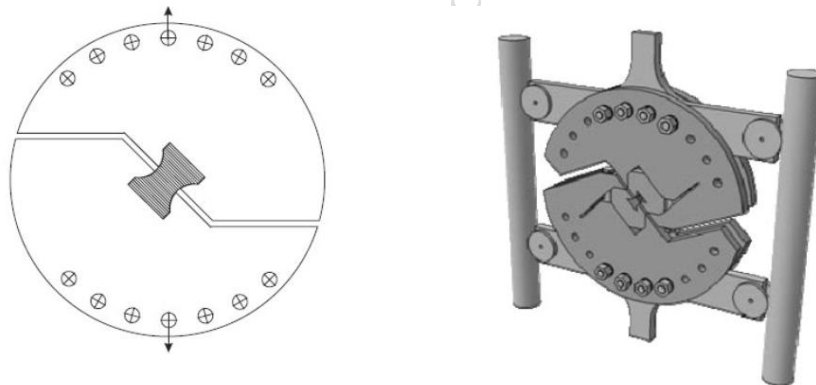


Figure 16. Representation of the Arcan test on a tensile (at left) and compression (at right) configuration for the identification of the interface strength [74].

4.2. Crack propagation characterisation

4.2.1. Energy release rate in mode I

The interlaminar fracture toughness in mode I (G_{IC}) can be determined using the standardised Double Cantilever Beam (DCB) test [75]. The test consists in applying tensile loading (opening mode) to the two arms of a specimen pre-cracked along its mid-

plane, as shown in Figure 17. The specimen dimensions and the pre-crack length, as well as the experimental protocol are defined according to the standard ASTM D5528 [75] and the standard ISO 15024:2001 [76] for unidirectional laminates with 0° plies. In this configuration, the interface is subjected to pure opening mode and the stacking sequence is supposed to not influence the interfacial response.

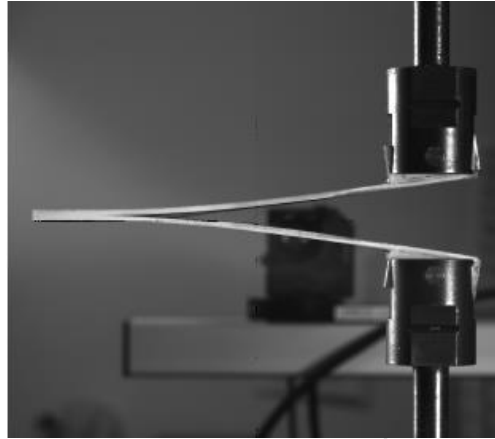


Figure 17. Application of the standard ASTM D5528 for the DCB test for laminated composites [44],[75].

On the one hand, the test enables the determination of the interface toughness when first signs of crack propagation occur, referred to as G_{IC} . This is calculated from the macroscopic force-displacement response using the Compliance Calibration Method (CCM) [75]. This method involves fitting a parameter n based on visually observed delamination onset values and all subsequent propagation data. The standard provides an expression for the calculation of G_{IC} as well as the relationship between compliance evolution and crack length as follows:

$$G_{IC} = \frac{nP_{max}\delta}{2Ba_0} \quad (1)$$

$$\text{Log}(C(a)) = A + n \text{Log}(a) \quad (2)$$

P_{max} denotes the maximum force measured during the DCB test, δ represents the corresponding opening displacement, B is the specimen width and a_0 is the initial crack length (pre-crack between the tip and the load application point, as shown in Figure 17). $C(a)$ represents the compliance of the specimen as function of the crack length evolution during crack propagation.

On the other hand, the test facilitates the definition of the resistance curve, or R-curve, which depicts the evolution of the apparent toughness as a function of crack length. Crack propagation remains stable during this test due to the specimen geometry and the loading mode. However, in the case of delamination, the crack propagation is influenced by the crack front. In mode I, fibre bridging occurs as the arms open under tensile loading. These fibre bridges result in R-curve effects. Consequently, an increase in the apparent toughness value with the progression of the crack length [44],[77] is observed. This phenomenon poses a challenge for the determination of G_{IC} as R-curve effects should not be included in the estimation. Only the initial test values should be used to characterise the delamination toughness of composites [44].

Mohsin et al. [78] investigated mode I toughness for carbon fibre reinforced thermoplastic composites. Figure 18 presents the R-curve effects, which are more pronounced in the T700/PPS system compared to the T700/PA6.6 system. They observed not only fibre bridging but also fibre/matrix debonding, matrix strain and void formation. These mechanisms contribute to plastic deformation and variations in the measured toughness.

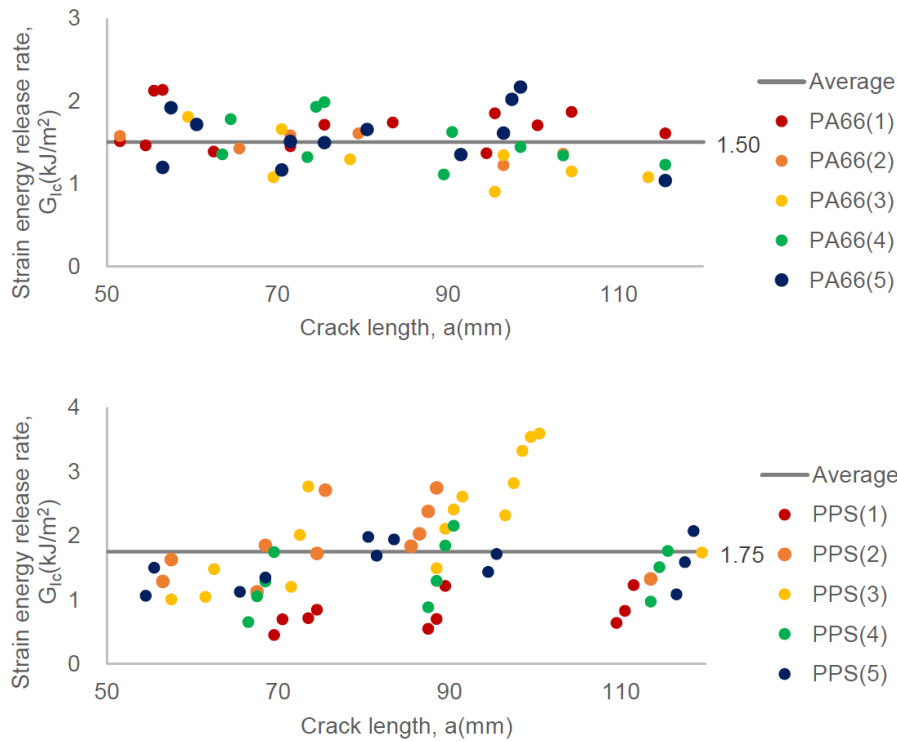


Figure 18. Application of the standard ASTM D5528 to identify the delamination R-Curve from the DCB test for the T700/PA6.6 system (top) and the T700/PPS system [78].

4.2.2. Energy release rate in mode II

R-curve effects are also commonly observed in mode II, but their characterisation is even more complex than in mode I due to the shear solicitation mode and the challenges associated with monitoring crack propagation. The mode II interlaminar fracture toughness G_{IIc} can be determined using various standardised tests. These tests involve the propagation of an pre-existing crack between two composites arms or adherents along the interface under shear loading. One of the most widely test is the End-Notched Flexure (ENF) test. This test is described in the standard ASTM D7905 [79] for the determination of the mode II fracture toughness of unidirectional fibre reinforced polymer matrix composites. Figure 19 presents the setup of an ENF test.



Figure 19. Application of the standard ASTM D7905 for the ENF test for laminated composites [44],[79].

The test consists in applying a three-point bending load to a pre-cracked specimen to induce shear stress to propagate the crack in mode II along the interface of the specimen within the plane defined by the pre-crack [3],[79]. The main dimensions of the specimen, as well as the expressions for calculating G_{IIC} and the evolution of compliance are specified in the standard ASTM D7905:

$$G_{IIC} = \frac{3mP_{max}^2a_0^2}{2B} \quad (3)$$

$$C(a) = A + ma^3 \quad (4)$$

P_{max} denotes the maximum force measured during the ENF test, a_0 represents the initial crack length (the pre-crack extending from the crack tip to the position of the lower support roller, as shown in Figure 19). B is the width of the specimen and m is the first derivative of the compliance $C(a)$ with respect to the cube of the crack length. At least two Compliance Calibration (CC) tests must be performed prior to conducting the crack propagation test up to failure (*i.e.*, delamination or debonding) [79].

The ENF test can also be performed in a 4-point bending configuration (4ENF), which involves two support points and two loading points. The calculation of mode II toughness for this test is based on the analysis of the compliance curve, analogously to the 3ENF test. However, the 4ENF test is not standardised due to the uncertainties associated with frictional effects and contact interaction arising from the use of 4 rigid rollers [80]-[83]. The set-up is presented in Figure 20.

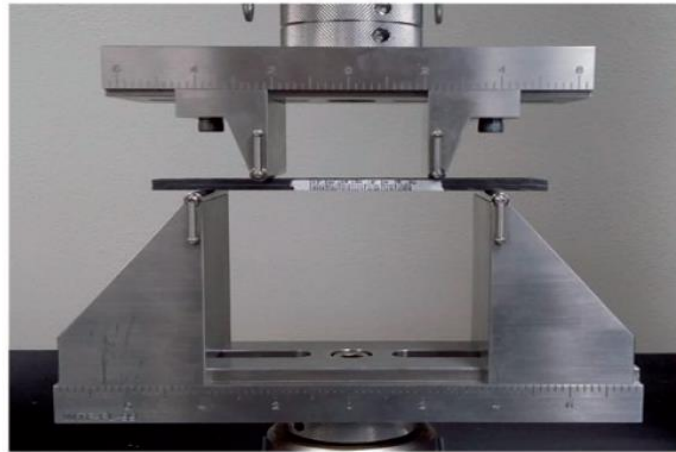


Figure 20. Application of the 4ENF test for laminated composites [84].

The Calibrated End-Loaded Split (C-ELS) test is described in the standard ISO 15114:2014 [85]. It also allows to characterise the interlaminar fracture toughness in mode II. This test involves applying cantilever bending to a pre-cracked specimen, as shown in Figure 21 [86]. The longer flexural arm used in this configuration provides more stable conditions for crack propagation [87].

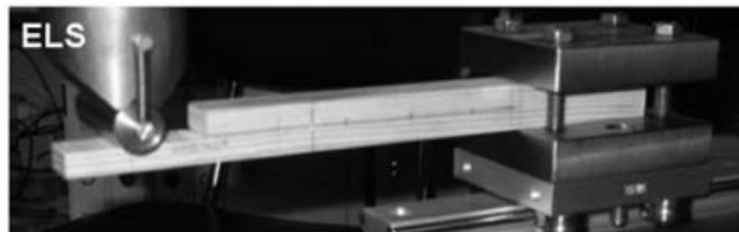


Figure 21. Application of the C-ELS test for laminated composites [86].

A calibrated crack length is determined from the compliance curve, avoiding the need for experimental measurement of the crack growth progression. This is particularly advantageous since optical monitoring of crack propagation in mode II is more complex than in mode I due to the nature of the stress mode (shear in mode II and opening in mode I).

Barcikowski et al. [88] evaluated the influence of different matrices on the mode II toughness of epoxy-glass laminates using the ENF test. They highlighted the impact of various modifying agents on the measured toughness, attributed to changes in the specimen compliance. Campilho et al. [89] performed ENF tests on carbon fibre and epoxy resin laminates to determine mode II toughness, as shown in Figure 22. They employed the Compliance-Based Beam Method (CBBM) to mitigate errors associated with the crack growth measurement, which can significantly affect the calculation of the energy release rate at failure.

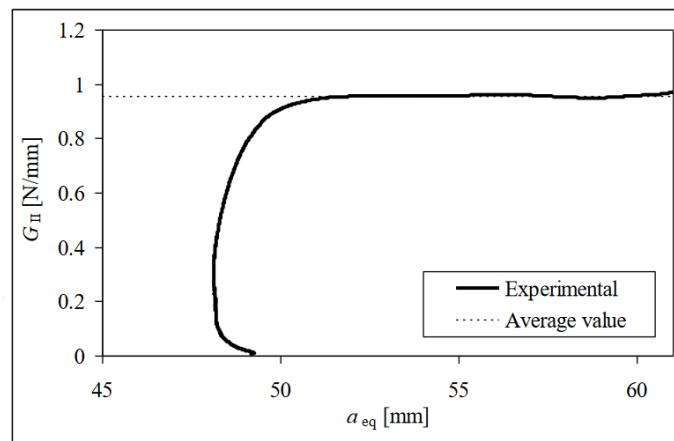


Figure 22. Application of the standard ASTM D7905 to identify the delamination resistance curve (R-Curve) using the ENF test for CFRP composite [89].

4.2.3. Energy release rate in mixed mode I/II

Other tests are designed to characterise toughness under mixed mode, distinguishing between mode I and mode II contributions. The most commonly used test

is the Mixed Mode Bending (MMB) test. The experimental protocol and the corresponding analysis are outlined in the standard ASTM D6671/D6671M [90]. This test consists in applying a combination of tensile and bending loads resulting in both opening (mode I) and a sliding (mode II) at the interface. The degree of mode mixity can be controlled *a priori*. The setup of the MMB test is presented in Figure 23 [44].



Figure 23. Application of the standard ASTM D6671 using the MMB test for laminated composites [44],[90].

The beam theory can be employed to estimate the G_{IC} and G_{IIC} from the macroscopic force-displacement response, while neglecting the shear contribution:

$$G_{IC} = \frac{3P_{max}^2 a_0^2}{4b^2 t^3 E_l L^2} (3c - L)^2 \quad (5)$$

$$G_{IIC} = \frac{9P_{max}^2 a_0^2}{16b^2 t^3 E_l L^2} (c + L)^2 \quad (6)$$

$$\frac{G_{IC}}{G_{IIC}} = \frac{4}{3} \left(\frac{3c - L}{c + L} \right)^2 \quad c > L/3 \quad (7)$$

L is the specimen half-length and c is the distance from the load application point to the mobile contact point with the specimen, which allows to vary the G_{IC}/G_{IIC} ratio [44].

Another tests to study the mixed mode I/II of interfaces are the Single Leg Bending (SLB) [91]. test and the Crack Lap Shear test [92], even if there is not recognised available standards. The first one consists in applying a bending load to an axisymmetric sample. The load application point is positioned to induce a fixed degree of mixed-mode ratio of $\psi = \tan^{-1}\sqrt{G_{II}/G_I} = 41^\circ$. The Figure 24 shows the configuration of the SLB test [91].

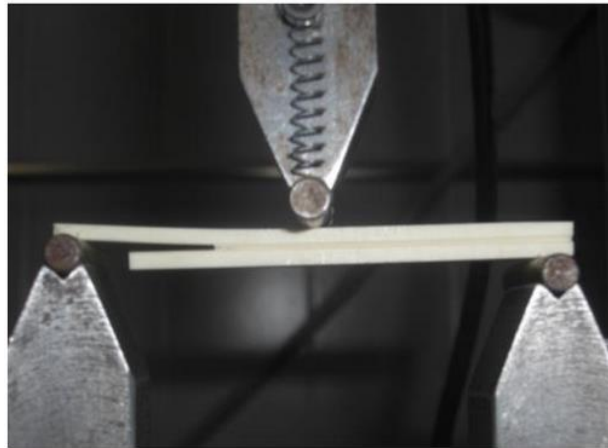


Figure 24. Application of the SLB test for bonded composites [91].

The Crack Lap Shear (CLS) test is conducted on an asymmetric pre-cracked sample mounted on a non-fixed support and subjected to tensile loading to induce a fixed degree of mixed-mode ratio of $\psi = \tan^{-1}\sqrt{G_{II}/G_I} = 49^\circ$. The Figure 25 shows the configuration of the CLS test [92].

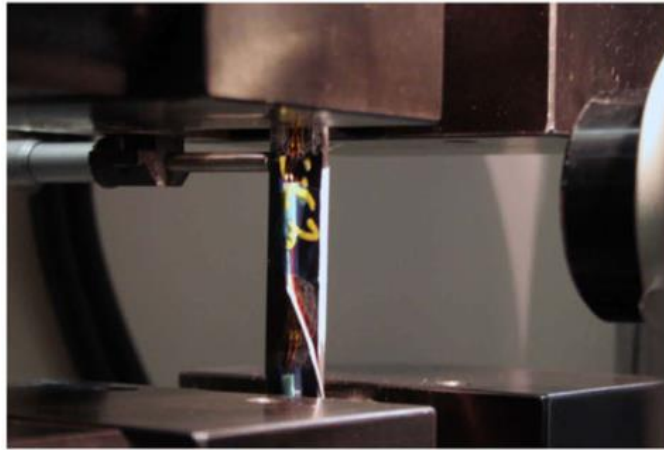


Figure 25. Application of the CLS test for bonded composites [92].

4.2.4. Energy release rate of bonded composites

For thin adhesively bonded composites, interface characterisation in mode I, mode II and mixed mode I/II can be performed using the analogous test previously presented. In this context, the tests do not characterise an interply interface, but rather a finite adhesive interface, whose mechanical properties allow to describe the debonding mechanisms leading to failure. Sanchez et al. [77] compared the mode I and mode II toughness of different adhesive layers using DCB and ENF tests on bonded composite specimens, as shown in Figure 26. They noted that the adhesive behaviour enhances the fracture toughness of the original composite material.

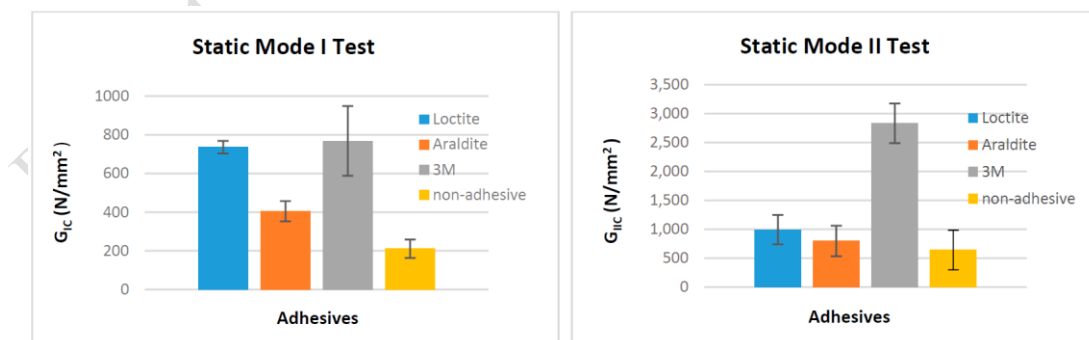


Figure 26. Comparison of the interlaminar fracture toughness in mode I (left) and mode II (right) from DCB and ENF tests respectively and for different adhesives [77].

Hereby, Table 1 present a synthesis of standard tests previously presented which are commonly used for both delamination and debonding characterisation:

Test	Target property	Standard formula	Reference
Delamination onset			
CBS	ILTS [MPa]	$ILTS \approx \frac{3 CBS}{2h \sqrt{r_i r_o}}$	ASTM D6415 [58]
Iosipescu / V-notched beam	ILSS [MPa]	-	ASTM D5379 [63]
SBS	ILSS [MPa]	$ILTS \approx SBS = \frac{3 F_{max}}{4 L h}$	ASTM D2344 [65]
DNS*	ILSS [MPa]	$ILTS \approx \frac{P_{max}}{B h}$	ASTM D3846 Erreur ! Source du renvoi introuvable.
Debonding onset			
Flatwise tensile**	σ^u [MPa]	$\sigma^u = \frac{P_{max}}{B h}$	ASTM D7291 [69]

SLS**	τ^u [MPa]	$\tau^u = \frac{P_{max}}{B L}$	ASTM D5868 [70] / ASTM D3165 [71]
TAST**	τ^u [MPa]	$\tau^u = \frac{P_{max}}{B L}$	ASTM D3983-98 [72] / ASTM D5656-10 [73]

Crack propagation

DCB	G_{IC} [J/m ²]	$G_{IC} = \frac{n P_{max} \delta}{2 B a_0}$	ASTM D5528 [75] / ISO 15024:2023 [76]
ENF	G_{IIC} [J/m ²]	$G_{IIC} = \frac{3 m P_{max}^2 a_0^2}{2 B}$	ASTM D7905 [79]
C-ELS	G_{IIC} [J/m ²]	$G_{IIC} = \frac{3 P_{max} \delta}{2 B a_0}$	ISO 15114:2014 [85]
MMB	G_{IC} / G_{IIC} [J/m ²]	$G_{IC} = \frac{3 P_{max}^2 a_0^2}{4 B^2 h^3 E_1 L^2} (3c - L)^2$ $G_{IIC} = \frac{9 P_{max}^2 a_0^2}{16 B^2 h^3 E_1 L^2} (c + L)^2$	ASTM D6671/D6671M [90]

Table 1. Standard test for crack onset and crack propagation to characterise delamination and debonding of composites and bonded composites. *In its variation IDNS, **Also adapted for composites.

5. Characterisation tests of delamination and debonding in temperature

To investigate the effects of temperature on delamination and debonding without the complexities detailed in Section 3.3 associated with the exposition of composites to fire or a more or less controlled flame, mechanical tests can be adapted to analyse the influence of temperature on the initiation and the propagation of both delamination and debonding.

5.1. Crack onset characterisation in temperature

Few studies on the characterisation of crack onset at high temperature focused on adapting experimental tests typically conducted at room temperature as presented in Section 4.1, to elevated temperature conditions. These adaptations are often performed by conducting tests within climate chambers [3].

5.1.1. Out-of-plane tensile strength

Sethi et al. [93] investigated the variation of interlaminar shear strength (ILSS) as a function of temperature and loading rate using the SBS test on short carbon fibre and epoxy resin specimens. They observed a decrease in ILSS with increasing temperature, as shown in Figure 27 (left). Additionally, for temperatures below 25°C, an increase in ILSS was reported for applied displacement rates (\dot{u}) below 200 mm/min, while a decrease was noted for higher rates. However, for temperatures above 25°C, an increase in ILSS was observed with higher loading rates as shown in Figure 30 (right).

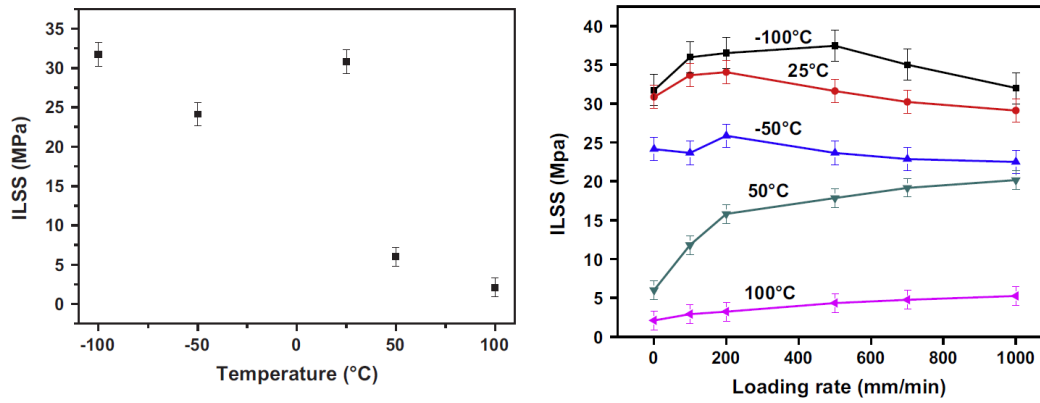


Figure 27. Evolution of the ILSS as a function of temperature (left) and for different temperatures and load rates (right) [93].

Argüelles et al. [94] analysed the influence of temperature on the delamination onset in two carbon/epoxy composites subjected to mode I fatigue loading. They examined the fracture surfaces of a brittle resin (3501-6) and a tough resin (8552) at room temperature, 50°C and 90°C. By analysing the fracture surfaces, as shown in Figure 28 (room temperature) and Figure 29 (50°C), they observed that delamination onset occurs at the fibre-matrix interface. This indicates that the dominant failure mode is governed by the mechanical properties of the matrix. For the brittle matrix, an increase in temperature enhances matrix strain, thereby improving the final toughness. In contrast, for the tough matrix, the temperature does not significantly affect the final toughness and instead reduces the matrix strain, resulting in poorer material performance.

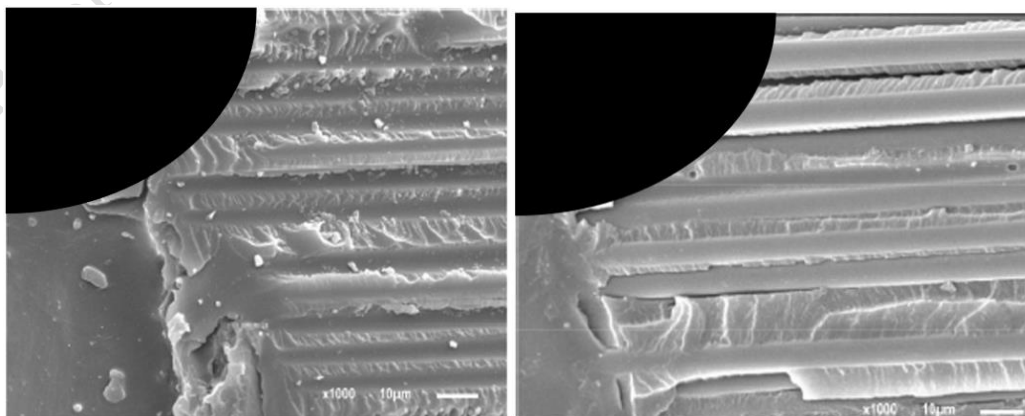


Figure 28. Fracture surface at 20°C of the AS4/3501-6 (left) and the AS4/8552 (right) (1000x) [94].

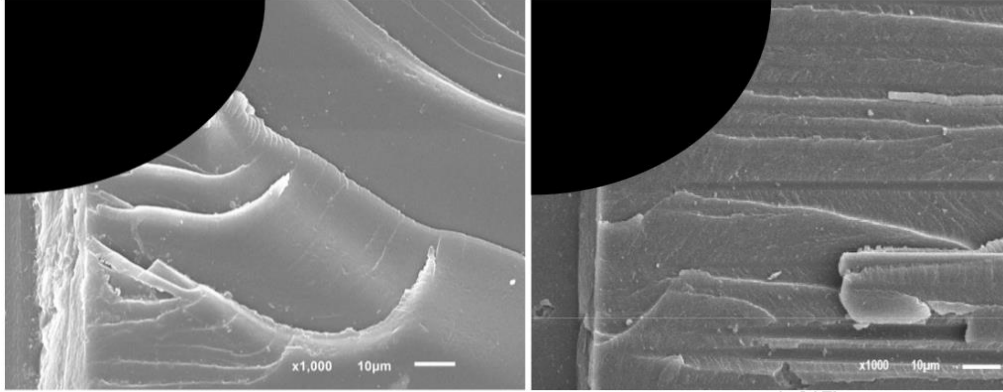


Figure 29. Fracture surface at 50°C of the AS4/3501-6 (left) and the AS4/8552 (right) (1000x) [94].

5.1.2. Out-of-plane shear strength

Sengodan et al. [95] compared experimental tests with numerical simulations for both mode I and mode II delamination of a carbon/epoxy composite material (IM7/8552). CBS test and SBS test were used to assess mode I and mode II respectively. Figure 30 presents the experimental and numerical comparison of the influence of temperature on the ILTS and the ILSS under different conditions: room temperature in dry air (RTD), elevated temperature in dry air (ETD) and elevated temperature in wet air (ETW). They observed a reduction in both ILTS and ILSS with temperature.

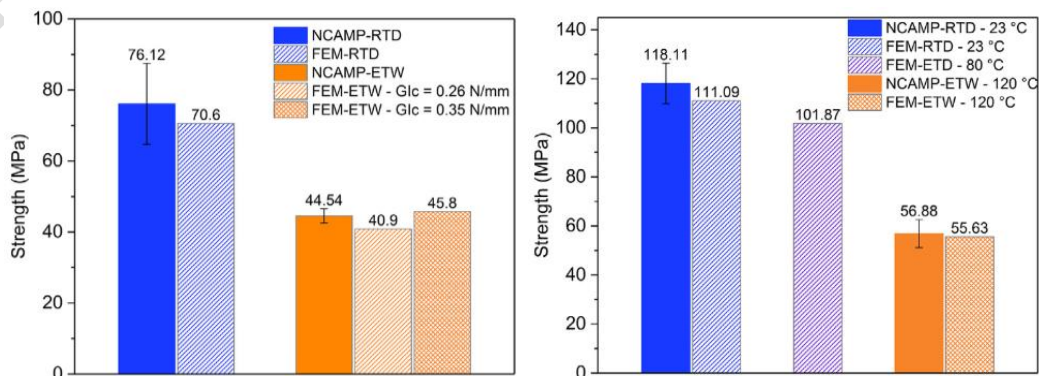


Figure 30. Comparison of experimental to predicted ILTS (left) and ILSS (right) of of IM7/8552 UD laminates at different hygro-thermally conditions: RTD, ETD and ETW [95].

5.1.3. Out-of-plane strengths of bonded composites

Regarding the study of crack onset in bonded composites, similar tests can be conducted to characterise the bonded interface strength depending on the loading mode. Fernandes et al. [96] investigated the onset strength for both mode I and mode II using DCB and ENF tests respectively. They estimated cohesive parameters to simulate long exposure time of bonded composites at temperatures ranging from 0°C and 50°C. The identified parameters are represented in the average cohesive laws presented in Figure 31 for the DCB and the ENF tests. They observed a significant reduction in G_{IC} and the normal interface strength, while not substantial differences in G_{IIC} and the shear interface strength with temperature.

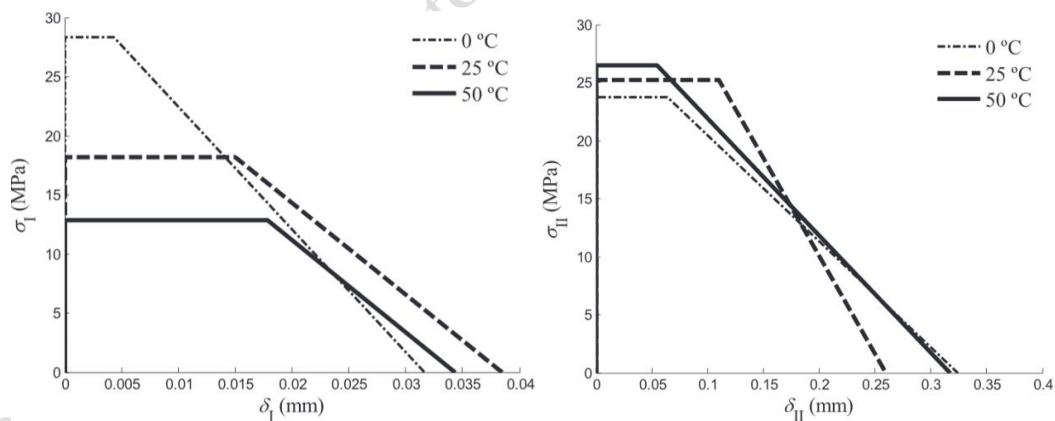


Figure 31. Experimental and numerical average cohesive laws for the DCB (left) and the ENF (right) tests [96].

5.2. Crack propagation characterisation in temperature

5.2.1. Energy release rate of composites

Concerning the characterisation of mode I, some authors have sought to evaluate the influence of temperature on interfacial properties at failure in this mode by employing classic characterisation methods for room temperature conditions. Most studies focus on the DCB test [42],[97]-[102] and report that the apparent macroscopic force-displacement response remains linear up to the onset of delamination. This apparent linear macroscopic response is observed even at elevated temperatures, despite the potential for nonlinear behaviour arising from viscoelastic strains within the matrix, which may alter the temperature response. Uematsu et al. [97] investigated the influence of temperature on mode I fracture toughness for carbon fibre and PEEK thermoplastic matrix composites using DCB tests. The variation in toughness as a function of temperature is shown in Figure 32. The authors observed that the apparent toughness increases in a first step with temperature but decreases in a second step at higher temperatures, a phenomenon attributed to creep effects.

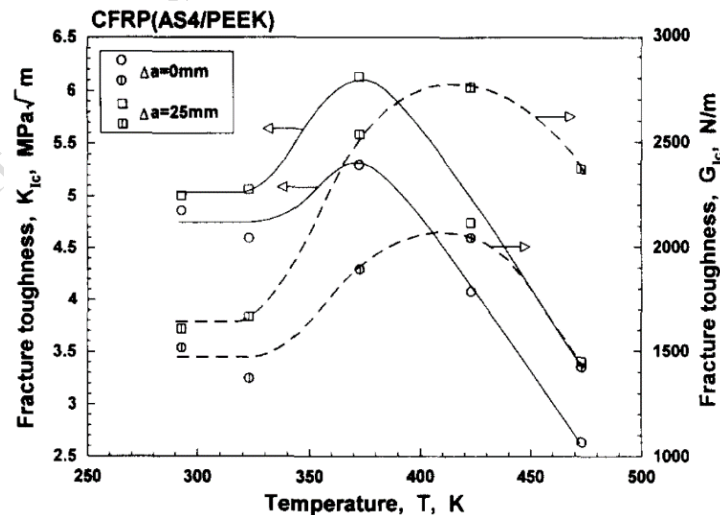


Figure 32. Influence of temperature on mode I fracture toughness using DCB tests [97].

Reeder et al. [98] demonstrated a dependence of mode I toughness on temperature and stress rate for graphite fibre and thermoplastic resin composites (IM7/K3B) also using DCB tests. They concluded that the mode I toughness is a complex function of both stress rate and temperature due to the nonlinear behaviour of the matrix.

However, they noted that it was not possible to derive a master curve to characterise the combined influence of temperature and stress rate on fracture toughness, as can be done for the transverse behaviour of the matrix. This limitation was attributed to the temperature dependencies shown in Figure 33. The authors hypothesised that the observed differences arise from three combined effects of increasing temperature: a reduction in modulus, an increase in strain at failure and a decrease in the material strength.

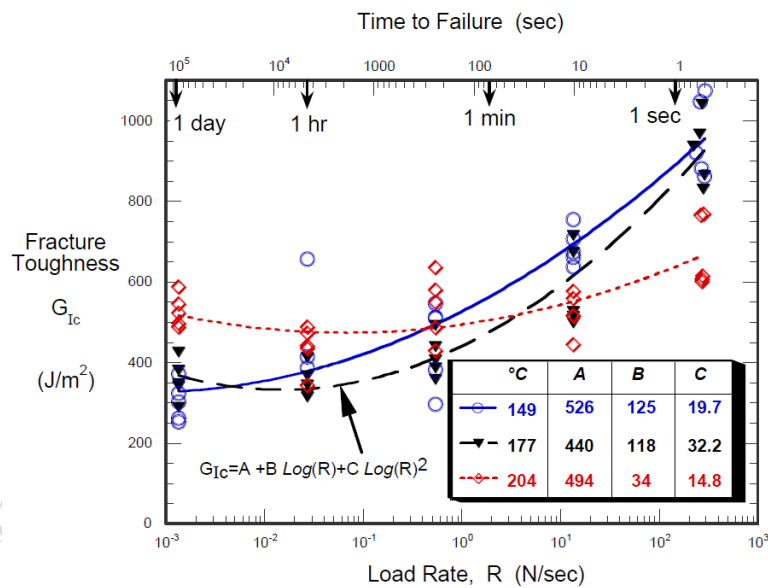


Figure 33. Dependence of the mode I fracture toughness on the temperature and the stress rate using DCB tests [98].

Oliver and Johnson [99] investigated the effect of temperature on mode I toughness using DCB tests for two types of carbon fibre and epoxy resin composites (IM7/PETI-5 and IM7/977-2) over a temperature range from -196°C to 160°C. Their

results indicated a minor variation in in toughness for temperatures below 25°C, followed by a significant increase at temperatures above 25°C, as shown in Figure 34.

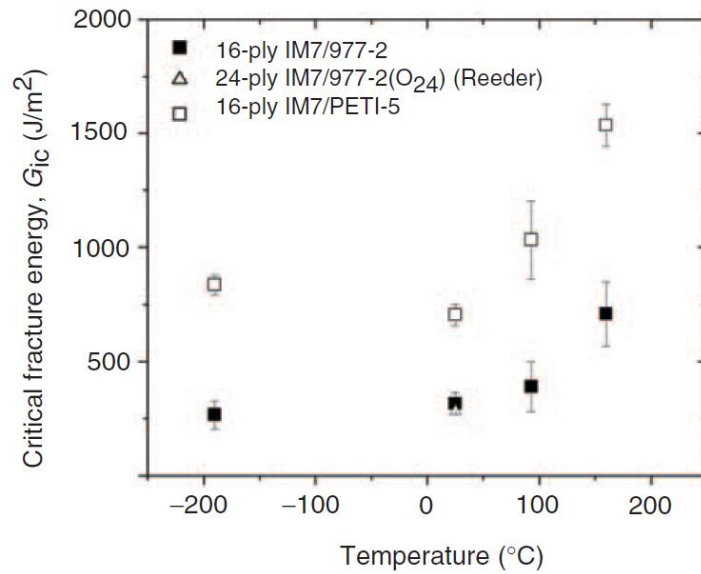


Figure 34. Mode I toughness at break for different temperatures between -196°C and 160°C using DCB tests [99].

Cao et al. [100] examined the temperature dependence of fibre bridging in mode I delamination of unidirectional laminates by DCB tests at temperatures between 20°C and 110°C. Their results showed that higher temperatures led to denser fibre bridging, higher values of G_{IC} , and R-curve effects leading to lower crack propagation rates, as shown in Figure 35. They attributed that to enhanced matrix ductility and fibre bridging densification in temperature.

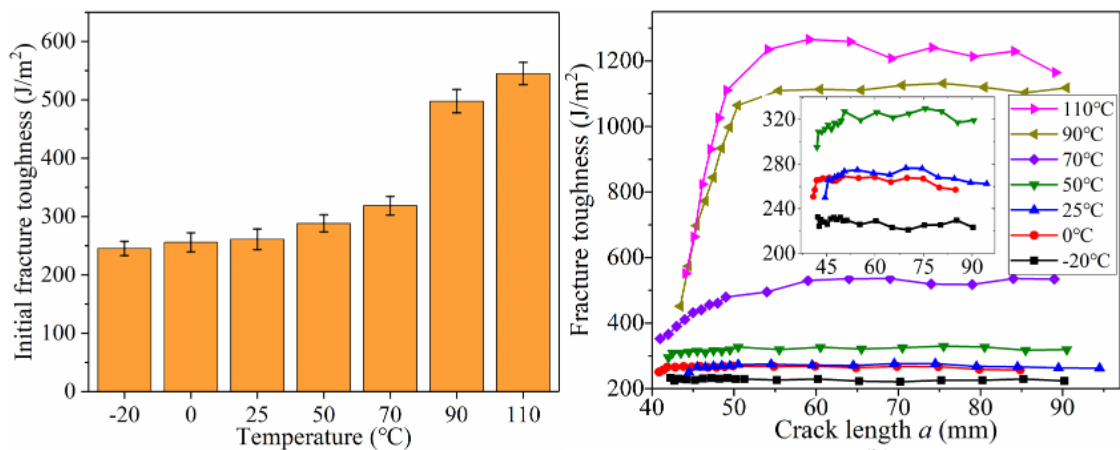


Figure 35. Initial fracture toughness G_{IC} (left) and measured R-curves effects (right) at different temperatures using DCB tests [100].

Other researchers have investigated the effects of different loading conditions (quasi-static, fatigue and dynamic) across varying temperatures and mode mixities (mode I or mixed mode I/II) [94], [101]-[106]. For instance, during fatigue loading, authors [101] reported that temperature increases apparent toughness as the crack length grows as shown in Figure 36. This phenomenon appears to promote more stable crack propagation due to the enhanced ductility of the matrix.

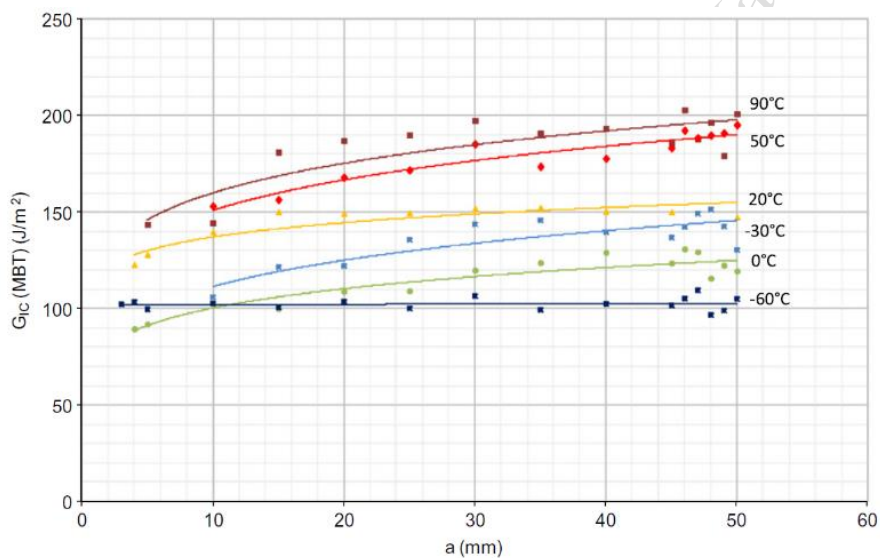


Figure 36. Evolution of the mode I toughness in temperature as function of the advancement of the crack length [101].

In the case of dynamic loadings, authors [101] observed an increase of mode I toughness with high loading rates, which is even more exacerbated in temperature, as shown in Figure 37. They deduced that the effect of temperature and loading rate can be attributed to the nature of matrix and the bonding between fibre and matrix, leading to multiple failure modes at high temperatures.

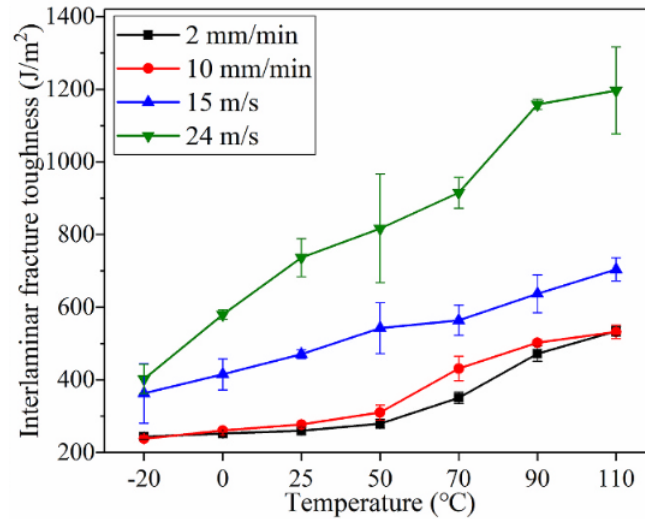


Figure 37. Evolution of the mode I toughness in temperature as function of the loading rate [101].

As discussed in the studies presented on the propagation of delamination in mode I, temperature has been shown to increase fracture toughness. However, there is not consensus among authors regarding the underlying mechanisms for this effect. Some authors attribute it to the more ductile behaviour of the matrix [99]. Others authors observed a non-negligible densification of bridging [100] or even loading rate effects [101], while others suggest a competition between the effects of increasing temperature, such as a decrease in modulus and material strength, coupled with an increase in strain at rupture [98], or even the influence of creep behaviour [97].

Few studies on mode II delamination of composites under temperature conditions have attempted to replicate standard tests as typically conducted at room temperature. Sales et al. [103] interlaminar toughness at interface failure as a function of temperature using 4-point bending tests (4ENF) on carbon fibre and epoxy resin composite materials. Their findings indicate that toughness decreases with increasing temperature. The evolution of the crack during delamination propagation is therefore influenced by temperature, as shown in Figure 38.

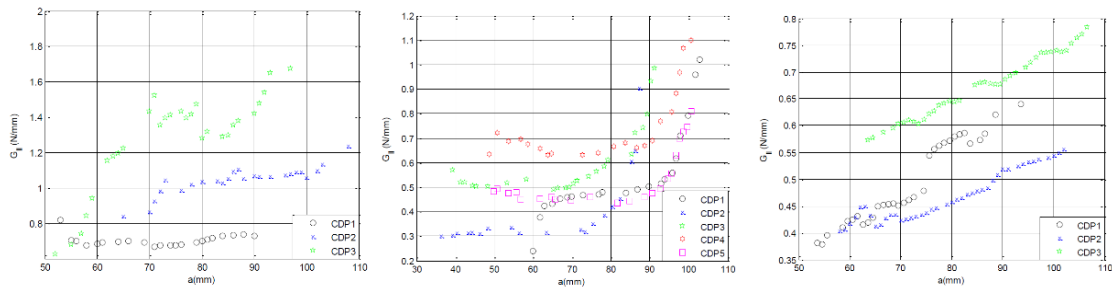


Figure 38. Evolution of the mode II toughness as a function of the crack length growth at ambient temperature (left), 80°C (middle) and -54°C (right) using 4ENF tests [103].

Asp [107] investigated the 3ENF and MMB tests for carbon fibre and epoxy matrix composites, reporting an increase in mode II toughness with temperature under mixed-mode MMB tests, but a significant decrease under pure mode II (3ENF) tests, as shown in Figure 39.

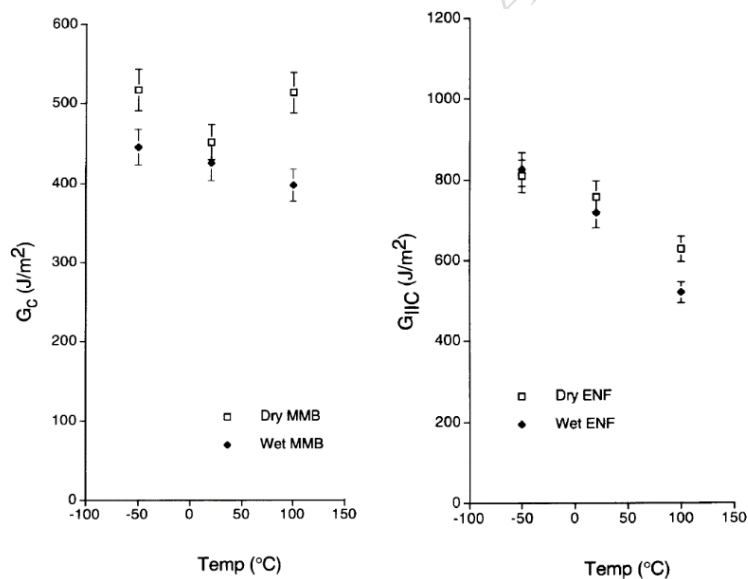


Figure 39. Influence of temperature on the mode II fracture toughness for dry and wet carbon/epoxy composites in mixed mode I/II (left) and in pure mode II (right) [107].

Chow et al. [108] characterised the interface mechanical properties of a glass fibre and aluminium composite for both mode I and mode II. Through experimental testing and verification of mechanical parameters via numerical simulations using cohesive zone

models, they observed a reduction in both the modulus and the toughness with increasing temperature, as presented in Figures 40 and 41.

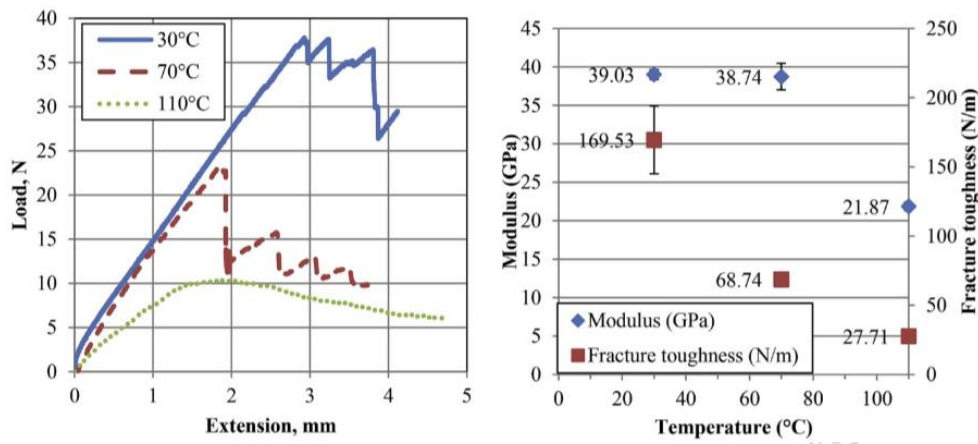


Figure 40. Representative load-displacement curve (left) and mode I modulus and fracture toughness as function of temperature using DCB tests [108].

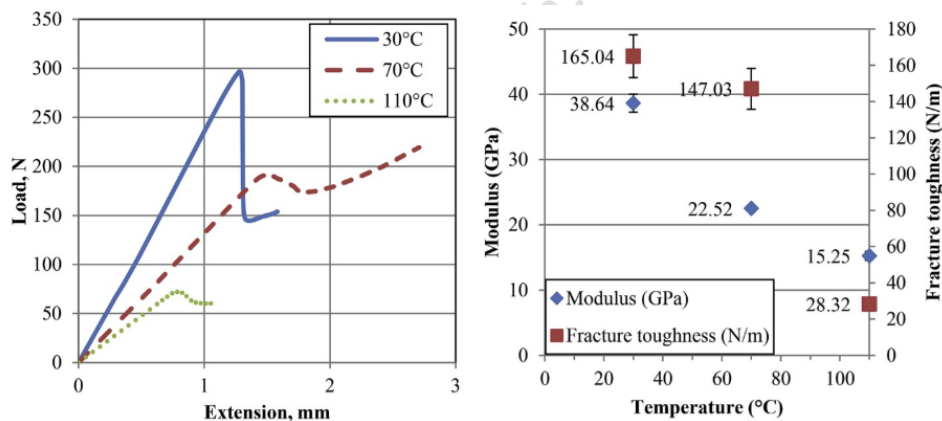


Figure 41. Representative load-displacement curve (left) and mode II modulus and fracture toughness as function of temperature using ENF tests [108].

Gong et al. [109] used ENF test for evaluate the effect of temperature on mode II toughness for CFRP T800/epoxy at four different temperatures: -50°C, 23°C, 80°C and 130°C. They compared G_{IIc} for non pre-cracked and pre-cracked configurations. They observed a negligible effect of temperature for the pre-cracked configuration, while a

decrease of G_{IIc} was highlighted for the non pre-cracked samples only from 80°C to 130°C.

Davidson et al. [110] examined the influence of temperature on toughness in mode I, mode II and mixed mode I/II for a thermoplastic particulate interlayered carbon/epoxy composite. Experimental results obtained from DCB, ENF and SLB tests for each mode are presented in Figures 42, 43 and 44 respectively. It is observed that G_{IC} increases with temperature, while G_{IIc} and mixed-mode toughness decrease with temperature.

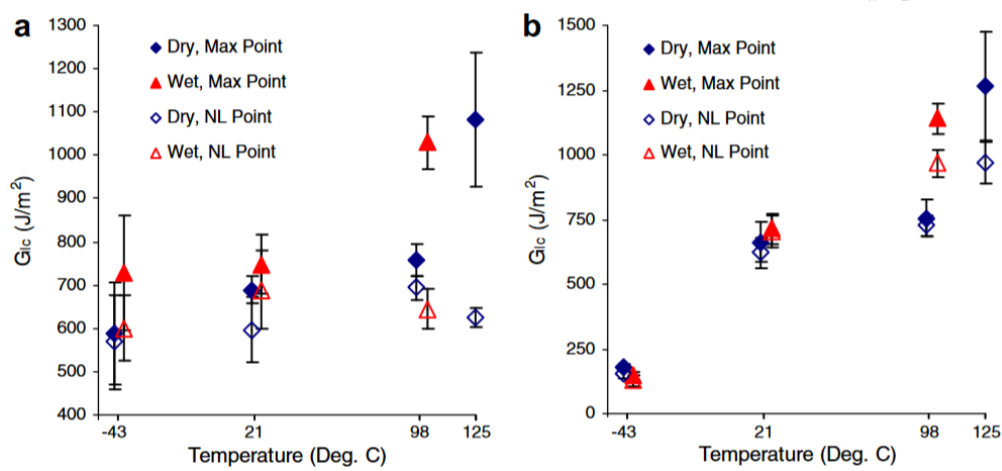


Figure 42. Evolution of G_{IC} with temperature from a teflon insert (left) and from a pre-crack (right) using the DCB test [110].

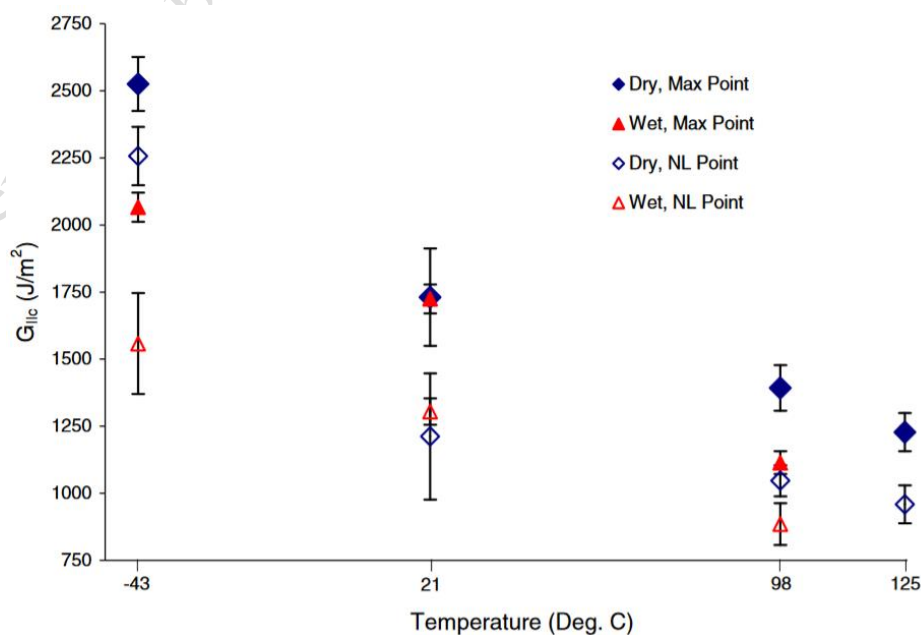


Figure 43. Evolution of G_{IIC} with temperature using the ENF test [110].

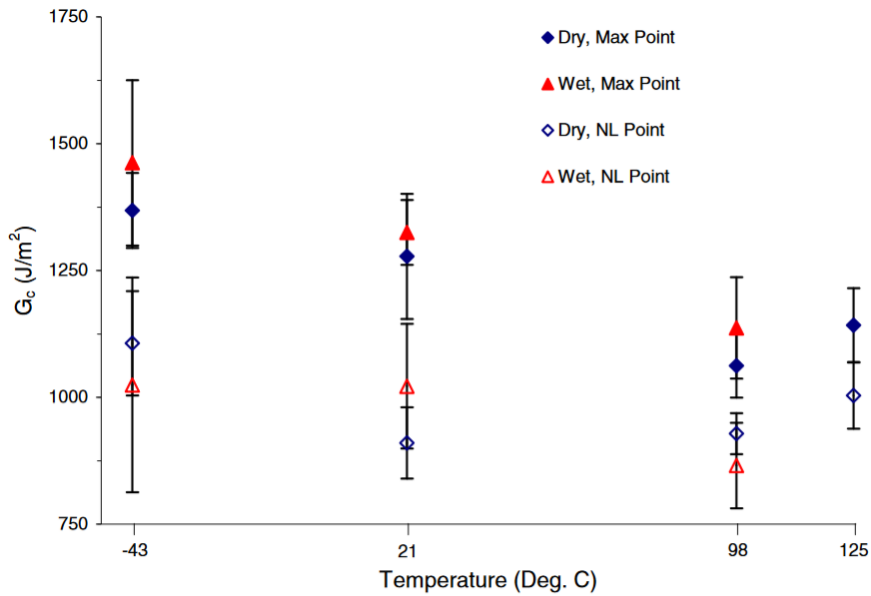


Figure 44. Evolution of mixed-mode G_C with temperature using the SLB test [110].

The Table 2 presents a summary of keys aspects found by previous studies concerning the temperature effect on fracture properties.

Test	Target property	Test conditions	Temperature effect	Reference
SBS	ILSS [MPa]	[-100°C, 100°C]	↑ T → ILSS ↓ and ↓ (dependent to \dot{u})	[93]
SBS	ILSS [MPa]	[25°C, 90°C]	↑ T → ILSS ↓ (brittle epoxy) ↑ T → ILSS ~ (tough epoxy)	[94]

CBS / SBS	ILTS / ILSS	[25°C, 120°C]	$\uparrow T \rightarrow \text{ILTS / ILSS} \downarrow$	[95]
DCB / ENF	$\sigma_c, G_{IC} /$ τ_c, G_{IIC}	[0°C, 50°C]	$\uparrow T \rightarrow \sigma_c, G_{IC} \downarrow / \tau_c, G_{IIC} \sim$	[96]
DCB	G_{IC}	[149°C, 204°C]	$\uparrow T \rightarrow G_{IC} \uparrow$ when load rate $< \sim 1$ N/s $\uparrow T \rightarrow G_{IC} \downarrow$ when load rate $> \sim 10$ N/s	[97]
DCB	G_{IC}	[-196°C, 160°C]	$\uparrow T \rightarrow G_{IC} \uparrow$ when $T > 25^\circ\text{C}$ $G_{IC} \sim$ when $T < 25^\circ\text{C}$	[99]
DCB	G_{IC}	[-20°C, 110°C]	$\uparrow T \rightarrow G_{IC} \uparrow$ $\uparrow \dot{u} \rightarrow G_{IC} \uparrow$	[100],[101]
DCB	G_{IC}	[-60°C, 90°C]	$\uparrow T \rightarrow G_{IC} \uparrow$ (exception in $T = 0^\circ\text{C}$)	Erreur ! Source du renvoi introuvable.
4ENF	G_{IIC}	[-54°C, 80°C]	$\uparrow T \rightarrow G_{IIC} \uparrow$	[103]

ENF / MMB	G_{IIC}	[-50°C, 100°C]	$\uparrow T \rightarrow G_{IIC} \uparrow$ for MMB $\uparrow T \rightarrow G_{IIC} \uparrow$ for ENF	[107]
DCB / ENF	G_{IC} / G_{IIC}	[30°C, 110°C]	$\uparrow T \rightarrow G_{IC} \downarrow / G_{IIC} \downarrow$	[108]
ENF	G_{IIC}	[-50°C, 130°C]	$\uparrow T \rightarrow G_{IIC} \uparrow$ (pré-cracked) $\uparrow T \rightarrow G_{IIC} \sim$ (non pré-cracked)	[112]
DCB / ENF / SLB	G_{IC} / G_{IIC} / G_{IC}/G_{IIC}	[-43°C, 125°C]	$\uparrow T \rightarrow G_{IC} \uparrow / G_{IIC} \downarrow / G_{IC}/G_{IIC} \downarrow$	[110]

Table 2. Standard test adapted in temperature for crack onset and crack propagation to characterise delamination and debonding of composites and bonded composites.

5.2.1. Energy release rate of bonded composites

Similar tests have also been employed to characterise the fracture toughness of bonded interfaces. Other authors [111],[112] have proposed experimental methods to identify the mechanical properties of epoxy adhesives at various temperatures in order to formulate a cohesive constitutive law for describe the bonding and debonding of the adhesive layer. However, the behaviour and the thickness of both the adhesive layer and the adherents are assumed to be known *a priori*, and the resulting model remains highly phenomenological. Such models are only applicable to the specific cases and conditions used for their identification. In the case of laminated composite interfaces, their thicknesses is typically on the order of one-tenth of a ply thickness. This makes it

challenging to accurately characterise the behaviour of such layer. This challenge is compounded by the difficulty of distinguishing the boundary between the interface and the adjacent plies. Moreover, for thin bonded composites, even if the adhesive behaviour has been previously identified, its fracture behaviour within a composite assembly has been proved to differ significantly [113]-[116].

Therefore, the temperature-dependent tests presented here should help to enhance the understanding of fracture mechanisms in both composites and bonded composites. However, the existing studies on the characterisation of the mode I, mode II and mixed-mode delamination and debonding remain insufficient to establish a clear consensus on the effects of temperature on interface properties during crack onset and crack propagation [3],[44],**Erreur ! Source du renvoi introuvable.**,[48]. The complexity of these tests makes it difficult to isolate and quantify the contributions of various factors on the interface properties, mainly: the stiffness degradation, R-curve effects, the more ductile behaviour of the matrix in temperature and the coupling between viscous effects, plasticity, damage and fracture.

6. Discussion about characterising delamination and debonding of composites

6.1. Nonlinear effects in the characterisation of delamination and debonding

The studies presented in Section 4 highlight several challenges in characterising the mechanical response of interfaces through experimental testing: (i) How to ensure the accurate determination of the crack onset and the monitoring of crack growth? (ii) How to select the most appropriate model to correctly describe crack onset and crack propagation? (iii) How to account for each contribution of different dissipative mechanisms (*i.e.*, viscous effects, plasticity, damage) to the total energy release rate? Is it possible to quantify these dissipation processes separately?

Numerous studies have been conducted at room temperature to characterise the initiation and propagation of mode I and mode II delamination [56],[57],[59]-[62],[64],[66],[68],[74],[77],[78],[80]-[84],[86]-[89]. However, the characterisation of the fracture properties of the interface seems to be less precise in mode II. As discussed in Section 4, normal stresses at the crack tip are significant in mode I, leading to the opening of the crack, while shear stresses dominate in mode II leading to sliding of the crack, which is most difficult to track. This induces local damage that affects the local behaviour in the region around the singularity, referred to as the fracture process zone or process zone. This apparent nonlinear behaviour raises questions about the definition of the crack tip, its influence on calculated fracture toughness values and its impact on the apparent toughness during delamination and crack propagation [3]. Isakov et al. [117] characterised mode I fracture toughness for high strain rates in CFRP. They used strain tracking instead of force data to bypass limitations of data reduction methods based on the Theory of Linear Elastic Fracture Mechanics. Garcia Perez et al. [118] analysed mode II toughness of carbon/PEEK composites through ENF tests, comparing the compliance method with an infrared thermography technique. They demonstrated that G_{IIC} is sensitive to the crack growth rate due to the transition from ductile to brittle fracture mode at low and high crack propagation rates respectively, as shown in Figure 45.

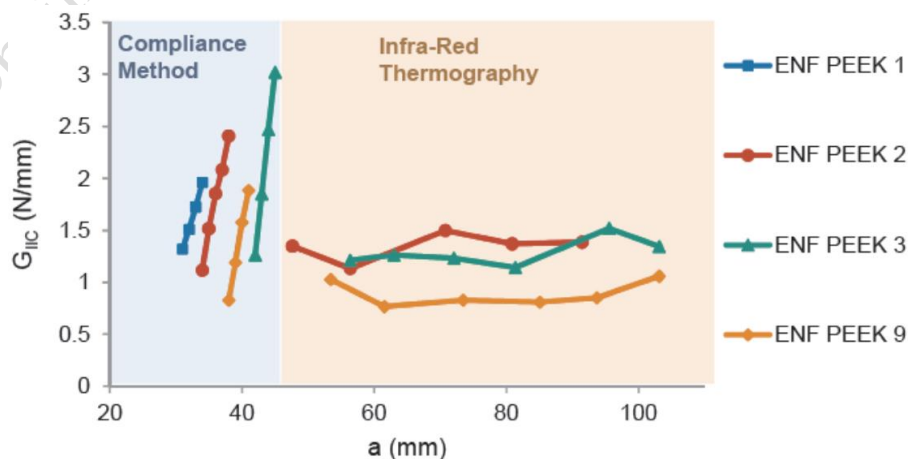


Figure 45. Comparison of the mode II fracture toughness through ENF tests using the compliance method and an infrared thermography technic [118].

Numerical efforts have been done [119]-[122] to predict delamination and debonding at room temperature. Davila et al. [123] proposed a model combined shell element for plies description with a cohesive zone model for interfaces allowing to reproduce both crack onset and crack propagation. Cohesive zone modelling is a local progressive failure models based on continuous damage mechanics which has been extensively used because of two main raisons: (i) the capability to localise nonlinear effects at interfaces between plies or bonded plies, in a zone called Process Zone and (ii) its simplicity and numerical cost-efficiency for consider both crack onset and crack propagation [124]. Other numerical approach is found in literature. For instance, Khalid et al. [125] proposed to use Finite element modelling for described in-plane damage and delamination based on a progressive damage model. Quintanas-Corominas [126] proposed to model delamination by a phase field approach enhanced with a cohesive model, whose numerical results were compared from DCB and ENF analytical solutions. They also proved the interest for addressing coupling between transverse damage and interlaminar failure of composites structures. Dusane et al. [127] studied bridging mechanisms in intralayer and interlayer, and crack simulation by coupling a phase field approach and a cohesive zone model. They showed the potential of this coupled approach for identifying not only delamination but also crack migration through material layers. Kaleel et al. [128] presented a 1D model to simulate the progressive delamination in laminated structures based on the Carrera Unified Formulation (CUF) and component-wise models [129]. They compared their approach with numerical and analytical data from ENF simulations and experimental and numerical data from MMB,

as presented in Figure 46. They proved a good accurate of the method for predicting the macroscopic force-displacement response.

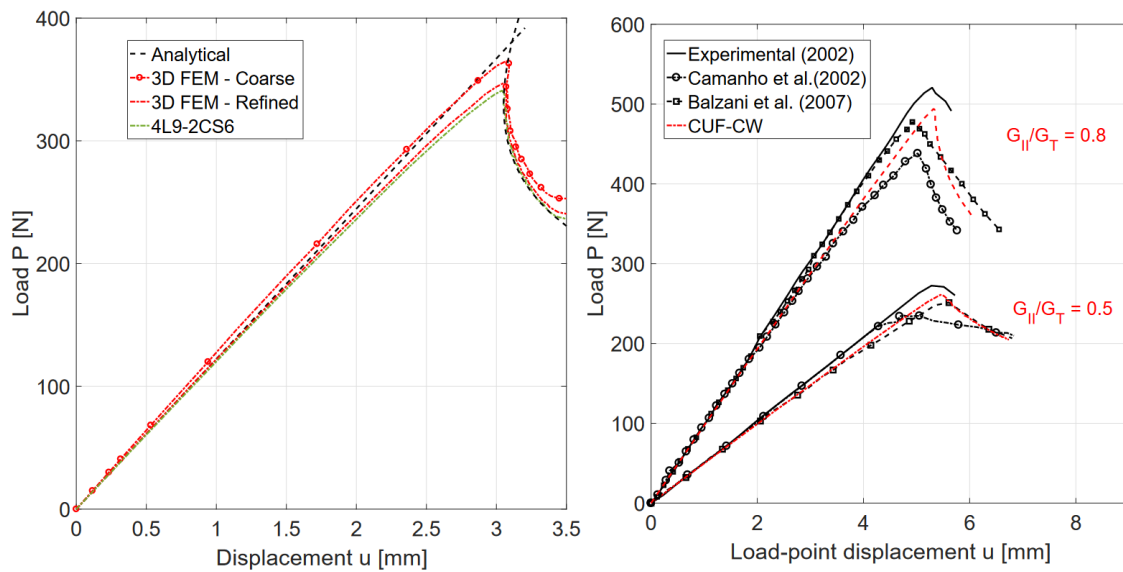


Figure 46. Numerical comparison of (left) ENF and (right) MMB simulations using the CUF-CW approach (green line for ENF and red line for MMB) respect to analytical and FEM approaches [129].

Several authors have proposed in a first instance experimental characterisation of the interface behaviour of composite materials at room temperature using the aforementioned tests to model delamination or debonding in a second time [89],[130]-[133]. Ponnusami et al. [133] developed an inverse modelling approach for wedge-DCB tests to characterise high-rate mode I interlaminar fracture toughness of fibre reinforced composites. De Moura et al. [132] introduced an inverse methodology combining experimental and numerical comparisons to characterise fracture toughness for ductile adhesive layers in bonded joints. They proposed a novel data reduction method based on the equivalent crack concept, due to the difficulty in accurately monitoring the real crack tip during ENF tests. Girolamo et al. [131] presented a combined numerical and experimental approach to determine the interlaminar fracture toughness of bonded composite joints to predict their failure in mode I, mode II and mixed mode I/II. Figure

47 shows the numerical and experimental comparison of the macroscopic response for each solicitation mode. They calculated the energy release rate using the J-integral, accounting for nonlinear mechanisms surrounding the Process Zone. Moreover, the plastic contribution for mode II was incorporated into their numerical model.

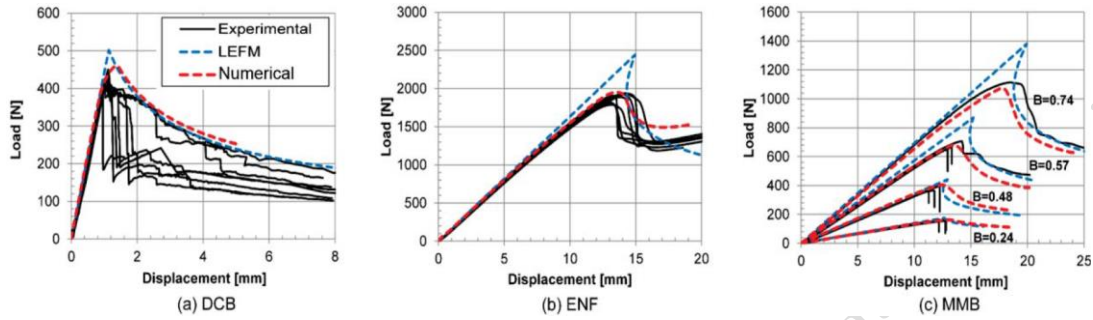


Figure 47. Comparison of the numerical and experimental response of bonded composites using DCB (left), ENF (middle) and MMB (right) tests [131].

Even at room temperature, some authors highlighted the challenges of analysis, particularly when materials exhibit nonlinear behaviour at the interface [134],[135]. Guimard et al. [135] proposed a numerical modelling for mode II delamination in composites based on experimental characterisation using CLS tests. They observed rate effects on the macroscopic response, as shown in Figure 48. They concluded that G_{IIC} is directly dependent on the crack growth rate.

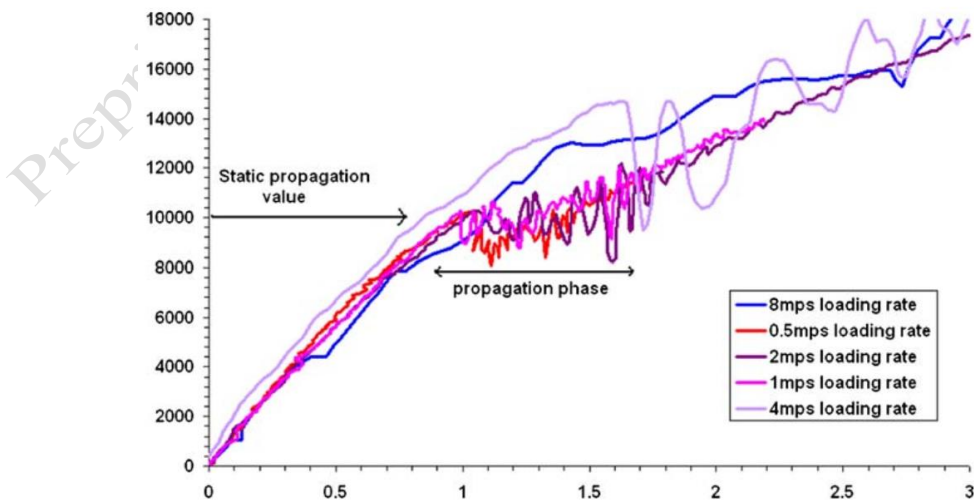


Figure 48. Influence of the solicitation rate on the macroscopic response of CFRP composites using CLS tests [135].

Silversides et al. [136] introduced Acoustic Emission (AE) techniques to detect the delamination onset in CFRP composites and to characterise fracture toughness. They identified micro-damage mechanisms occurring prior to observable delamination, whose contribution to the total energy release rate cannot be addressed by classical data reduction methods. A comparison of results obtained via AE, the compliance method and numerical modelling for DCB, ENF and MMB tests is presented in Figure 49.

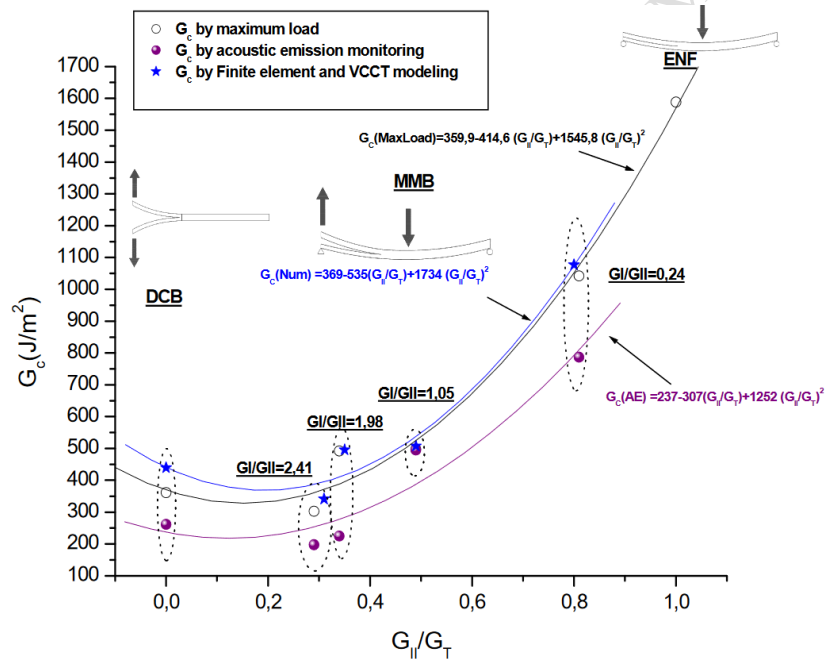


Figure 49. Comparison of different techniques to identify the energy release rate as function of the G_{IIc}/G_{Total} ratio for delamination of CFRP composites [136].

6.2. Nonlinear effects induced by temperature

Addressing the dissipative sources and their contributions to fracture mechanics may be expected to become even more important under elevated temperature conditions leading to the following challenges: (i) Can the reliability of data reduction methods often

proposed in standards remains exploitable for identifying interface's mechanical properties in temperature? (ii) How to overcome the difficulty in the application of experimental test protocols and in the interpretation of these data reduction methods for composites and bonded interfaces? From works presented in Section 5, it seems clear that the adaptation of aforementioned tests to varying temperature conditions is complex, both in terms of experimental setup (e.g., temperature stabilization, thermal inertia) and data analysis.

As mentioned in Introduction and exposed in Section 3.3, some authors [1],[16],[25],[26] have noticed a significant effect of delamination and to some extent matrix cracking on fire behaviour due to the formation of unbound interfaces between the plies. In addition, this is precisely the preferential mode of propagation where temperature delamination remains a catastrophic failure mechanism for composites. Indeed, even if the aperture in mode I could be easily quantifiable by optical measurement compatible with high temperature, the sliding in mode II requires more sophisticated optical and data processing means. Concerning bonded composites, the characterisation of adhesive interfaces is a challenge itself, even if the bulk adhesive behaviour is known. Some authors [48],[110]-[113] propose to use standard tests to get the mechanical fracture properties, but using classical reduction data process.

Based on standardised experimental tests, all the temperature studies mentioned in Section 5 [93]-[110] try to improve the understanding of the temperature effect on the fracture mechanisms by measuring the interface properties in a first time. In presented cases, authors used a climatic chamber or an oven mounted on the load cell to perform the test under temperature conditions for the study of fracture under mode I, mode II and mixed mode I/II. This requires a long heating time and makes multi-instrumentation difficult to install. In addition, the characterisation of mode II is typically more

complicated than mode I due to the different mechanisms of delamination propagation or debonding. This is because mode II is driven by the shear stress, and the crack tends to close due to the bending induced in the specimen. Thus, the visual follow-up of the crack is much more difficult, especially if unstable propagation is observed for the ENF tests. However, as we have already noticed previously, mode II is the preferred mode observed in composite materials exposed to high temperatures as shown in Figure 9 [3],[26] due to the bending load of the composite induced by the thermal gradient.

Most of studies presented in Section 5 highlight the lack of experimental tools for the characterisation of temperature delamination and debonding leads to a big question: How to improve the understanding of the associated mechanisms, in particular temperature-dependent degradation, its impact in the ply stiffness and the interface behaviour as well as matrix cracking and fibre / matrix debonding?. Marquez et al. [137] proven the temperature dependent interface behaviour of CRFP by ENF tests and how the mechanical response at the interface scale is load rate and temperature dependent when viscoelastic behaviour is assumed for the ply behaviour. This aspect seems critical in order to well determine the contribution of different temperature dependent mechanisms (*i.e.*, viscosity, plasticity, damage) to the fracture energy.

For bonded composites, the characterisation of both the bonded interface and the plies interfaces should be done more precisely. In fact, the thin adhesive layer between two bonder plies of the adherents tends to get smoothed and integrated with the plies' interfaces, which make it difficult to separate each mechanical contribution to the material failure.

Concerning the influence of the behaviour of the adhesive or interface layer to the fracture energy, works focused on fracture in durability conditions may be adapted to highlight rate effects and temperature effects on the crack evolution during steady

solicitations [138],[139],[140]. This topic is typically studied in order to propose bonded solution for structure repairing [48],[141].

Modelling the temperature effects remains complex and very phenomenological based [20],[142],[143]. Deeper description of mechanisms involved should be preferred for accurate modelling. For instance, Carpier et al. [144] developed a numerical model based on a representative volume element at the yarn-matrix scale of carbon fibre and thermoplastic resin composites and considering the influence of temperature on both the thermal and mechanical properties (*i.e.* stiffness), below the matrix degradation onset. They do not include such a temperature dependent behaviour on the interface.

The last point is necessary in order to propose adapted model for simulating crack onset and propagation in temperature conditions for both composites and bonded composites. This topic, which is not on the scope of the present review, is addressed by the present literature by adapting existing models at room temperature to a time/temperature dependency, for instance based on cohesive zone models [112],[134]. Despite of the availability of this kind of models some open questions arises: How to validate their accurate to correctly describe observed dissipative mechanics at lower scale (*i.e.*, the interface)? How to efficiently propose temperature-dependent models? This topic is out of scope of this review and will be addressed in a future review in order to discuss the most adapted strategy for good accurate description of mechanisms descriptions and good numerical efficiency.

7. Conclusions

The present work aims to provide a review of experimental fracture characterisation of delamination in composite materials and bonded interfaces subjected to high temperatures. Temperature increase can result from various sources, such as fire, laser impact, thermal flow or environment conditions. However, in such cases, the coupling

between different mechanisms complicates both the characterisation of fracture phenomena and their modelling. Therefore, this review focuses on the temperature effect on fracture behaviour to enhance the understanding of this issue and to support the design of structures exposed to high temperature.

The thermal and mechanical degradation mechanisms have been described, distinguishing between those appearing before and after thermal mass degradation. Damage and fracture mechanisms are considered as catastrophic ones, specially delamination, which can lead to the failure of composite or bonded composite structures.

Delamination and debonding phenomena have been detailed at room temperature to improve the understanding of their causes and their impact on the residual mechanical performance of materials. Specific delamination mechanisms in temperature are described to emphasize the complexity of the coupling between material behaviour and damage evolution leading to delamination and failure.

Standard characterisation tests for both crack onset and crack propagation have been presented in Section 4. Crack onset tests aim to identify the threshold for damage initiation. Crack propagation tests are used to determine the energy release rate for crack growth through data reduction analysis. Standard tests adapted in temperature have been presented in Section 5 and their limitations have been discussed in Section 6.

The main challenges can be identified in two key aspects: (i) the stabilisation of temperature to ensure controlled and repeatable test conditions and (ii) the analysis of data to distinguish the influence of temperature on the different mechanisms of interest (*i.e.*, viscosity, plasticity, damage, fracture).

Further research is required on the fracture mechanics of composites and bonded composites at elevated temperatures to address the following issues: (i) How to improve the experimental setup to ensure reproducibility while maintaining well-controlled

temperature conditions? (ii) How to enhance test observation and data analysis for a better understanding of the underlying phenomena and their interactions?. The next section provides a brief overview of proposed perspectives related to these challenges.

8. Perspectives on advanced experimental setups and data-driven analysis

Based on the present review of the experimental characterisation of fracture properties in temperature for delamination and debonding in composites and bonded composites, the main perspectives can be outlined as follows:

- Development of innovative test devices and setups;
- Integration of multi-instrumentation techniques;
- Advanced temperature-dependent data reduction methods;
- Enhanced synergy from the dialog between test and simulation.

The first point concerns the proposition of novel experimental benches and setups capable of testing samples under controlled temperature conditions. Limited studies have been published on the characterisation of the temperature-dependent interface behaviour. This is mainly due to the difficulties in ensuring both test setup reliability and temperature stability, as discussed in Section 5. For instance, dual actuator device enable experimental testing under various mechanical loading conditions (*i.e.*, mode I, mode II and mixed mode I/II). Such setups can be enclosed to maintain a constant temperature and facilitate the integration of multi-instrumentation. Laser impact devices provide better control over the thermal degradation of materials and could be particularly useful for studying fracture behaviour in composites under isothermal degradation conditions [26].

Multi-instrumentation provides additional data to enhance the understanding of the mechanisms leading to material failure. This facilitates a more detailed analysis of experimental data and should be expected to favour the separation of the different mechanism contribution to the total fracture energy. For instance, techniques such as

acoustic emission [129], infrared thermography [118], digital image correlation [3],[145], infrared spectroscopy, magnetostrictive sensors [146], C-Scan or tomography are particularly useful to detect damage and aiding in the identification of failure scenarios.

Regarding data analysis, most existing standards rely on stress or energy-based criteria for crack onset and on the Elastic Fracture Mechanics Theory for crack propagation. However, these approaches may be questionable under high-temperature conditions, where the temperature-dependent behaviour of composites and bonded composites becomes significant. Therefore, new data reduction techniques should be developed to account for temperature dependency and rate loading effects.

Moreover, the dialog between tests and simulation appears to be a suitable methodology for gaining deeper insight into the failure mechanism of materials in temperature [3],[147],[148]. How can existing model be leveraged to better understand and identify the mechanisms leading to failure? Can the interaction between numerical and experimental data reveal limitations in current modelling approaches and ultimately contribute to design of new experimental setups? These questions remain open and require further investigation. However, this approach seems highly relevant to establish accurate constitutive laws and to improve the identification of rupture criteria directly linked to underlying mechanisms.

The author extends its gratitude to all department involved (DMAS, ONERA, Université Paris Saclay; Institute Pprime, ISAE-ENSMA; IRDL, ENSTA Bretagne) as well as to Copyright holders allowing possible the successful competition of this review.

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Preprint submitted to Mech. Adv. Mater. Struc.