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Review of fatigue of bulk structural adhesives and thick adhesive joints

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ABSTRACT

Fatigue of structural adhesives has been investigated through joints and a little number of works investigate bulk adhesive behaviour itself. Aerospace and automotive engineering focuses more on joint configuration studies, which are correlated with practical applications. Previous works showed that for thin adhesive joints, material properties measured by bulk adhesive testing and joint testing are similar despite the triaxial stress states developing in the adhesive bondlines. However, with the introduction of structural adhesives in construction industry, thicker bondlines have emerged where the bulk adhesive material dominates the joint behaviour. This review work summarises works on the fatigue of bulk structural adhesives used mainly in the construction industry investigating structural adhesives fatigue behaviour either through experiment on joints or on bulk adhesive specimens. The work focuses on thick adhesive bondlines in joints, and discusses the controversy that is over whether adhesive properties from joints or from bulk material should be used.

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Introduction

The history of adhesives and sealants goes hand in hand with that of adhesively bonded connections in several applications. Probably the first ‘inventor’ of a ‘glue’ that is acknowledged in several documents is Daedalus, who, according to the legend of the Greek mythology, engineered wings fashioned from feathers joined with wax to escape from Crete together with his son Icarus. The Greek legend dates more than 3000 years back in time, however, it was not before the seventeenth–eighteenth century when Galileo suggested that there should be in nature a ‘gluey or viscous substance to bind materials together’ and Newton conjectured that ‘There are agents in nature able to make the particles of bodies stick together by very strong attractions’ [1]. The first mention for a glue in patent literature comes from a British patent of 1754, although a massive production of adhesives (glues) started much later, at the beginning of the twentieth century [1]. Glues were sold on test; i.e. the price of a product was governed by its strength and other properties, measured with several divergent tests. It is interesting to mention that by 1922 the most common testing procedure was the ‘finger test’. An experienced engineer was breaking a sample of the glue with the thumb and forefinger of each hand giving an indication of the glue quality. If the glue was fractured evenly and bended but little, low strength and brittleness were indicated. If a thin sheet was bended well and in case it was breaking, showing a

splintery fracture, good strength was indicated. This test, however, was not more than a method to rank glues by comparison [2].

Until that time, most of the structural adhesives were of natural origin. Phenol-formaldehydes are generally regarded as the first true, fully synthetic polymers, with other formulations introduced the following years as shown in Table 1 [3].

Since their introduction, modern structural adhesives were used as alternatives to traditional ways of joining similar and dissimilar materials. Initially adhesives were used as sealants in secondary applications, later on for gluing non-structural elements, while with the experience gained from their use, progressively they gained their place as structural adhesives in primary structures in a wide range of engineering domains. As such, epoxy adhesives gained rapid success in aerospace, automotive, construction, electronic and woodworking applications, largely because of their ease of use, versatility and mechanical properties.

The mechanical properties must be measured as precisely as possible – in contrast to the ‘finger test’ that was initially adopted – through an abundance of experimental fixtures, machines, techniques and procedures that were devised during the last century in order to measure the load necessary to break the adhesive. Examples of equipment and methods for measuring the mechanical properties is the jelly

Table 1. Historical development of structural adhesives [3].

Approximate date of commercial availability	Adhesive
1910	Phenol-formaldehyde
1930	Urea-formaldehyde
1940	Nitrile-phenolic, vinyl-phenolic, acrylic, polyurethane
1950	Epoxies, cyanoacrylates, anaerobic
1960	Polyimide, polybenzimidazole, polyquinoxaline
1970	Second-generation acrylic

strength testing machine [2] or methods for the stress-strain analysis of the bulk specimen, the derivation of the physical properties of the adhesive, tensile tests of joints (butt joint) or shear adhesive testing through tensile joints testing (single/double lap joints), see Brockmann et al. [4]. In parallel, a number of standardised procedures were developed, as well described in Hussey and Wilson [5].

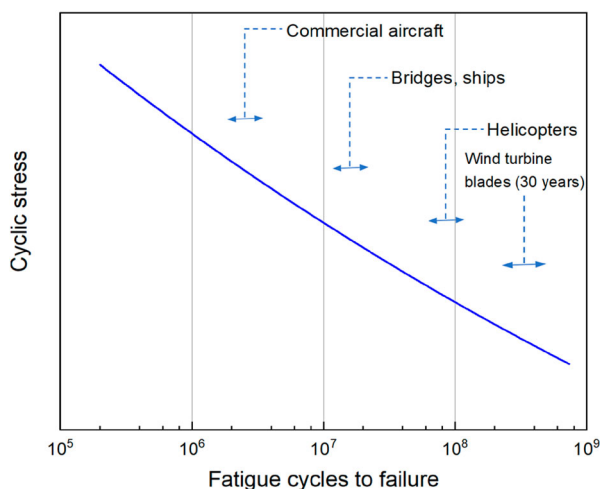
A large volume of data has been collected, especially after the Second World War, compiled and tabulated in order to be available to designers. These data contained, among others, shear, tension, fatigue, and stress-strain information on laboratory specimens, mainly adhesively bonded joints [6]. Nevertheless, controversy still exists as to whether adhesive properties in the thin film form (which is how they are normally used in aerospace joints) are the same as when bulk specimens are prepared. Useful data on the elastic properties of structural adhesives can be provided by both specimen configurations if purely elastic behaviour is considered [7]. However, the stress state in bulk adhesives is simpler than in adhesive bondlines, while bulk adhesive specimen dimensions can meet (fatigue) testing standards [8]. Differences in the stress state within the specimen configuration, rather than material related differences can cause differences between the apparent properties, namely the shear strength and the tensile strength in Burst et al. [9] for thin film in-situ and bulk adhesive samples. The behaviour of a

joint is affected as well as from the dimensions. A typical long-overlap structural bonded joint behaves frequently very different from equivalent short-overlap test specimens [9]. While, simple, tensile-shear tests on joints are adequate for determining certain parameters, they barely apply to different bond dimensions or surface conditions, or other adherend materials [10]. Bulk specimens are not susceptible to the problems inherent in joint tests and are more suitable for providing reliable data on the response of adhesives to various known states of stress although care should be exercised in controlling the curing schedule [7].

Adhesives, like metals, suffer from fatigue, though the phenomenon is far from being understood [11]. As for the composite materials [12], the fatigue performance of adhesives has been investigated systematically since the 1950s. As the polymer matrices play a significant role in the fatigue behaviour of composite laminates [13–16], adhesives play a significant role in the fatigue behaviour of adhesively bonded connections.

Works on the fatigue behaviour of structural adhesives were mostly limited to joint configurations [17–22] while fewer works have been carried out on bulk adhesive specimens, e.g. [23–33]. In civil engineering, adhesives have been used for more than 50 years [34,35] for connecting structural elements without harming their structural integrity by drilling. The use of adhesives, initially, has been limited for strengthening, maintenance, or secondary connections [36] as was evidenced by the development of several retrofitting reinforcement methods [37,38]. Nevertheless, recently structural adhesives have been used in critical bonding of structures, notably in bridge engineering and the wind energy, as they offer advantages against other joining methods [39,40]. Applications in bridge and wind-turbine engineering have large and thick bondlines, of several millimetres and usually undergo multi-axial loading conditions [12,39,41–43] for a high number of fatigue cycles as shown in Figure 1.

In civil and structural engineering, for example in bridge construction [39], wind energy [41,44], or marine applications [45] only few experimental data exist concerning the fatigue performance of bulk structural adhesives, despite the existence of numerous structural applications operating under fatigue loadings. Big volumes of, mainly, epoxy, vinyl ester, acrylics and urethane adhesives are used today. In wind energy for example, adhesives used for structural bonding of panels and webs in wind turbine rotor blades, for bolt, stud and metal insert fixing, for void filling, for bonding the magnets in the nacelle, for blade repair etc. In these engineering domains, adhesive bondlines are much thicker compared to aerospace engineering. Bondlines reaching or exceeding 10 mm can be found in bridge construction and marine applications, while much thicker ones reaching 30 mm can be found in large wind turbine rotor blades [41,46–48].

**Figure 1.** Fatigue lives of several applications from EN 1991-2 Eurocode 1 [43].

Paste, rather than film adhesives are commonly used in such thick bondlines to compensate for tolerances.

In such cases, the study of the bulk material properties becomes inevitable, since the adhesive volumes in connections are significant and are usually dominating the fatigue performance of the adhesively bonded joints. Since certain properties, such as the (fatigue) tensile strength, stiffness, strain at failure, and Poisson ratio can hardly be estimated by joint experiments, investigations of the bulk specimen performance are indispensable when such material data should be implemented in numerical/analytical modelling [49]. Nevertheless, compared to single lap-shear tests, fatigue data from bulk specimens might be more reliable and would certainly be more applicable to other designs of joint, although suitable configurations for evaluating the fatigue properties of adhesives, such as the thick adherent specimen were introduced [7].

The objective of this article is to review works investigating structural adhesives fatigue behaviour either through experiment on joints or on bulk adhesive specimens. The work focuses on thick adhesive bondlines in joints, mainly used in civil and structural engineering applications, and discusses the controversy that is over whether adhesive properties from joints or from bulk material should be used.

Bulk structural adhesive or joint testing?

According to the ASTM definition, [50], adhesive is a substance capable of holding materials together by surface attachment. Adhesive is the general term and includes among others cement, glue, mucilage, and paste. Bulk adhesive is an adhesive *unaltered* by the adherend. Structural adhesive is a bonding agent used for transferring required loads between adherends exposed to service environments typical for the structure involved. This is also a general definition. The difference between structural and non-structural adhesive is not clear. Some researchers try to differentiate between structural and non-structural adhesives in terms of some arbitrary tensile shear strength procedure, such as e.g. the one designated in ASTM D-1002 [51]. Bolger [52] narrowed the definition of structural adhesives to thermosetting resin compositions used to form permanent, load-bearing joints between two rigid, high-strength adherends.

Scientists did not always agree on the appropriate way of testing an adhesive in order to estimate its properties and use them for the design of bonded joints. Although structural adhesives are used explicitly in joints, it is questionable whether or not their mechanical properties should be measured from joint experiments, or from bulk adhesive experiments. In any case, to determine the stresses and

strains in adhesive joints in a variety of configurations, it is necessary to characterise the adhesive behaviour to know its mechanical properties, particularly the stress-strain curve and the modulus [53]. Researchers and designers need true strength and modulus values of the adhesive for simulation purposes (such as finite element analysis). These properties are generally obtained by testing a bulk specimen in tension, see, e.g. Figure 2, or in torsion as described in Zarouchas and Nijssen [41], or alternatively by testing adhesive joints under pure shear, see, e.g. Figure 3 for a geometry of a double lap joint. ASTM D5656 suggest the methods for determining the stress-strain behaviour of adhesives in shear by tension loading. The adherends used in this standard test method are thick and rigid in order to minimise the peel forces obtain shear stress-strain properties of the adhesive. Nevertheless, it is clearly stated in the same standard that the stress is not totally uniform across the over-lap of the specimen, factors such as adhesive stiffness, adherent stiffness and location of shear strain measurement along the length of the bond can affect the experimentally obtained load-displacement curve.

For bulk material investigations, the specimens and test methods are comparable to those used for plastic materials. Properties determined are intrinsic to the material, and they are obtained under a uniform and uniaxial state of stress, with no influence of the adherends. Deformations of bulk specimens are easily measured using standard extensometers or strain gauges. Joints are structural components and their investigation provides an estimate of their behaviour, but in most of the cases they do not provide adhesive material properties, since complex stress states develop

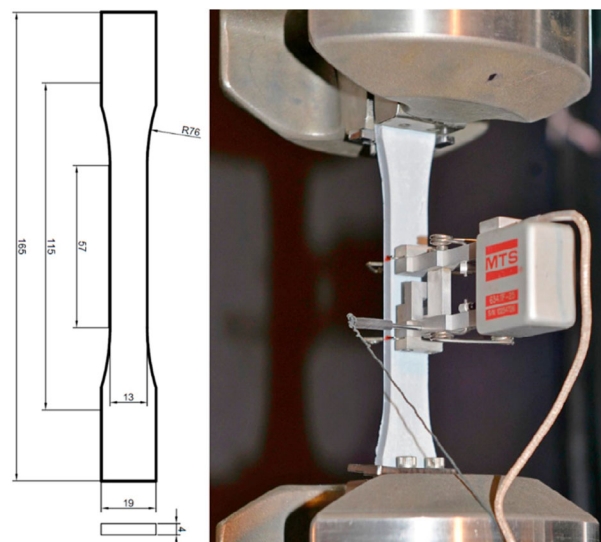


Figure 2. Geometry of dumbbell specimen (ASTM D7791-12) (a) and dumbbell specimen mounted on the loading frame equipped with clip gage [54].

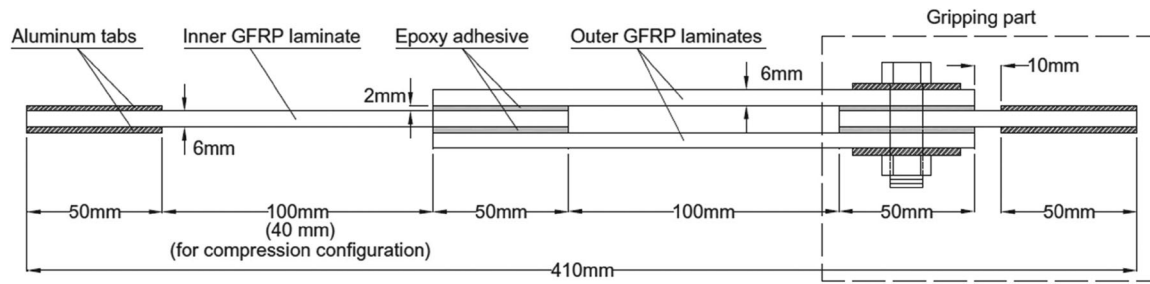


Figure 3. Double lap structural joint geometry [55].

in the adhesive [7,48]. When interpreting the data obtained from joint specimens, it should also be remembered that most of the adhesive is subjected to a triaxial stress state which, apart from increasing the stiffness, will probably affect the yield or failure behaviour. The triaxial stress state depends on the adhesive itself, as was shown in Imanaka et al. [56] for an epoxy and an acrylic adhesive. The popular belief that an adhesive has different properties in bulk or in joints is essentially due to the fact that, in almost all bonded specimens, the adhesive layer is in a complex and non-uniform state of stress [57]. However, for film adhesives it was proved that properties were similar for film form joints and bulk specimens after testing in tension, compression on bulk specimen and shear tests in joints, [57–61], although it was mentioned that the adhesive layer is in a complex stress state, see, e.g. [57]. In the linear elastic range, the stress/strain relation obtained from shear tests on thin-film adhesive specimens can be directly compared with the stress/strain relation obtained from tension tests on bulk adhesive specimens. For tougher adhesives joint testing can give slightly higher properties than bulk adhesive testing as shown in Refs. [58,61].

The building construction industry represents one of the largest users of adhesive materials. Uses of adhesives in the construction industry and typical adhesive specifications are presented in Lees and Hutchinson [62] together with a representative set of adhesive material data that are necessary for design and specification. A combined experimental and numerical investigation was performed in Burst et al.

[9] to determine whether the material properties of structural adhesives differ between their thin-film ‘in-situ’ and bulk forms. The investigation showed that the apparent shear strength is independent of bondline thickness and similar to that obtained from bulk adhesive V-notched Iosipescu specimens. The tensile strength estimated by dumbbell bulk adhesive specimens and butt joints (see Figure 4) was slightly different depending on the bondline thickness. The results of this investigation suggest that the observed differences in apparent shear and tensile strengths of structural adhesives between their thin-film in-situ and bulk forms are due to differences in the stress state within the specimen configurations and not material related differences. The authors supported that the mechanical properties of adhesives determined from the testing of bulk adhesive specimens are suitable for use in the design and analysis of adhesively bonded structures. Investigation of both bulk adhesive behaviour and joint behaviour in Lees and Hutchinson [62] revealed that the bulk properties have more influence in a thicker bondline, although the investigated thicknesses were still limited – between 0.2 and 0.6 mm – compared to the thicknesses of structural applications. The investigation showed that the changes in bulk behaviour with increasing temperature occur at around the same temperature as changes in the behaviour of the bonded joints. Therefore, changes in the joint with temperature are dominated by the adhesive behaviour, and thus bulk adhesive testing seems necessary to gain fundamental knowledge. This is supported by the fact

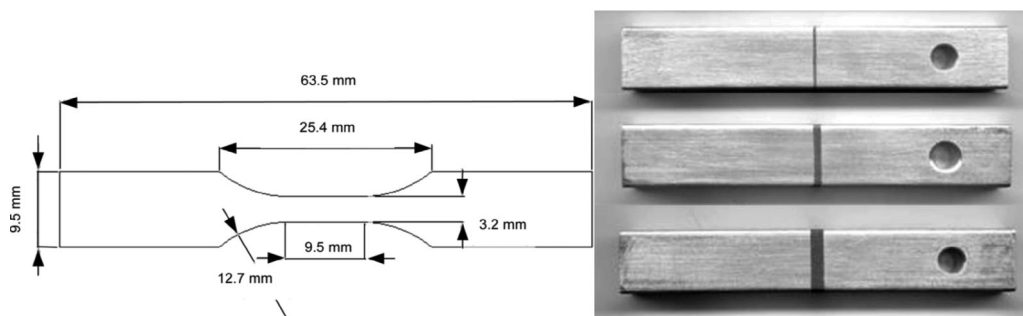


Figure 4. Bulk adhesive tensile specimen and butt-tensile specimens with three different bondline thicknesses (0.25, 1.27, 2.54 mm) [9].

that the joint behaviour varies with temperature/time/ bondline thickness/overlap length/surface treatment etc. [63]. Therefore, data developed over a wide range of conditions are required to assess adhesive material characteristics. Great care must be taken in carrying out the tests and interpreting the results, since most of the tests involving adhesive joints do not give directly interpretable values. In Adams [64], it was suggested that the best test for determining the mechanical properties of an adhesive is to make specimens from bulk samples, taking care that these have reached the same state of cure as the investigator believes to be the case. For some adhesives, exothermic reactions on curing may cause a runaway temperature increase, leading to overheating and damage. Nevertheless, although as shown in e.g. [9,62] bulk property data can be used to predict the mechanical performance of bonded joints, joint testing cannot be ignored as there is no other suitable means of determining adhesion properties.

In a joint configuration, failure is essentially always cohesive, in the adhesive or adherends or both, or in some boundary layer [65]. For Sharpe [65], it is troublesome to consider adhesive joints to be 'simple' composite structures of adherend 1, adhesive and adherend 2, in their three bulk solid states, each characterised by its own single properties. Therefore, although most failures in joints are cohesive, with cracks running solely in one of the constituent materials (adhesive or adherends) each solid may have *surface layers* that can affect their mechanical response. Therefore, the analysis of the mechanical behaviour of adhesive joints must take into account all the possible present boundary layers, which can be achieved by testing the assembly as a whole. Nevertheless, this approach does not allow the estimation of the material properties, but rather the estimation of the mechanical performance of the given joint configuration, necessitating the investigation of a wide range of conditions. This approach has another deficiency since the stress distribution in the bondline depends on the adhesive stiffness,

however, this can be estimated only by bulk adhesive testing [66].

In most bulk specimens, but also in joint configurations, a main problem associated with adhesives or adhesive bondlines is the void formation during manufacture, although great care is taken to avoid them [64]. Lees et al. [62] managed to improve the material quality by centrifuging the material before casting. Jeandreau [57] suggested the fabrication of a plate for cutting the adhesive specimens, to prevent voids and surface parallelism. This method was proved efficient with epoxies, polyurethanes and modified acrylic adhesives but was not applicable to anaerobic and cyanoacrylic adhesives because of their curing process. Foletti et al. [66] suggested the degassing with vacuum before casting. Nevertheless, although such methods are feasible for specimen testing, voids in bondlines of structural applications cannot be avoided. Typical examples of this are the wind turbine rotor blades that contain several square metres of thick bondlines [67], see, e.g. Figure 5. In a wind turbine blade with a length of 60 m approximately, 500 kg of bonding paste are used and adhesives contribute strongly to the structural integrity. This accounts for around 12% of the total blade weight, since a 60 m blade weights around 4000 kg [48]. In order to model the adhesive mechanical behaviour with hydrostatic pressure dependence, the response of the material for different loading modes is needed in the experimental database [68].

The adhesive layers that can have thickness reaching 30 mm [48] usually include several local, but large, voids, as shown in Figure 6 [69] and Figure 7 [70,71]. Gaps as large as 12–15 mm and voids in bond lines – some up to 50 mm long – were observed in a 43 m rotor blade [72].

The adhesive bonding of the different parts in a wind turbine blade and other applications of the construction industry provides a special challenge as it is difficult to scale down the thickness of the bondline. Bondlines in full-scale wind turbine blades are typically 10–15 mm thick and with a scaling

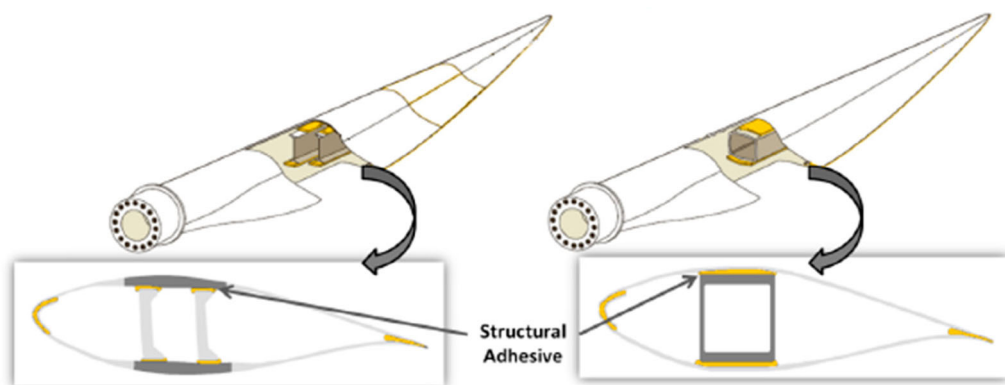


Figure 5. Example of structural bond lines on two wind blade designs [68].

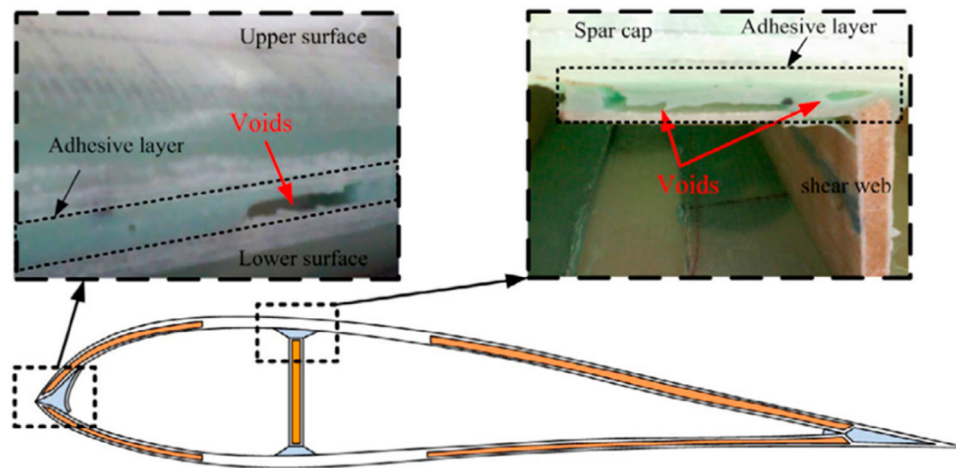


Figure 6. Voids in the bonding lines of composite wind turbine blades [69].

factor of 5 it is a challenge to realistically make bondlines of 1–2 mm thickness for the down-scaled blade [73]. Adhesive bondline thickness of contemporary wind turbine blades is much larger [41,63] and therefore the problem of downscaling for experimental investigations becomes more pronounced. This scaling problem was identified very early by Hart-Smith [10] who recognised that the behaviour of typical long-overlap structural bonded joints is frequently very different from equivalent short-overlap test specimens.

Bulk adhesive behaviour becomes more important with thickness [9,62], and in such cases fatigue data on bulk adhesives might be more reliable than those from joints [7]. Nevertheless, it seems that for thin bondlines, experiments for joint and bulk adhesive provide similar adhesive properties if appropriately designed and executed. However, most of the evidence comes from quasi-static investigations and only few works on fatigue exist. For thicker bondlines that are usual in the construction industry, e.g. in the wind and bridge-engineering, the bulk adhesive properties seem to be more crucial for an appropriate modelling of the assembly behaviour. Due to the sizes, downscaling for laboratory testing is not always appropriate and can lead to erroneous results. Such thick bondlines associated with complex stress configuration require a multiscale approach starting from the characterisation of the bulk material, to model the behaviour of thick adhesive joints and eventually subcomponents

as it was shown by Fernandez et al. [70]. Furthermore, engineering structures with thick bondlines usually operate under fatigue loadings and are susceptible to fatigue failure. It is often assumed that the safety factors used in wind blade design are taken too high [68,74,75]. In order to determine safety factors more precisely, detailed information about the effects of different loading conditions (including fatigue loading) of the wind blade materials, including the adhesives, is necessary.

Fatigue behaviour of adhesives by testing bonded joints

Basic considerations – thickness effect

Adhesives are generally designed to work in shear, so details of their stress–strain behaviour under shear loading are of the first requirements for design [1]. In an abundance of applications, adhesives used in joints [38,76–80] in order to transfer loads between adherends. Bulk adhesive and/ or bonded joint tests are used for, e.g. comparing the mechanical properties of a group of adhesives, or as a quality check for a batch of adhesives, or for determining quantitative mechanical and physical data on adhesives for structural design and analysis [80]. While bulk adhesive tests are useful for determining quantitative mechanical and physical properties, data from joint tests usually reflect the mechanical properties of the

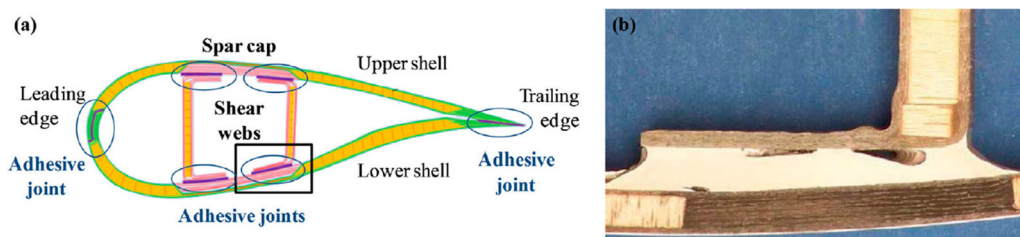


Figure 7. Typical wind turbine blade cross section (a) and cross section of the adhesive joint from spar cap to shear web in detail (b) [70,71].

adhesive as well as the degree of adhesion and the effectiveness of surface treatments. It is widely accepted that adhesive layers in joints are not in simple loading conditions (e.g. not in pure shear in a lap joint, as tearing stresses develop at the end of the bondline) and that triaxial stress fields develop in the adhesive layers. Nevertheless, joint testing has been used, more than bulk adhesive testing, for the investigation of the fatigue performance of adhesives. Yet, there is always a debate (similar to quasi-static loading) as to whether the in-bondline, or thin-film form, properties of the adhesive are the same as they are in bulk [81].

De Goeij et al. in 1999 claimed that 'Designing joints using adhesive properties that are based on the results of experiments on bulk adhesives cannot be used to predict the fatigue performance; the fracture behaviour depends strongly on the adhesive bond line thickness' [42]. This difference was attributed in De Goeij et al. [42] to the difference of the adhesive stress state in bulk and in the joint. The adhesive layer is in plane-strain and the triaxial stress field is expected to increase the stress for yielding compared to the plane stress conditions in the bulk adhesive. However, Xu et al. [82] suggested that if the restriction by the substrates is relaxed, e.g. by increasing the bondline thickness, the fatigue crack growth (FCG) rate will tend to approach that of the bulk adhesive; This implies that FCG rate in thick adhesive joints should be the same as FCG rate in the bulk of the adhesive itself. Xu et al. [82] supported this argument with fatigue/fracture experiments of joints with different adhesive thickness and two bulk adhesives, one brittle and one ductile, assuming that for the bulk adhesives the bondline is taken as infinite.

The experimental results, shown in Figure 8, suggest that the increase in the bondline thickness is more effective in reducing the fatigue crack growth rate for the joints bonded with the ductile adhesive

B. Ductile adhesives are more crack resistant since they are more capable of extending their plastic zone in the presence of constraining substrates [82].

To the authors' knowledge, works comparing the adhesive fatigue/fracture from bulk adhesive specimens or thick adhesively bonded joints are very scarce in the literature. In contrast, the effect of the adhesive bond thickness on the fatigue/fracture behaviour of adhesively bonded joints, as well that of other parameters affecting the fatigue performance of adhesively bonded joints, have been investigated in several works, e.g. [21,80–96]. A comprehensive review of the influence of parameters on failure of composite-composite adhesive joints under cyclic loading is presented in De Goeij et al. [42]. Works were focused either on fracture mechanics joints or on structural joints [83] and the investigation in both cases (for the majority of the works) was on the system level and not on the individual materials aka the adhesive and the adherends. Fracture mechanics joints for Mode I, Mode II, mixed-Mode I/II have been studied under fatigue loads, while Mode III and mixed-Mode I/III and II/III fatigue loading conditions have not been investigated [84]. Nevertheless, Mode III has been proven the dominant fracture mode in the trailing edge of wind turbine rotor blades, as described in Eder et al. [85].

A brief literature review, given in Pascoe et al. [86] shows that the fatigue crack growth (FCG) rate reduces with the increase in joint thickness, and fracture toughness increases [87–90], which is assigned to the effect of constraint (provided by the adherends) on the plastic zone size in the adhesive layer. Removal of constraint (by increasing thickness) is thought to result in more plasticity, which then increases the crack resistance, thus reducing the fatigue crack growth rate [82,86]. The reduction of failure load with increasing the bondline thickness is very common when dealing with

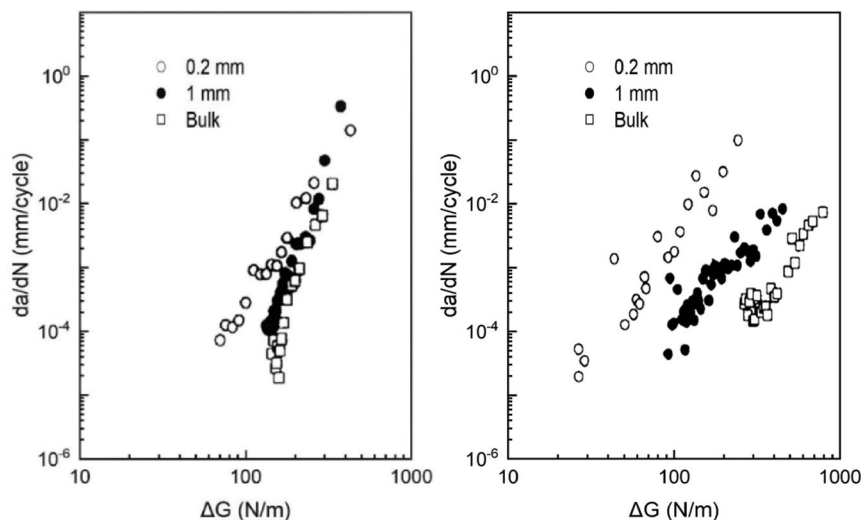


Figure 8. Fatigue crack growth rates in bulk adhesive and joints with 0.2 and 1 mm bondline thickness at 2 Hz. Adhesives: A – brittle, B – ductile [82].

structural adhesives [91]. Fewer works showing no effect of thickness [92] or even increase of FCG rate with thickness [93] exist, while others suggest that the effect of thickness is related to the adhesive itself and is different for brittle epoxy resins and different for thermoplastics, see, e.g. [88,94]. This agrees with the findings for the thickness effect on quasi-static behaviour as described in Refs. [7,61,87]. The critical strain energy release rate for quasi-static fracture increased linearly with bondline thickness [87], resembling the scaling effects on Mode I fracture of GFRP DCB laminates showing R-curves dependent on the specimen thickness, which considerably affected the energy release rate value at the plateau [95,96]. Investigations on the specimen thickness effects (not the adhesive bondline) on the fiber-bridged fatigue delamination growth in composite laminates [97,98], showing that the traction–separation behaviour in the bridging zone is dependent on the specimen geometry as well as the constituent materials [97].

Effect of loading parameters on fatigue/fracture behaviour

Fracture mechanics joints with several different materials configurations and geometries were used for the investigation of the R -ratio (the ratio between the minimum over the maximum cyclic load/stress/displacement) effects on their fatigue performance. Results from Mode I, Mode II, and mixed-Mode experiments are usually presented on the so-called fatigue crack growth (FCG) plots, such those shown in Figure 7. While the ordinate of this double logarithmic diagram is always the crack propagation rate, different parameters were proposed for the abscissa [99–106] attempting to assess the R -ratio effect. In principle, as a rational result of the experimental process the $G_{\max}$ (maximum cyclic strain energy release rate) or ΔG was used [99–101]. However, other parameters were proposed, e.g. the range of the applied energy release rate in the fatigue cycle ($\Delta\sqrt{G}$) in order to use a modified version of the Hartman-Schijve crack-growth equation for the modelling, or the energy dissipation per fatigue cycle [104], a measure of the amount of energy available for crack growth. According to Pascoe et al. [104], the total amount of crack growth depends on both the available energy and the resistance to crack growth, i.e. it depends on both the maximum load and the load range, and therefore it is not appropriate to use $G_{\max}$ in the abscissa of the FCG diagram.

Shahverdi et al. [99,100] investigated the R -ratio effects on the Mode I, Mode II and mixed-Mode fatigue crack growth of adhesively bonded pultruded GFRP DCB joints under constant amplitude loading and plotted the results on the da/dN vs. $G_{\max}$, see, e.g. Figure 9 and vs. the $\Delta G_{\max}$.

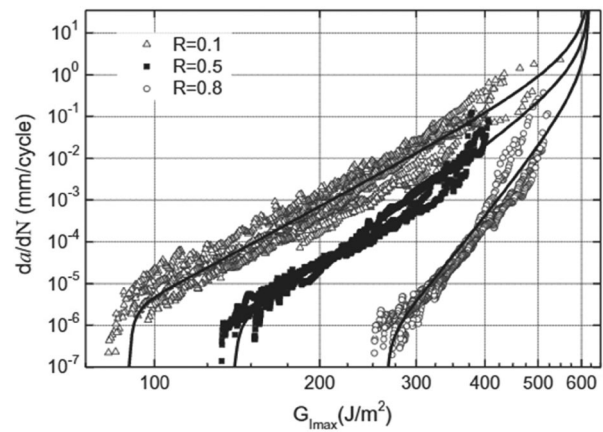


Figure 9. Crack growth rates vs $G_{\max}$ for Path II-A [99].

Fatigue crack growth curves exhibited high dependence on the R -ratio independent of the strain energy release rate fatigue parameter used for the representation. The FCG curves corresponding to higher R -ratios exhibited higher slopes in the middle part resulting in higher fatigue threshold values. When plotted on the $G_{\max}$ - da/dN -plane they all reach the same maximum value, similar to that of the critical strain energy release rate derived from quasi-static experiments. When plotted on the ΔG - da/dN -plane, FCG curves corresponding to higher R -ratios show higher slopes and tend to exhibit the same fatigue threshold value, compared to those corresponding to $R = 0.1$. Crack growth rate was also dependent on the crack path inside the multidirectional pultruded adherends of the examined joints [107].

The authors suggested also a total life model for the prediction of the R -ratio effects on fatigue crack growth of adhesively bonded pultruded GFRP joints (solid lines in Figure 8) and they proved that the model could accurately simulate the behaviour of the examined material and was capable of predicting the behaviour exhibited under unseen loading conditions in Shahverdi et al. [20].

In the abovementioned works the joint performance was investigated and not the behaviour of the adhesive as a material in itself. Rocha et al. [108] attempted to compare the fatigue/fracture behaviour of three different adhesive systems, an epoxy-based, an acrylic and a rubber-like adhesive. Nevertheless, no valid comparison was established since the adhesive thickness was different for each material and only the failure mechanisms involved in the crack growth process were discussed.

For the epoxy based adhesive a shear yielding mechanism was dominant, crazing was the main mechanism for the acrylic adhesive, while the rubber-like adhesive that had a glass transition temperature close to the room temperature, failed by cavity initiation, growth, and coalescence, under an unstable viscoelastic fracture. Naito et al. [109] suggested that

the fractured surfaces of epoxy adhesives under Mode I static and fatigue loadings have fractal characteristics. In their work it was concluded that the fatigue crack growth resistance and threshold dramatically increased due to acid treatment of steel adherends, confirming the argument of Sharpe [65] regarding the existence of the surface layers of each solid (in this case the adherends and the adhesive) that can affect their mechanical response.

Fracture mechanics joints have pre-cracks and for design purposes the fatigue threshold (G_{th}) should be estimated for a safe life design. Nevertheless, in practice pre-existing fatigue cracks rather than well-defined pre-cracks are present. According to Dessureault and Spelt [110] such cracks take more time to begin growing under a new load range than with a fast Mode I pre-crack. The time (or the number of cycles) for crack initiation is important in the case for structural joints as well, since no pre-crack exist and the fracture is expected to initiate in a natural way from a defect, i.e. a pre-existing fatigue crack. For structural adhesive joints, the life to crack initiation showed a high degree of variation, reaching from 0.7% to 96.7% of total life [19]. This variation can be explained by the fact that the examined structural joints contained no pre-crack and no special care was taken to precisely shape the adhesive edge (no fillet was formed, see above) during fabrication. Therefore, the crack could initiate at different locations in the joint, caused by existing defects, possible small asymmetries due to fabrication and/or loading misalignment. Other works report that the fraction of life for crack initiation represents a significant part of the joint fatigue life, ranging from a minimum of about 20% up to more than 70% [111]. The relative fraction of life spent in the crack initiation is also reported to depend on several parameters related to the joint materials, the overall and corner geometry, the load level and the presence of defects in the bondline, among others [111–113].

During the initiation phase, and before the development of a visual crack, short cracks in the adhesive microstructure, might be present. There are several works discussing the short crack effect on the fracture behaviour of metals, all agreeing that under the presence of short cracks the crack growth rate can be significantly higher than in tests with long cracks. Only a limited number of attempts to investigate this same effect on

composites and adhesives were presented, however, see, e.g. the works of Rhys Jones and Tony Kinloch [105,114,115], mainly questioning whether the fatigue threshold for defects in composite structures is a function of the size of defect for small defects in composite structures [114] and if a Hartman–Schijve formulation can be appropriate for the modelling of the fatigue crack growth behaviour of composite materials, considering the short crack growth and the R-ratio effects [105].

The short crack effect on composites has been investigated experimentally since the 1990s, see, e.g. [116] investigating cleavage specimens with no cracks, short cracks (0–1 mm) and longer cracks (1–3 mm) under Mode I and mixed-Mode loading. The results showed that the presence of short cracks did not affect the specimen strength. Although a numerical analysis showed rapidly increasing failure loads at vanishingly small crack lengths, the authors of Ref. [116] clearly stated that this does not happen in practice as failure occurs through plastic deformation and rupture rather than by crack growth at these short crack lengths.

Structural joints – fatigue life approach

Structural adhesive joints have been intensively investigated aiming to assess the effects of geometrical, loading and environmental parameters on their fatigue life, usually in the form of double lap, or single lap joints as those shown in Figure 10.

In such joints, the adhesive type affects the fatigue behaviour, since the commonly used adhesives, especially in civil and structural engineering, are sensitive to loading type and rate effects, and temperature and humidity changes. Nevertheless, as suggested by Hart-Smith [118] the adhesive should never be the weak link in a joint, and therefore, well-designed composite adhesively bonded joints were failing outside the adhesive bondline. Consequently, from these experimental surveys, the adhesive material properties were not derived, but rather it was the overall joint performance that was investigated.

Most of the works on fatigue of adhesively bonded structural joints report the observed influence of each effect (loading, temperature, humidity) on the fatigue life of the examined joint. Although some common trends and results can be identified in this procedure, it can be affirmed that a general scientific description of involved phenomena is not available [119].

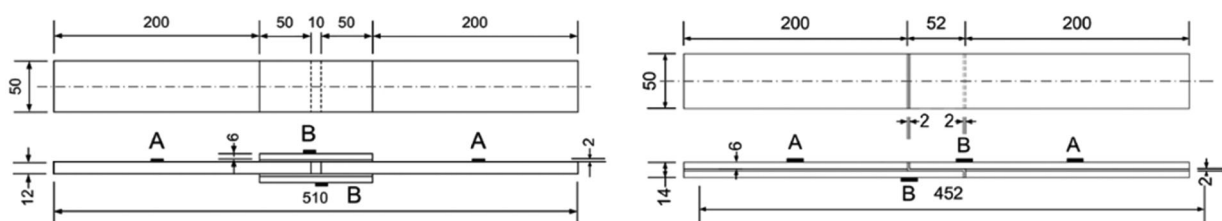


Figure 10. Double lap (left) and single lap (right) joints [117].

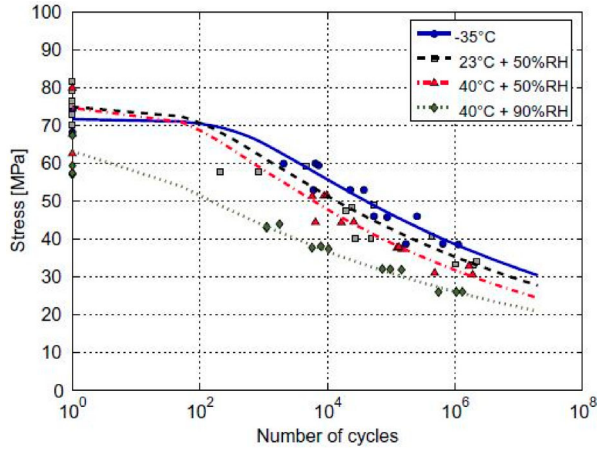


Figure 11. Double lap joint S–N curves at different thermo-mechanical loadings [121].

As expected, the quasi-static [120], as well as the fatigue life of composite adhesively bonded joints under different temperatures is dominated by the adhesive behaviour, the most sensitive of the joint materials to temperature changes, see, e.g. [121–124]. The joint fatigue life at higher temperature is usually shorter than at lower temperature (see Figure 10), especially when reaching or going above the glass transition temperature of the adhesive [121,122]. The fatigue performance is deteriorated further due to the increase in moisture as also presented in Figure 11.

The failure mechanism changed with increasing temperature from fiber-tear to adhesive failure in Zhang et al. [121], showing a deterioration of the joint performance due to the sensitivity of the adhesive material to temperature increases. The addition of humidity tends to shift the failure to the adhesive/composite interface. Although the joint behaviour is driven by that of the adhesive, no information about the adhesive properties can be taken from such experiments.

The mean cyclic load affects as well the fatigue behaviour of structural adhesive joints, as described in a series of publications, e.g. [17,125,126]. All published works agree that the behaviour is dominated by creep mechanisms at low frequencies and by the temperature at higher frequencies, especially when ductile adhesives are used [126]. The fatigue data of adhesively bonded joints, independent of the adhesive used, can be fitted by typical S–N curve equations, as those used for the simulation of the fatigue behaviour of composite materials [12,127]. Between them, the power law equation, introduced by Basquin in 1910 [128], is the most commonly used, see Equation (1).

$$\sigma_{max} = \sigma_0 N^{-\frac{1}{k}} \quad (1)$$

The value of the exponent, which denotes the slope of the S–N curve, is related to the examined material. It is now documented that for composite materials and for

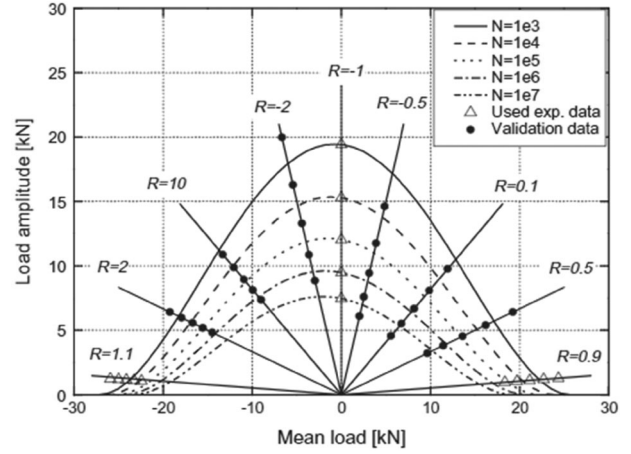


Figure 12. Polynomial constant life diagram for pultruded adhesively bonded joints [18].

adhesively bonded joints [120,128] this exponent ranges between 0.05 and 0.15, being higher (less steep curve) for stiffer and lower (steeper curve) for less stiff material systems. Although laws of this kind are empirical and do not have any explicit physical meaning, they follow the accumulation of the microscopic damage of the examined material which they finally describe [128].

The effect of the mean cyclic stress can be simulated/predicted by using similar methods as those used for composite materials [127,129–132] as shown in Figure 12.

Thick adhesive joints

Most of the works addressed the behaviour of joints with adhesive layer thickness ranging from some microns (mainly film adhesives for aerospace joints) to max 2–3 mm (addressing mainly the behaviour of paste adhesives in civil/structural engineering, marine and automotive applications). Only few works investigating thick adhesive bondlines, representative of real joints in e.g. wind energy or marine applications, can be found in the open literature. Hart-Smit [10] suggested that structural-bonded joints with thin film adhesives have shown a remarkable insensitivity to quite large bond flaws and that prior repair practices have actually been detrimental to the life of bonded structure. According to Hart-Smith, in most instances, it would have been better not to repair the structure, since even at the presence of voids and cracks, structural components can sustain a certain number of operating cycles. For instance, as was shown in [133], a flight box beam subcomponent with an artificial void that undergone four aircraft lives (24,000 effective flight hours) without failure or propagation of the void.

Nevertheless, thicker adhesive joints contain more voids in the adhesive, mainly due to manufacturing defects. Adams and Peppiatt [134] and Park et al. [135] suggested that chances of having porosity and

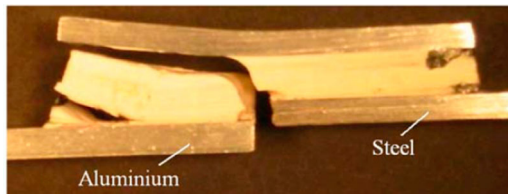


Figure 13. Transverse cracks in thick adhesive joints under quasi-static loading [45].

microcrack in joints with thicker bondline increase and hence there is a greater probability of early failure for joints with a thicker bondline. Adhesive thickness is responsible for the development of bending stresses in lap joints in tension. As the bondline thickness increases, there is an increase in the bending stress since the bending moment has increased. Consequently, the strength of the joint is reduced [136]. However, contradictory results were reported regarding the effect of voids in the adhesive of thick joints in wind turbine rotor blades. In Sayer et al. [137], an I-beam structure was designed, manufactured and tested under static and fatigue loading in an asymmetric three-point bending configuration. The I-beam was used to investigate the influence of different design variables and manufacturing techniques. It was reported that the axial stress component in length direction of the blade, longitudinal, was the dominant, although additional stress components existed; axial stress in the transverse direction about to 10–25% of the longitudinal axial stress and shear stress components up to 10% of the longitudinal axial stress. The authors highlighted the importance of taking into account the multi-axial stress field in the structural design process of the joints. Moreover, they observed that single voids causing surface stress concentrations did not affect the structural fatigue life. In contrast to this, Sharp et al. [138] developed an I-beam aiming to represent the behaviour of the connection of the shear web to spar caps in wind blades, and observed that voids in the adhesive would lead to reduced joint strength with earlier crack initiation. Joints with thick adhesive bondlines show also different damage progression under both quasi-static and fatigue loading when compared to thinner joints.

Experimental results from Jarry and Shenoy [45] have shown that transverse cracks occurred in the

adhesive bond. These cracks are due to the presence of a gap between the bottom adherends of the joints thus presenting an adhesive-free surface. When the joint is loaded, the strap bends because of the eccentricity of the load path, causing the horizontal adhesive free surface to be torn apart as shown in Figure 13.

Transverse cracks were also observed in the adhesive bondline of a generic composite I-beam adhesive joint that was designed and manufactured, mimicking the axial-to-shear stress ratio in the bond line between spar caps and shear web of a MW scale wind turbine blade [46], see Figure 14. In that case, however, the authors attributed the crack formation to the residual strains developing in the adhesive bulk due to the exothermic curing of the polymer adhesives. Similar cracks were observed in a wind turbine rotor blade spar to shear web bond line [46,139]. The heat due to the reaction is dependent on the processing protocol (curing cycle) and the material volume, being obviously more for thicker adhesive bondlines [48].

In state-of-the-art guidelines for wind turbine blades [140], towards the adhesive joint design verification for a life-span of at least twenty years, several damage modes are considered. Among others, it is recommended that the bond line should be designed against cohesive failure. Therefore, besides other stress components, shear and axial stress limits derived from demonstrated tests have to be considered. For thick bondlines this means that the bulk adhesive material properties with the minimum uncertainty should be available. Nevertheless, despite the availability of extensive literature, there is still a considerable lack of knowledge in the understanding of both bulk adhesive and bonded joint behaviour, particularly in the case of large thickness adhesive bonds [141]. For the corresponding fatigue analysis, a stress-life approach is advised based on reliable experimental S–N data. Moreover, it is also stated that the performance of the adhesive joints is strongly dependent on the bondline thickness and therefore, the characteristic strength of the materials has to be reduced by safety factors. The literature review reveals that the thick bond lines are subjected to multi-axial stress fields and, contrary to the adhesively bonded joints with thin bondlines which mainly serve to transfer shear stress, it is crucial to taking this stress field

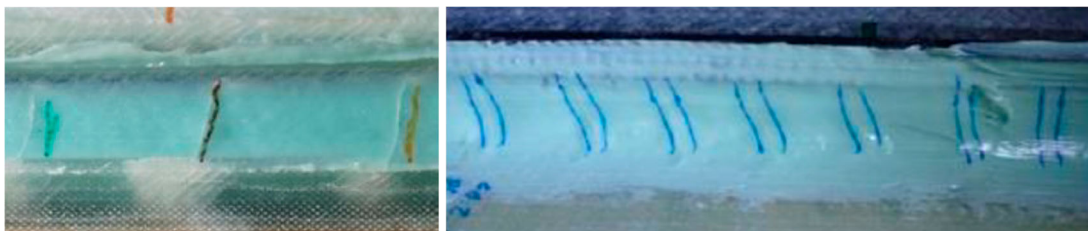


Figure 14. Transverse cracks in the beam adhesive bond line (left) and in a WTRB spar to shear web bondline (right) – not in scale [46].

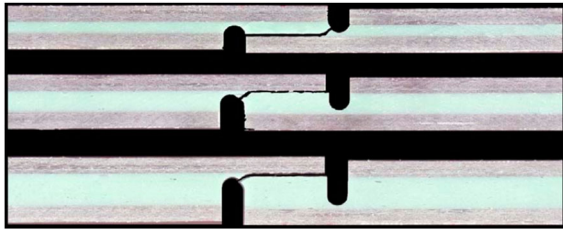


Figure 15. Failed specimens with 3.25, 6.50 and 9.75 mm bondline thickness of an epoxy adhesive [143].

into account in the design process. Thus, for better understanding of the damage process and the failure patterns observed in thick bonded joints, more knowledge is required on the multi-axial behaviour of the bulk adhesive itself [41].

A couple of other publications can be found in the literature investigating the behaviour of thick adhesive joints and confirming the dependence of their performance on the bondline thickness [142–144]. The variability of bondline thickness, that is inevitable in thick adhesive joints, is a major concern because of inclination of thinner bonded area to attract more load and to cause stress concentration in adjacent adherends [142].

In Samborsky et al. [143], it has been shown that thick adhesive joints (notched lap shear joints, see Figure 15) with a relatively brittle and a relatively tough adhesive exhibited different fracture behaviour. The more brittle adhesive failed quickly after crack initiation, whereas crack growth was slow for the tougher adhesive involving stable cracks form both notches. However, similar S–N curves derived for specimen with both adhesive types for tension-tension ($R = 0.1$) and reversed fatigue loading ($R = -1$).

Fatigue behaviour of bulk adhesives

Most of the commonly used test methods on joint configurations are incapable of providing reliable engineering material data because the test geometry induces a complex state of stress in the adhesive layer, thus invalidating the results. Most of these tests can only be used for qualitative measurements, providing a means of checking the effectiveness of different surface preparations and comparing mechanical properties of different adhesive systems (i. e. for ranking adhesive formulations). One approach that can be adopted in order to overcome this problem is to measure the properties of bulk adhesive specimens [139]. Although several problems are associated with casting bulk adhesive specimens, they are relatively straightforward to test and easily accessible for visual inspection, failure observation, and measurements (see, e.g. Figure 16), while the derived properties are directly assigned to the material itself and not to a structural system. Bulk material

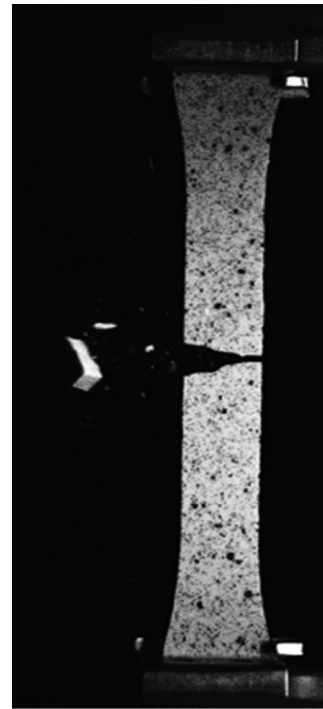


Figure 16. Photo at the moment of failure – speckle pattern for DIC measurements is visible on the specimen surface [66].

properties, and the understanding of the mechanisms involved in their fatigue failure are essential for the design of fatigue resistant adhesively bonded joints.

Fatigue life of bulk resins and adhesives

Fatigue life investigations

Specimen geometrical characteristics and fabrication procedures are recommended in several technical reports and standard procedures such as the [145–151] for both resins (plastics/polymers) and adhesives fatigue life investigations.

Bulk material specimens are cast and machined to the required shape, e.g. dogbone or dumbbell specimens used for tensile fatigue loading (see Figure 17 (a)), the V-notch and the Arcan specimens for shear testing (Figure 17(b and c)) [145].

The fatigue behaviour of bulk polymer materials, resins and adhesives, has been investigated since early, see, e.g. [152–154]. First results showed that the fatigue properties of different material types can vary significantly (see Figure 18) [152].

The fatigue data presented in results Figure 18 indicate a superior behaviour of the, e.g. phenolic and epoxy polymers compared to others. Nevertheless, the results are weighted heavily by the larger number of cycles necessary to initiate a crack in the epoxy resin under the test conditions associated with these experiments (unnotched specimens with the test frequency set at 30 HZ). When crack propagation rates are however compared [155] the relative ranking of these materials with regard to fatigue resistance is reversed with fatigue cracks propagating faster in the

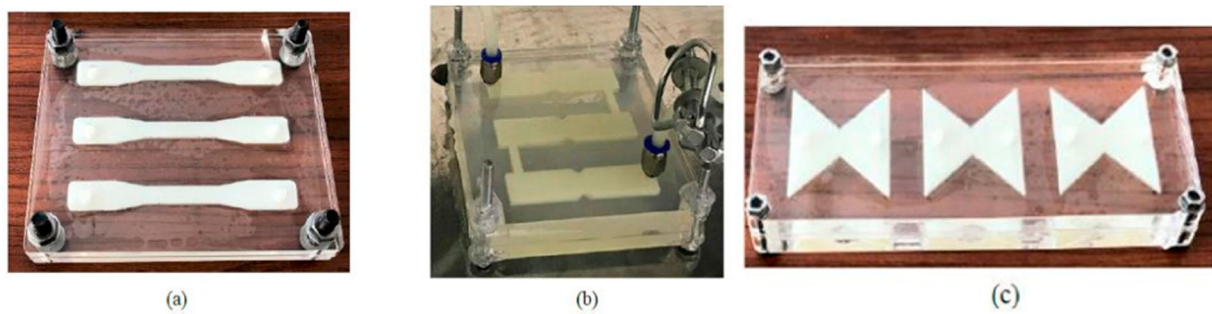


Figure 17. Moulds used in the production of bulk specimens: (a) standard tensile specimen; (b) losipescu test specimen; (c) Arcan test specimen [145].

epoxy resin. Furthermore, the relative ranking for total fatigue life in Figure 18 is strongly influenced by the aforementioned test frequency. At 30 Hz, the amount of hysteretic heating is not the same in all polymers. The epoxy resin exhibits little damping, as was also confirmed in Refs. [23,66] and epoxy specimens do not heat up. Therefore, the results in Figure 18 reflect a mixture of mechanically and thermally induced damage mechanisms. Were the test frequency to be lowered to preclude thermal-type failures, the relative fatigue resistance ranking of the materials shown in Figure 18 would be different [155].

The effect of porosity [66,70,141] on the fatigue life of epoxy adhesives used in wind energy was also investigated. As mentioned above, in contrast to traditional guidelines for adhesive joints, wind turbine blade joints have high thickness. Although particular care is taken in preparing such thick adhesive joints, a high degree of porosity cannot be avoided, mainly due to the high viscosity of the bonding paste. This degree of porosity affects the stress distribution in the adhesive and it is a potential source for the initiation of both quasi-static and fatigue failures [66,141]. Typical failure surfaces of adhesive specimens under tensile fatigue are presented in Figure 19.

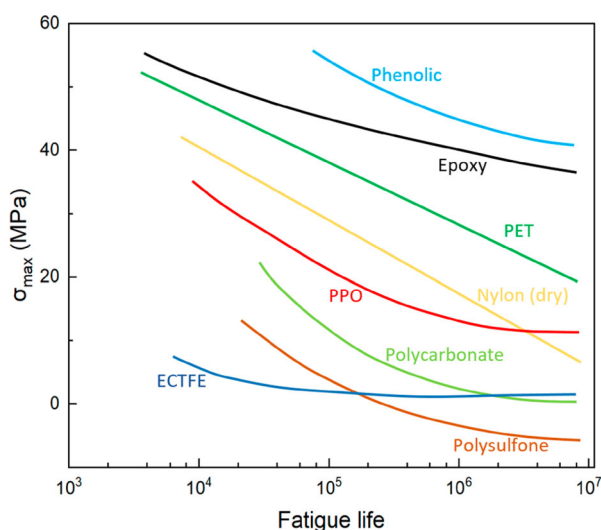


Figure 18. Representative S–N curves for numerous engineering plastics (reproduced from Hagerup [152]).

The specimen cross section dimensions were 4×13 mm [66]. Bigger voids appear in all specimens, probably due to the manual pouring and casting, although their presence does not seem to affect the material strength. It is supposed that the regular ellipsoidal shape of such big voids does not create any stress concentrations in the material volume, and therefore these voids did not seem to affect considerably the fatigue behaviour of the examined epoxy adhesive in Foletti et al. [66].

The authors of Ref. [66] measured the cyclic Poisson ratio of epoxy adhesives by using a digital Image correlation (DIC) system. The results showed that the Poisson ratio is not sensitive to the displacement rate and the cyclic Poisson ratio (shown in Figure 20) is similar to that derived from the quasi-static experiments [66].

Time dependent phenomena – hysteresis area

Fatigue failure in polymer adhesives occurs either as thermal failure, due to softening and melting from hysteretic heating or as mechanical failure from crack initiation and propagation [156,157]. In practice, both thermal and mechanical fatigue occurs [158–160]. All polymers are viscoelastic to some extent [161]; therefore, their long-term behaviour (fatigue life) depends on the applied loading cycles, the loading rate and the time spent at load – a clear combination of creep and fatigue. Fatigue loading profiles usually accommodate a certain amount of creep, depending among others, on the material, the loading pattern, and the environmental conditions. The fatigue failure is usually a result of the combination of creep (time related) and fatigue (cycle related) phenomena.

The time dependent phenomena are more intense when softer materials, e.g. thermoplastics, that commonly used as adhesives, e.g. [162,163], or as resins in their composites, [164–168] are involved. Riddell et al. [169] showed that the fatigue mechanisms of thermoplastic polymers are controlled by internal damping, especially related to the loss compliance. The fatigue behaviour of thermoplastics was different from that of metals and other elastic materials. Constable et al. [170] explained that cracks propagated

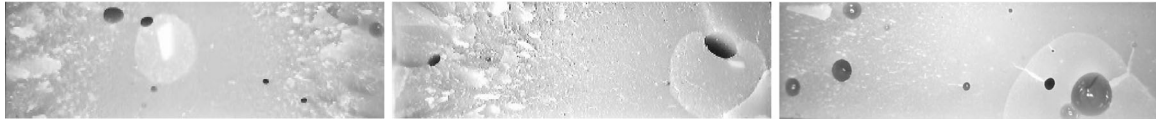


Figure 19. Representative failure surfaces of bulk adhesive specimens [66].

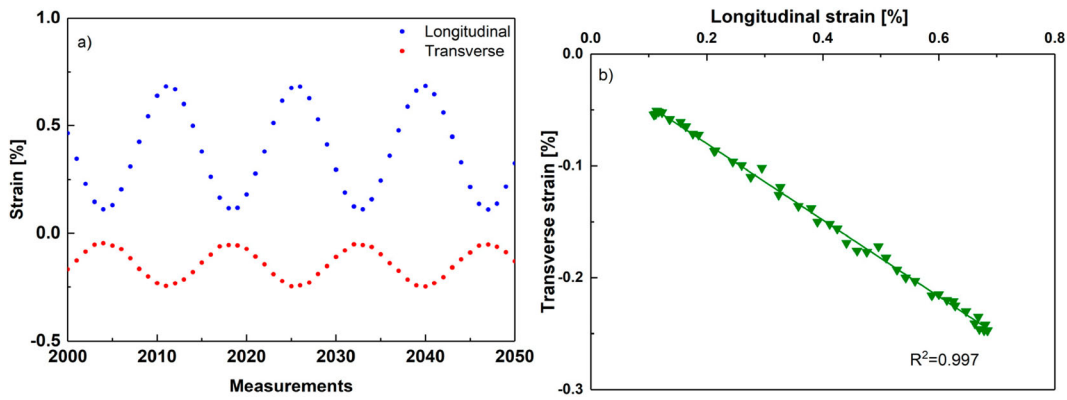


Figure 20. Cyclic Poisson's ratio measurements [66].

incrementally from existing defects in thermoplastics under cyclic loading, providing that this was not preceded by thermal softening at some section. The authors also confirmed the relationship among cyclic thermal softening, loss compliance, specimen geometry and the frequency and magnitude of the cyclic load.

Experimental investigations focusing on the fatigue life of PEEK, a thermoplastic adhesive involved in numerous applications, e.g. [171–175], ABS (Acrylonitrile Butadiene Styrene) or PLA (Polylactic Acid) [167], polyethylene [176], and polypropylene [177] concluding that the fatigue life of all examined thermoplastic materials is very sensitive to the testing temperature, frequency and mean cyclic stress [177]. Same frequency and mean cyclic stress dependence were revealed for certain elastomers used as adhesives [178–181]. Polycarbonate, polyphenylsulfone and polyetherimide unfilled polymers' fatigue behaviour has been investigated in Kanters et al. [182] under different R -ratios. The authors distinguished between a frequency independent plasticity-controlled failure (mainly at high cyclic loads) and a faster, frequency dependent, crack-controlled failure (usually at lower stress levels), see Figure 21.

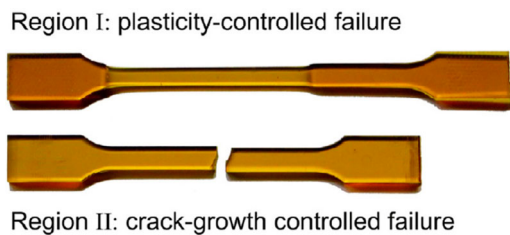


Figure 21. Plasticity-controlled and crack-growth controlled fatigue for polyetherimide, PEI 1010 bulk specimens [182].

In modern industrial applications, epoxies are used either as adhesives in joints or as matrices in FRP composites, and their fatigue-life behaviour has been intensively investigated in the past. As mentioned above, epoxy resins exhibit little damping, and epoxy specimens do not easily heat up with repeated cyclic loads. For materials with a purely elastic behaviour, the elastic energy per loading cycle is equal to the total energy and no dissipation energy is measured. However, for the majority of materials, energy is dissipated at each load cycle, either due to additional structural changes, or due to hysteretic heating. Typical definitions regarding the hysteresis area and relevant measurements are shown in the schematic diagram in Figure 22. Epoxy, although does not present large hysteresis area, shows a significant ratcheting behaviour, especially when loaded under fatigue spectra with significant mean cyclic stress components [23,27,66,183]. Cyclic creep strain is progressively

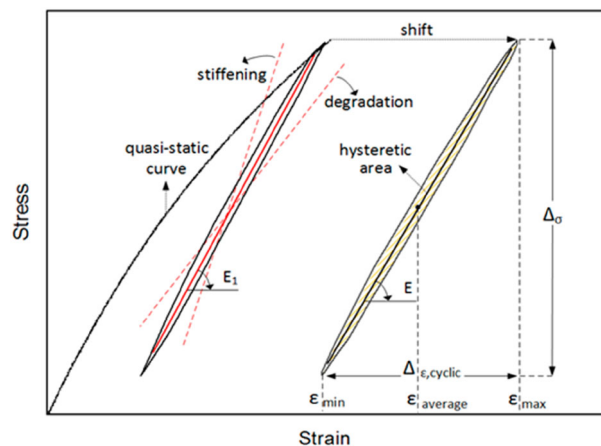


Figure 22. Schematic representation of the stress strain loops [23].

accumulated under stress-controlled cyclic loading. This strain is of a viscoelastic nature, and is completely recovered after load removal [66]. Tao and Xia [27] performed fatigue tests with multiple resting periods (zero loading) during the fatigue processes and observed that most part of the ratcheting strain was already recovered during the resting period, although the recovery process may still continue if a longer resting period was applied. The same observation was reported for an angle-ply composite laminate subjected to similar interrupted fatigue loading patterns in Movahedi-Rad et al. [184]. The amount of the accumulated strain depends on the applied cyclic mean stress [27]; the former is diminished under inversed fatigue loading, $R = \sigma_{\min}/\sigma_{\max} = -1$, while it becomes significant for tension-tension ($0 < R < 1$) and compression-compression ($1 < R < \infty$) fatigue loadings [185]. The cyclic fatigue behaviour of epoxy resins under uniaxial and biaxial stress states, as well as the mean stress/strain effect on the cyclic response has been investigated in Refs. [25–27, 186–188].

The mean stress effect on epoxy resin was also investigated in Brunbauer and Pinter [189] showing a steeper S–N curve for specimens loaded under a reversed fatigue spectrum when compared to the slope of the S–N curves at $R = 0.1$. Only few works exist on the investigation of the mean stress effect on epoxy, as most of the investigations focus on other effects and use only one stress ratio, usually either under tensile-tensile fatigue at $R = 0.1$, e.g. [23, 28, 66, 119, 190–194] or under reversed loading at $R = -1$ as e.g. in Refs. [25–27, 186–188]. Recently an investigation on 3D printed PLA specimens' fatigue was published with results from both $R = 0$ and -1 [195, 196] confirming the dependence of the S–N curve slope on the R -ratio with reversed loading resulting in steeper S–N curves.

Fatigue of modified adhesive systems

Several researchers suggested different approaches for modification of epoxy adhesives/resins aiming to improve their fracture toughness and eventually their durability performance and fatigue resistance [28, 119, 190, 191, 197]. Bortz et al. [190] showed that adding 0.5 and 1.0 wt.% of one-dimensional (1-D) carbon nanofibers (CNF) into the bulk of an epoxy resin system improved the fatigue performance significantly, and increased the fatigue life by a factor as high as 3, especially at lower stress levels. An increase in the fatigue life was also observed in Kang et al. [198] for adhesively bonded composite to aluminium joints. Addition of 2 wt.% carbon nanotubes increased the endurance limit of the examined joints by ca. 13%, although reduced the static strength by ca. 30%. Unfortunately, there is not a clear explanation for this controversial result in Kang et al. [198] with the authors attributing this phenomenon to the delay of the crack initiation and propagation during fatigue

life due to the dispersion of the nanotubes into the adhesive layer. Manjunatha et al. [191, 197] proved that the addition of ca. 10 wt.% of rubber nanoparticles [191] and silica nanoparticles [197] improved the fatigue life of an epoxy polymer by three to four times under any stress level. S–N curves for the modified resin were shifted towards longer lifetime having similar slope as that of the neat resin. In both cases, the life improvement was attributed to the plastic deformation and void growth around fatigue cracks caused by the presence of the inclusions. Contrary to this, Wang et al. [28] have demonstrated that the addition of hard particles was shown to increase but soft particles decrease the fatigue life of nanocomposites compared to unmodified epoxy. Silica particles increase but rubber fillers decrease the fatigue life at the same stress amplitude silica fillers, while a combination of both silica and rubber does not alter the fatigue performance of the neat resin.

Beber et al. [119, 193, 194] investigated the fatigue performance of different types of toughened structural epoxy adhesive specimens with and without notches. The results, showed a significant sensitivity of the investigated adhesives to notches, different for each adhesive type, Figure 23. The more brittle adhesive (A1) was sensitive to notches, in both low and high cycle fatigue regions. More ductile adhesives, such as the A3, were able to deform plastically around the notches relieving the surrounding stress field. Therefore, in the low cycle fatigue range, under high cyclic stresses, un-notched and notched specimens of the A3 adhesive showed similar fatigue strength. However, at high cycle fatigue, the notch effect was significant and reduction of up to 27% of the fatigue life was observed. Based on FEA, this may be associated with stresses above the yield stress that might have caused plastic reactions in both notched and un-notched samples [193]. Nevertheless, S–N curves of notched specimens were in all cases steeper than those from

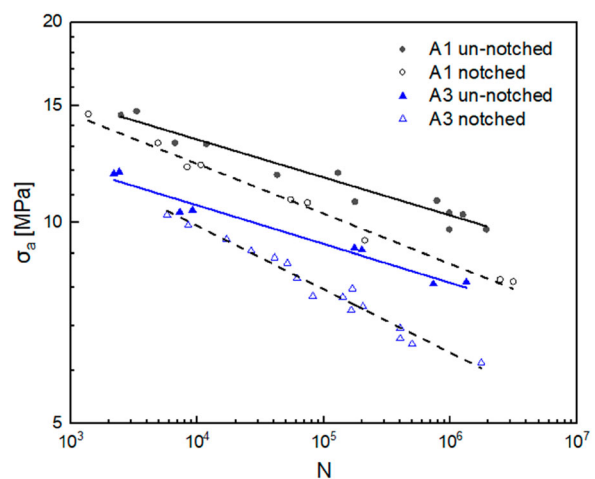


Figure 23. S–N curves for un-notched and notched bulk adhesive samples [193, 194].

Table 2. Typical S–N curve slope values for epoxy adhesives.

Material type	R-ratio	Slope 1/k Equation (1)	Reference
Neat epoxy resin EPIKOTE 135/1366 (un aged)	0.1	0.068	[192]
Monomer (70–100% 4,4'-Isopropylidenediphenol-Epichlorohydrin Copolymer and 0–30% 1,6-HexanediolDiglycidyl Ether) and a hardener (25–50% Alkyletheramine, 20–25% Isophoronediamine and up to 20% Aminoethylpiperazine)			
Neat epoxy resin EPIKOTE 135/1366 (saturated)	0.1	0.110	[192]
Epoxy adhesive Sikadur 330 (dry)	0.1	0.079	[23]
Thixotropic two-component epoxy resin			
Epoxy adhesive Sikadur 330 (wet)	0.1	0.117	[23]
Epoxy adhesive Sikadur 330 (dried)	0.1	0.080	[23]
Resoltech 1800/1805 (un aged)	0.1	0.078	[190]
Resoltech 1800/1805 (un aged)	–1	0.139	[190]
Resoltech 1800/1805, low viscosity epoxy resin (neat)	0.1	0.08*	[190]
Resoltech 1800/1805, low viscosity epoxy resin (0.5 wt.% CNF)	0.1	0.106*	[190]
Resoltech 1800/1805, low viscosity epoxy resin (1 wt.% CNF)	0.1	0.112*	[190]
Toughened epoxy adhesive for automotive (room temperature)	0.1	0.069	[122]
Toughened one component epoxy adhesive for automotive (–10°C)	0.1	0.080	[122]
Toughened one component epoxy adhesive for automotive (50°C)	0.1	0.060	[122]
Toughened one component epoxy adhesive for automotive (un-notched)	0.1	0.063	[193]
Toughened one component epoxy adhesive for automotive (2 mm notch diameter)	0.1	0.102	[193]
Epoxy (Araldite F form Sigma-Aldrich)	–1	0.102	[28]
Araldite-F (diglycidyl ether of bisphenol A, DGEBA) and Piperidine, 40 wt-% nano-silica/bisphenol A, and 25 wt-% nano-rubber/bisphenol A			

*Values from Bortz et al. [190] – not consistent with the corresponding values given in Bortz et al. [190].

un-notched ones with the results suggesting a relationship between the S–N curve slope and the notch size.

Fatigue sensitivity

All reviewed studies agree that the fatigue data can be fitted by a Basquin like equation, such as that shown in Equation (1). The sensitivity to fatigue loading can be estimated by evaluating the slope of the S–N curve [12], a measure of the fatigue strength decrease rate with cycles. All examined epoxy resins exhibit a consistent behaviour with S–N curve slope around 0.075 as shown in Table 2, similar to the slope of S–N curves for composite materials as described in Vassilopoulos [12]. Therefore, it is expected, although works proving this do not exist, that similar CLD models to those used for composite laminates, see, e.g. [126–129], and would be appropriate for the modelling of the mean cyclic stress effect on the fatigue life of adhesive materials. The engineering applications with the adhesive joints are usually operating in humid and hot/cold environments. The environmental effects on the fatigue behaviour of epoxy adhesives have been investigated in the literature, e.g. [23,119,192], although limited works exist. In those works, a consistent S–N curve with a slope of ~ 0.07 – 0.08 was derived for the unaged epoxy adhesive.

Nevertheless, either due to water ingress that causes plasticisation [23,192], or due to temperature that softens the material [119], the slope of the S–N curve corresponding to aged specimens was significantly increased reaching approximately 0.10 – 0.11 as shown in Figure 24 and Table 2. The wet specimens showed significantly reduced strength and stiffness values, as well as much higher strain to failure, due to plasticisation caused by the water uptake [54]. However, the plasticisation effect seems to be

reversible, as can be seen in Figure 23 and the S–N curve slopes in Table 2 for the ‘dried’ and the ‘dry’ specimens [23,192]. Interchange bonds are reformed after water removal, and as described in Zhou and Lucas [199] additional secondary crosslinking resulting from water-resin interaction can occur after long immersion periods, as was the case for the dried specimens. The S–N curves for reversed loading are higher, reaching values ~ 0.1 – 0.14 .

The dependence on the fatigue life to the cyclic stress level is similar to that observed for adhesively bonded joints with epoxy adhesives as presented in Table 3 showing typical slope values for metal and composite adhesive joints bonded with epoxy adhesives. Nevertheless, in contrast to bulk adhesives testing, the cyclic fatigue performance of joints is highly sensitive to the bondline thickness and the adherend material, as well as to geometrical parameters, e.g. adhesive filler etc. As mentioned in

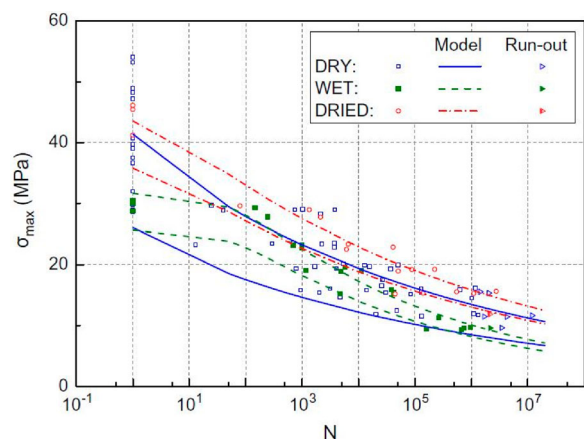


Figure 24. Fatigue behaviour and modelling of all specimen [23].

Table 3. Typical S–N curve slope values for bonded joints with epoxy adhesives ($R = 0.1$, $f = 5$ Hz) [200].

Joint type	Slope $1/k$ Equation (1)
Scarf (steel adherends with 15° angle)	0.063
Thick adhered shear test (steel adherends)	0.063
Double-lap (titanium adherends)	0.075
Double strap (aluminium adherends)	0.088
Single-lap (mild steel adherends)	0.093
Double-lap (woven fabric)	0.097
Scarf (steel adherends with 60° angle)	0.098
T-joint (aluminium adherends)	0.104
T-peel (mild steel adherends)	0.130

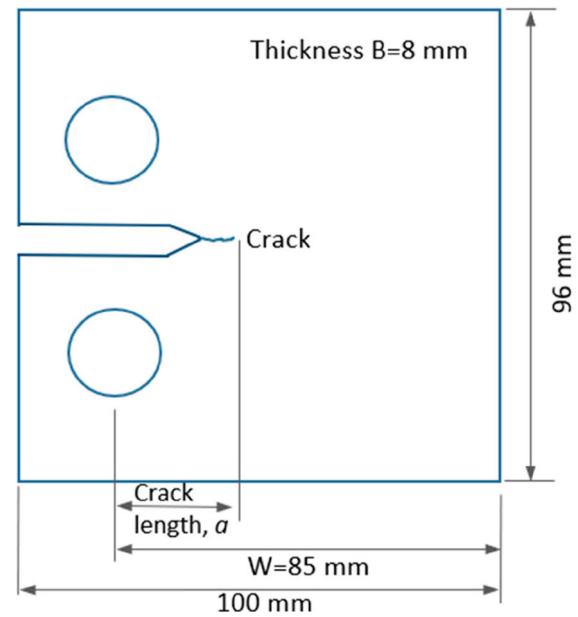
Broughton and Dean [200], a scarf joint with an angle of 15° with respect to the horizontal where failure is dominated by shear stresses has a far better fatigue performance than tests where peel or hydrostatic tensile stresses are the major cause of failure, e.g. T-peel joints and 60° scarf joints. Unfortunately, not a lot of information is given in Broughton and Dean [200] regarding the sources of the listed fatigue data shown in Table 3.

A bondline thickness of the order of 0.2–0.3 mm tends to have far superior fatigue resistance than that of thicker bondlines (> 0.5 mm); probably because of lower porosity within the adhesive. An increase in temperature or moisture ingress will compromise the fatigue performance of polymers and adhesive joints (k can be expected to increase).

Fracture behaviour of bulk resins and adhesives under cyclic loading

Fracture investigations

The use of structural adhesive in the construction industry was limited until the late 1980s [201]. Most of the research in this field led by works in the automotive or airplane/aerospace industry, while only few applications of primary adhesive bonded, e.g. external bonding of steel plates to concrete beams, or bonded cover plates on the tension flanges of steel girders [201]. Toughened epoxies stand out as the most promising for structural uses, provided that appropriate measures are taken to prevent moisture uptake (e.g. painting exposed edges of the bonds). Investigation of the fatigue/fracture behaviour of structural adhesives has been extensively performed through joint testing simulating different fracture modes, but also different applications as was discussed in the previous section. Only limited works can be found in the literature investigating the fatigue/fracture behaviour of structural adhesives by tests on the bulk adhesive itself. Works on quasi-static fracture behaviour of structural adhesives, e.g. [202] showed no significant difference between the bulk and the adhesive in joint fracture behaviour, probably because in Bascom et al. [202] the so-called adhesive testing was a modified version of the tapered-DCB configuration used for Mode I joint testing. Nevertheless,

**Figure 25.** Adhesive compact tension specimen according to ASTM E647 [203].

similar works on fatigue/fracture do not exist (to the authors knowledge). The (Mode I) (fatigue) fracture behaviour of structural adhesives can be studied by using the compact tension specimens suggested by ASTM E647 [203] and shown in Figure 25.

The Izod impact specimen has been used to perform impact fatigue tests on the FM73 adhesive in order to study the evolution of damage and to characterise this via measurable parameters, such as the maximum force and contact time [204]. Dumbbell specimens were also employed see, e.g. [205] presenting results from low cycle fatigue tests on bulk adhesive dumbbell specimens aiming at the development of a damage mechanics model to predict damage curves. Other specimen types were also used, e.g. the edge-notch specimen in three- or four-point bending for fracture analysis of adhesives, but mainly for quasi-static loading, see, e.g. [206,207] (Figure 26).

Fatigue crack growth curve

The compact tension specimen was used in Luckram and Vardy [201] to investigate the fatigue/fracture behaviour of a toughened epoxy adhesive. The results shown that the rate of Stage II (stable crack propagation) crack growth satisfies the 'Paris law' introduced in the 1960s after the examination of several aluminium alloys and the observation that plots of crack growth rate against the range of stress intensity factor (also called fatigue crack growth – FCG – curves) at the second stage gave straight lines on log-log scales [208–210]. This observation implies Equation (2):

$$\frac{da}{dN} = C(\Delta G)^m \quad (2)$$

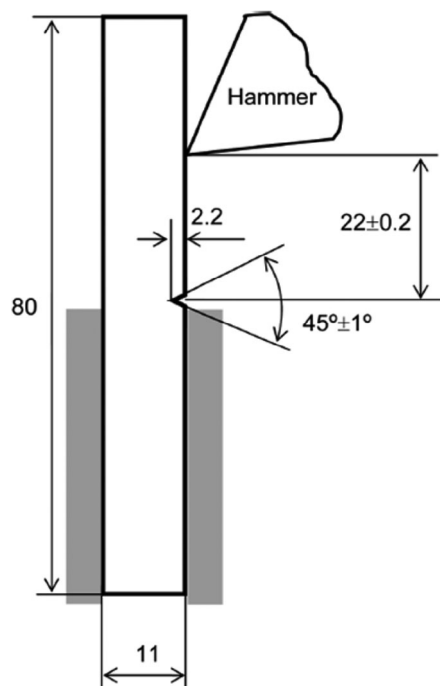


Figure 26. Dimensions of the Izod impact specimen [204].

With da/dN the crack growth rate, ΔG the range of the fracture energy and C , m model parameters that need to be estimated by fitting the experimental data. The faster the rate of crack propagation, the lower the fatigue resistance. Therefore, smaller values of the 'Paris law' exponent signify higher material resistance to fatigue crack growth [211].

The 'Paris law' has been used successfully in Luckram and Vardy [201] and other works to describe crack growth in various configurations, including bulk polymer adhesives and systems (joints) that satisfy the assumptions of linear elastic fracture mechanics (LEFM). Linear elastic fracture mechanics is the simplest existing approach for the analysis of crack growth in adhesive joints, as it relies on a single parameter, the fracture toughness, to describe the fracture process. LEFM is valid as long as small-scale fracture process zone is assumed ahead of the crack tip in relation to the other dimensions of the specimen [209].

Fatigue/fracture tests performed on compact tension specimens of PEEK [212–214], toughened epoxy polymer adhesives [215,216], epoxy nanocomposites

[211] and thermoplastic polymers [217], among others, also exhibited fatigue crack growth curves with linear second stage, that can be fitted by a 'Paris law' equation. Some of the works identified a fatigue threshold at the FCG curve, a threshold value of the maximum strain-energy release rate in a fatigue cycle, G_{th} , (below which no significant crack growth occurs), e.g. [216]. The fatigue threshold depends on the material and geometrical and loading parameters, as well as the 'Paris law' exponent, that as mentioned above, is an indication of the fatigue/fracture resistance of each material. On average, the FCG curve slope was 2.28 with a difference between the extreme values of 37% for different temperatures, thicknesses and load levels for PEEK [213], while for the same material, but by testing a different specimen configuration (the circumferential U-shaped grooved specimen in Sobieraj et al. [218]) the slope was estimated to around 4.5. Sobieraj et al. [218] also revealed that more than 70% of the lifetime was spent for the crack initiation and argued that there is the potential for gross failure of the PEEK implant devices of their study without any substantial period of detectable difference in structural behaviour. The neat epoxy resin investigated in Sobieraj et al. [218] showed an exponent of 2.89, while this value fluctuated between 2.89 and 5.19 for different contents of rubber inclusions between 2% and 30% with a peak of 7.37 for 10%. Addition of 0.1–0.5 wt-% of multi-walled carbon nanotubes with a typical length of 140 μm and diameter of 120 nm led to a significant improvement of the fatigue performance [219]. This was attributed to the contribution of the toughening mechanisms of nanotube debonding, and nanotube pull-out, also supported by other investigations [220] and presented in Figure 27.

Nevertheless, it is clear in the results presented in Hsieh et al. [219] that the addition of the carbon nanotubes increased the threshold of the FCG curve, but not affected the rate of the crack propagation in the 'Paris Law' region. Similar increases in the threshold strain energy release-rate have previously been reported by Blackman et al. [221] using silica nanoparticles. Hwang et al. [222], pointed out that rubber-toughening does improve both impact and fatigue crack growth resistance, but slightly lowers Young's

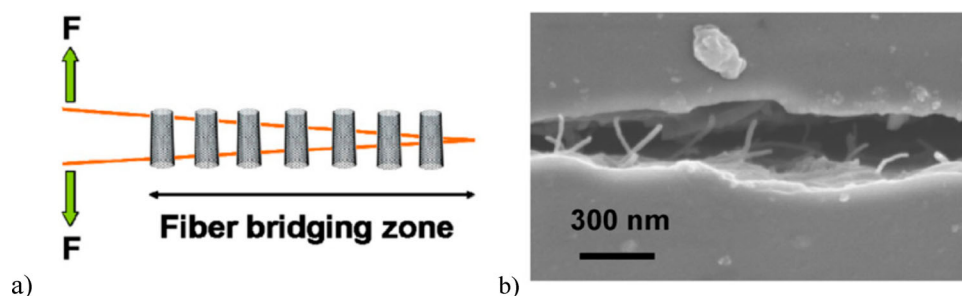


Figure 27. Mechanism for fatigue crack growth suppression in nanotube epoxy composites. (a) Schematic of fiber bridging zone. (b) Fractography analysis confirms MWNTs bridging the crack and forming a 'bridging zone' in the wake of the crack tip [220].

modulus. Similarly, Naito and Fujii [109] found the addition of rubber can increase the fracture dimension of the fracture surface of epoxy adhesive under fatigue loading. Some studies [223] also compared the fatigue resistance of epoxy/glass interface using neat and rubber-toughened epoxies. The results showed that smaller size rubber particles give the greatest improvement, over 75% higher resistance than the non-toughened epoxy through the additional energy absorbing mechanisms of cavitation and shear yielding.

Similar 'Paris law' slopes were estimated for epoxy adhesives when tested in thick adhesive joints as presented in Samborsky et al. [143] where crack growth exponents were between 4.0 and 4.6 for all the examined cases, and similar to those reported in the literature for cracked lap specimens with thin epoxy adhesive layers [224], or in Chen et al. [225] for an automotive adhesive (exponents for Mode I, II were $m_I = 4.14$, $m_{II} = 4.44$), or in Monteiro et al. [226] for an epoxy adhesive exhibiting exponent values between 4.5 and 7 for different R-ratios. Bureau et al. [227] found values between 2.5 and 3.9 from compact tensile specimen fatigue tests of injection-moulded polystyrene/polyethylene blends. The fatigue crack propagation (FCP) results showed that the fatigue crack growth rates were significantly reduced at low and moderate range of stress intensity factor (ΔK).

Brown et al. [228] studied the fatigue crack propagation in microcapsule-toughened epoxy adhesives (although in joints) and the results showed a significant reduction in fatigue crack growth rate and corresponding increase in fatigue life as presented in Figure 28. This agrees with results presented in Refs. [229,230] suggesting that the slope of the power-law regime in DGEBA-based type epoxies modified by rubber and/or silicone rubber dispersions is a function of the modifier content. Same exponent values (around 10) were estimated for the neat epoxy resin in Neuser and Michaud [231]. However, high variability existed for

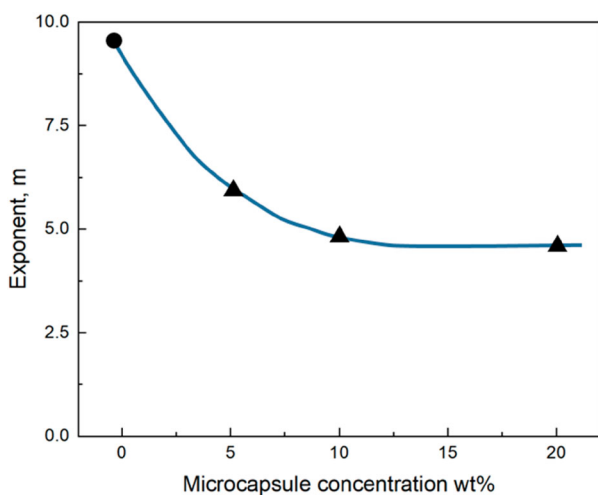


Figure 28. Influence of microcapsule concentration on 'Paris law' exponent (reproduced from [228]).

samples tested less than 2 weeks after casting, and when the material tested after two weeks, the slope was decreased having an exponent of ca. 5. The Paris's law exponent decreased with time, varying from 10.5 to 4.6 but stabilising towards about 5.0 after 2 weeks. This stabilisation over time was attributed to minor water absorption (up to 0.3 % after 2 weeks), which has a plasticising effect on the epoxy matrix.

Not to forget however that the crack growth of epoxy (and of other types of) adhesives in joints is significantly affected by the adherends, the bondline thickness, the surface pretreatment and a number of other parameters [109]. Such effect can be avoided by estimating the fatigue/fracture performance of adhesives by tests on the bulk adhesive itself.

Summary and conclusions – open scientific questions

Thin adhesive bondlines, of the order of μm to a couple of mm from film or paste adhesives, were used in the past to transfer shear stresses between structural members. Nevertheless, the developments in wind-turbine industry and the growing number of primary adhesive joints in marine, automotive and bridge engineering, called for thick adhesive bondlines reaching thicknesses of 20–30 mm in some cases. Most of these structural applications are subjected to a significant number of fatigue cycles (usually of irregular loading spectra) during their lifetime, see Figure 1. The following conclusions were drawn from the literature review shown in this paper.

- Adhesive tests can be divided into those methods that provide mechanical property data for the adhesive, which aids the selection of adhesives, and those methods which can be used to determine the quality of adhesively bonded structures, and thus aid the design process of adhesive joints. Most of the commonly used test methods for adhesives are incapable of providing reliable material engineering data because the test geometry induces a complex state of stress in the adhesive layer, thus invalidating the results. Two approaches have been adopted in order to overcome this problem. The first and direct approach is to measure the properties of bulk adhesive specimens. Bulk adhesive specimens are cast and machined to the required shape (e.g. dumbbell tensile specimens). The second approach for determining engineering properties of adhesives is to use specially designed joint geometries with a thin bond line, often referred to as in-situ testing [139]. When bondlines become thicker, however, additional problems arise, since more complex stress states develop in the adhesive bulk and its behaviour becomes more sensitive to the existence of more and bigger

voids compared to those occur in thin adhesive bondlines [69,70,134,135].

- While experience has shown this to be the case for the majority of well-made metallic joints, composite adherends can often fail by delamination before the adhesive fails [232]. It is not yet clear in the literature whether adhesive fatigue properties measured from joint testing are the same as when bulk specimens are examined. It can be argued that both experimental choices allow the derivation of valuable information, and one should select the first or the second approach depending on the required output data. According to Adams and Wake [7] useful data on the elastic properties of structural adhesives can be provided by both specimen configurations if purely elastic behaviour is considered, while different values are derived when plasticity appears.
- The review presented in this paper highlighted the lack of investigations regarding the (multi-axial) fatigue performance of structural adhesives, either in thick joint configurations or as a bulk material. Only few works could be found in the literature investigating the fatigue performance of bulk adhesives, while works investigating the multi-axial fatigue and moreover the variable amplitude fatigue performance of bulk structural adhesives are non-existent. Experimental results on fatigue of bulk adhesives under compression do not exist (except a limited number of studies at reversed loading $R = -1$, e.g. [70] for epoxy adhesives and [28,190] for modified epoxy resins). Investigations were focused on the effect of frequency, the effects of additives etc., but the effect of the stress ratio on the fatigue life of bulk adhesives has not been investigated. There are several works on the mean load effect on the fatigue life of adhesively bonded joints, e.g. [17,18] that investigated double lap pultruded joints used in civil engineering under several R -ratios for tension-tension (T-T) and compression-compression (C-C) loading, or [125] that presented S-N curves for R -ratios 0.1, 0.5 and 0.75 for single lap joints steel joints. Experimental results from both programmes confirmed the trend observed also in composite laminates fatigue; the S-N curve slope is higher for lower stress ratios [12], becoming minimum when R approaches towards 1, i.e. for cyclic creep. Nevertheless, no model has been established yet to simulate this behaviour neither for adhesives/adhesive joints nor for composites. Both works [17,125] presented the data in a σ_m - σ_a plane as well and in Sarfaraz et al. [18] a new constant life diagram model has been introduced. Although it can be assumed that adhesives have similar tensile and compressive fatigue behaviour (as isotropic materials) it is not obvious how creep components in a fatigue spectrum

(e.g. at R ratios approaching 1) can affect their fatigue performance and what happens at high cyclic stress levels where the adhesive might become nonlinear. To the authors' knowledge, no work investigating the fatigue behaviour of bulk structural adhesive under block or variable amplitude loading exists, while only few can be found in the literature for adhesively bonded joints [55,233–235].

- The R -ratio effect on the FCG curves, i.e. on the fatigue/fracture behaviour of adhesively bonded joints on the other hand, has been extensively investigated, e.g. [99–106]. Controversial discussions regarding the appropriate parameter to be used in the abscissa of the FCG plot can be found in the literature and no consensus between different research groups has been reached yet.
- Probably this lack of data has its origin in the pre-occupation with fatigue studies based on joint configurations with thin bondlines as they are more directly related to applications. Joint testing cannot be ignored, as there is no other suitable means of detecting adhesion properties. Nevertheless, the introduction of thick adhesive joints and the need for reliable material data that can be used in modelling calls for additional experimental efforts at the material level. The bondline thickness determines the extent to which the bulk material behaviour is important. Yet, this is not quantified. Bulk structural adhesives' fatigue testing under different thermomechanical loading conditions is necessary to gain fundamental knowledge.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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