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Static and cyclic strength properties of brittle adhesives with porosity

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Abstract

Adhesive joints play an important role in structural reliability and durability of assembled load carrying structures. In the application of wind turbine blades the wing box is built up of shear webs and shear caps which are joined to each other with brittle adhesives. With blade dimensions in the order of 40 to 50 m and with current manufacturing tolerances and assembly procedures, adhesive joint thickness may be up to 10 mm, with a high probability on the presence of voids and cavities. As the blade is subjected to simultaneous bending, torsion and shear force, the stress state in the adhesive layers is multi-axial and stress components are non-proportional. The machine has an economical life of 20 years and fatigue may be a critical phenomenon.
This research focuses on a bottom-up adhesive properties characterization and its validation in composite joints. It starts from the characterization of bulk adhesive going through bonded joint specimens and subcomponents. This paper focusses on the levels of the adhesive material itself and of the joint. After an extensive experimental campaign with particular attention to porosity in the adhesive a probabilistic approach is used to identify the most appropriate failure criterion. The strength prediction method considers a statistical size effect in the strength of the material by considering not only the magnitude of the stress distributions, but also the volume over which they act. This approach is subsequently used for the numerical prediction of the strength of joints in simple joints and in spar-cap-shear web subcomponent. The predicted resistance of joints are in agreement with experimental joint tests.

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Keywords: brittle adhesive; wind turbine blade; experimental campaign; probabilistic approach.

1. Introduction

Adhesive bonding and other load transfer details have become subjects of major magnitude as wind blade size has increased. Commonly, wind turbine blades use bonding paste to glue the different parts between them, also the connection between the shear webs to the spar cap. Therefore, this adhesively bonded joint is a fundamental part of the structural integrity of the blade. Typical blade joints use paste adhesives several millimeters thick of varying geometry. 10 mm thick bonded joints are common in wind turbine blades. Consequently, this increase in adhesive thickness leads to an increasing probability of the presence of voids and cavities (Galappaththi et al. (2013); Wetzel (2009); Griffin and Malkin (2011)).

They can be expected to experience significant static and fatigue loads under various environmental conditions over their service life. Apart from static loading conditions and multi-axial loads, cyclic fatigue and time dependent creep/stress relaxation are major loading issues. Furthermore, the variability of joint strength can be greater than that of typical laminates due to a higher sensitivity to flaws such as porosity in the adhesive, poor mixing, unbonded areas or poor dimensional control. Extreme strength issues may occur when the adhesive does not fill the bond gap, and large unbonded or partially bonded areas. This effect is usually not included in testing procedures.

This research focuses on a bottom-up adhesive properties characterization and its validation in composite joints. It starts from the characterization of bulk adhesive going through bonded joint specimens and subcomponents. This paper focusses on the levels of the adhesive material itself and of the joint. Even with carefully controlled preparation procedures for the adhesive, significant degrees of porosity in the adhesive and large scatter in mechanical properties were identified during the first stages of testing campaign which required taking into account the stochastic nature of the materials properties during characterization.

2. Experimental analysis

The test campaign is performed using a bi-component adhesive from Momentive/Hexion. This is an epoxy based polymer which includes a glass-filler that makes it suitable for applying on vertical surfaces. The resin is Epikote BRP 135 G 3 and the hardener is Epikure BPH 137 G. This adhesive is approved by Germanischer Lloyd to be used in wind turbine blades. It has to take into account that this bonding paste has a high degree of viscosity which affects coupon manufacturing and subsequent properties calculation and analysis.

In a first set of experiments, monotonic and cyclic tests are conducted on coupons of "pure" adhesive in uni-axial tension, in pure shear and in bi-axial states of stress (Sections 2.1 and 2.2). Samples have the shape of a dog-bone or a hollow cylinder (Fig. 1 (a), (b)). The second stage set of experiments uses test specimens with a glued glass fiber reinforced composite component (Sections 2.4 and 2.5). A special test coupon is developed to create a uniform stress state which combines the relevant stress components in any desired ratio: a hollow cylinder with a window which is filled with adhesive (Fig. 1 (c)).

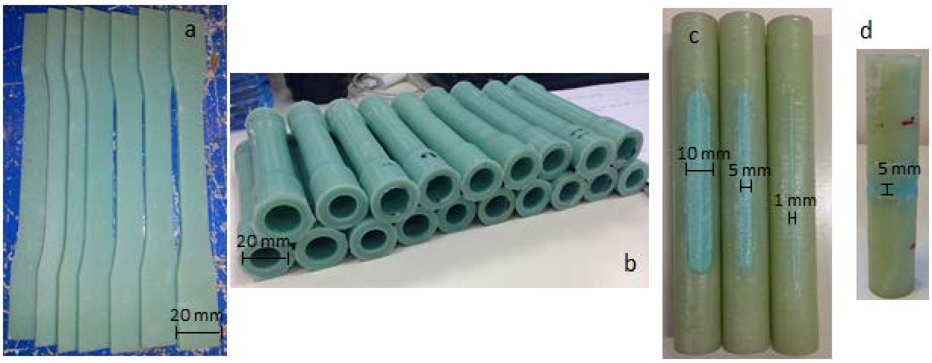

Fig. 1. (a) dog-bone shape coupons; (b) hollow cylinder; (c) composite coupon; (d) butt-joint.

2.1. Quasi-static tests on bulk adhesive coupons: uni-axial tension, torsion and combined tension and torsion

A tensile machine and a Digital Image Correlation (DIC) device are employed to perform quasi-static tests in uni-axial tension (Fig. 2 (a)). A total number of 25 dog-bone shape coupons are tested following the ASTM D 638. This test leads to the determination of Young's modulus, tensile strength and maximum strain.

A tensile and torsion machine is used to perform quasi-static torsion tests (Fig. 2 (b)). The angle of rotation of one extreme end with respect to the other is gradually increased at a velocity of 1°/min and torque is measured. As there is no standard, a sufficiently low velocity is taken as reference for this type of test. A total number of 9 tubular adhesive coupons are tested. The shear properties (shear modulus, shear strength and shear ultimate strain) of the bonding paste are estimated using classical rules of stress analysis in axisymmetric rods.

For the quasi-static tests in combined tension and torsion the previous same machine is employed (Fig. 2 (b)). First, a desired level of tensile load is set and torque is gradually increased until the coupon breaks. The dots shown in Fig. 6 summarize the data obtained from all the described tests: the dots in the *y* axis represent the failure points obtained from uni-axial tension tests, the dots in the *x* axis refer to pure torsion test results and the dots in between correspond to the failure points obtained at different combined tension and torsion load rates.

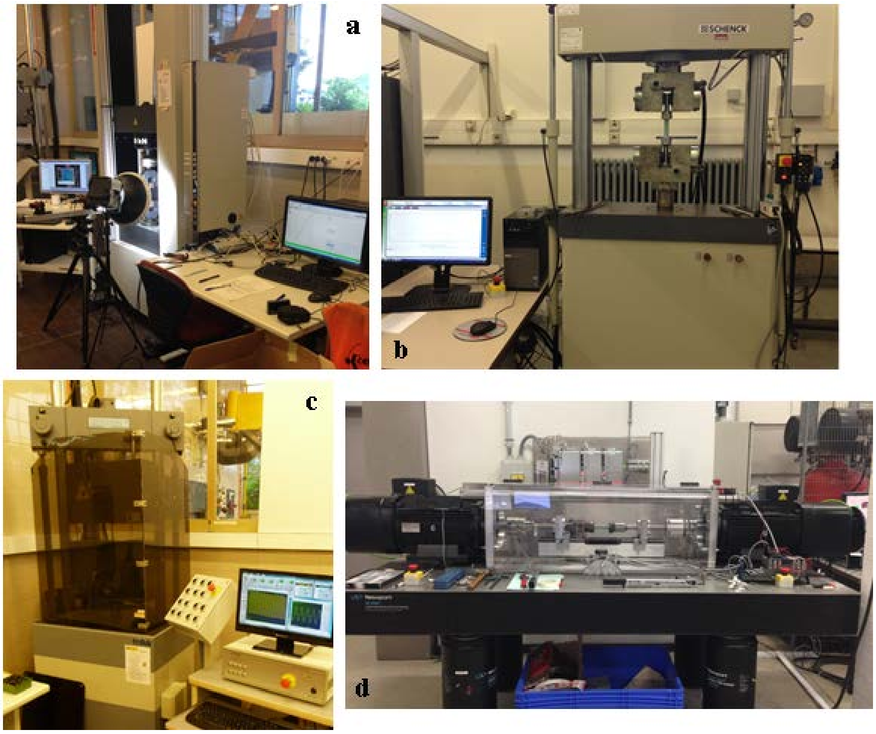

Fig. 2. (a) tensile machine with DIC for quasi-static uni-axial tension test; (b) tensile and torsion machine for quasi-static torsion and combined tension and torsion tests; (c) tensile machine for uni-axial tension fatigue test; (d) accelerated multi-axial fatigue test machine developed in the PhD of Van Hooreweder (2013) for torsion fatigue tests.

2.2. Fatigue tests on bulk adhesive coupons: uni-axial tension and torsion

An experimental campaign is conducted to obtain tensile fatigue properties from bulk adhesive (Fig. 2 (c)). A total number of 12 samples are tested with R = 0.1 ($R=\sigma_{min}/\sigma_{max}$). The frequency of loading is 3 Hz. As illustrated in Fig. 3 (a), the adhesive coupons are tested at different load levels. Two tests at low tensile load values are interrupted early because they ran out without failure within a predefined time frame. A run-out is considered when the number of cycles exceeds 4 million.

On the other hand, the torsion fatigue tests are performed in the accelerated multi-axial fatigue test machine developed in the PhD of Van Hooreweder (2013) (Fig. 2 (d)). This machine is capable of applying torque and bending moments both independently and simultaneously. In this test campaign however, only torsion is applied to the adhesive coupons. A total number of 10 samples are tested at various shear strength percentages. The tests are performed at 7 Hz and with R = -1. The results of the different tests are shown in Fig. 3 (b).

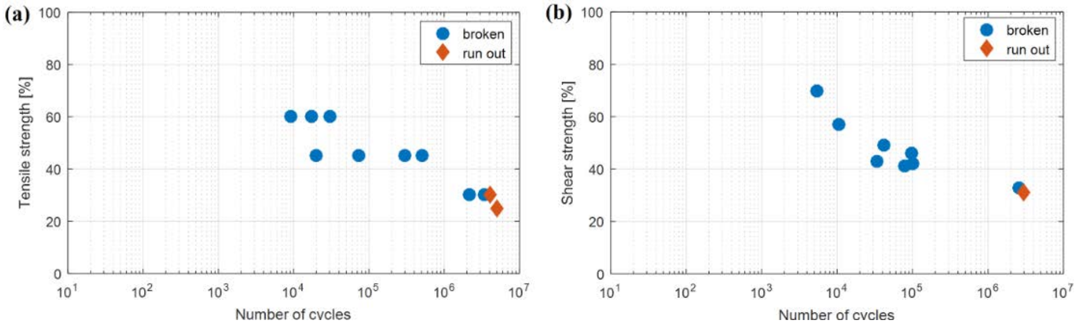

Fig. 3. Fatigue results (a) uni-axial tension; (b) torsion.

2.3. Porosity analysis on pure adhesive coupons

Due to the high porosity found in the coupons, a porosity analysis is performed on pure adhesive coupons. The main reason of the large porosity is the high viscosity of the bonding paste, as it is observed in both dog-bone shape and tubular specimens.

The procedure consists on cutting an adhesive sample, embedding it in metallographic testing resin and polishing it until a smooth surface is obtained. Taking photographs in the microscope and using the software called ImageJ, it is possible to obtain some conclusions. Fig. 4 is an example of the porosity obtained in one section of an adhesive coupon. The majority of the pore sizes are really small, but, a few pores are much larger. This porosity is usually responsible for the initiation of both quasi-static and fatigue failures. These results are for a section cut from the cylinder coupon type. It would be interesting to perform the same analysis of different specimen dimensions to see what are porosity sizes and therefore to analyse the size effect, this is, the relationship between porosity and specimen dimensions.

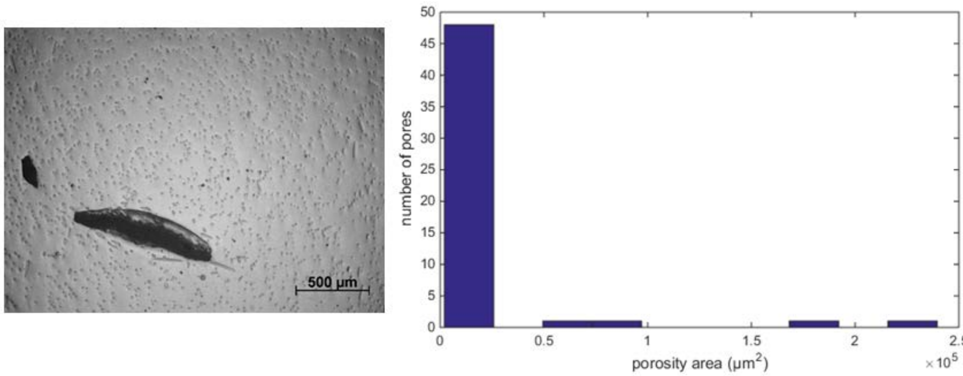

Fig. 4. Example of porosity study in an adhesive coupon.

2.4. Quasi-static tests in combined tension and torsion tests on bonded joint coupons

As the particular focus of this research is on large joint thickness and on multi-axial stress/strain state, the test specimen needs to be designed with similar characteristics. For that purpose, several joint designs are studied and compared, and after considering different geometries, the tubular spar cap - shear web specimens realize the desired stress distribution in compliance with real application (Fig. 2(c)). These composite coupons have three window sizes

(10, 5 and 1 mm). Five coupons of each size are tested. Almost in all coupons the failure type is cohesive, this is, in the adhesive itself.

2.5. Torsion fatigue tests on bonded joint coupons

Butt joint type coupons are employed to conduct an experimental campaign in torsion fatigue (Fig. 2 (d)). All coupons are manufactured with 5 mm of adhesive. These tests are performed with the same test parameters as the bulk adhesive fatigue torsion tests, this is, at 7 Hz and R = -1. Fig. 5 shows the results of this set of tests. Many difficulties are encountered while manufacturing these coupons. Good surface preparation is essential to avoid premature interfacial failure. The discarded results observed in Fig. 11 are the first batch of coupons where this type of failure occurred. However, improved surface quality by cleaning it properly and giving appropriate roughness, dramatically increased fatigue strength, even leading to some run-outs, interrupting tests at 10 million cycles. The results may suggest that fatigue in bonded joints may be higher than in bulk adhesive coupons.

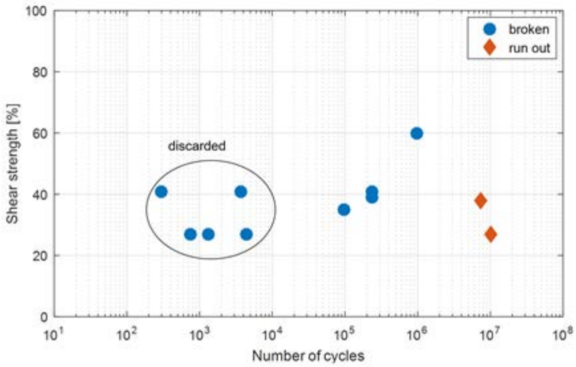

Fig. 5. Fatigue results in composite butt joint.

3. Failure criteria analysis and probabilistic method for strength prediction

In the current paper, instead of applying failure criteria in a deterministic way, a probabilistic approach is used. The knowledge of the failure criterion proved to be insufficient to predict joint strength capacities, since the strength of brittle materials exhibits a size effect (Vallée et al. (2011)). Size effects are related to the concept of randomly distributed defects and flaws. This concept states that due to randomly distributed flaws in the body, the probability that a flaw leads to failure increases with increasing specimen size. Therefore, larger specimens have higher probability of containing flaws, resulting in lower strengths.

For the implementation of any strength prediction method a failure criterion for the material is needed. Different failure criteria are compared and the most appropriate one is chosen. Therefore, the strength prediction method considers a statistical size effect in the strength of the material by considering not only the magnitude of the stress distributions, but also the volume over which they act. In this paper, material strength is modelled using a Weibull statistical function. Experimental data obtained for bulk adhesive material are taken as a reference and the information extracted from this experimental campaign is applied to other specimens.

Several studies have investigated the influence of size effect in the strength prediction. In composite materials, the strength of brittle fibers such as carbon or glass exhibits a size effect due to statistically distributed flaws in the microstructure of the fibers. The strength increases with decreasing diameter of the fibers (Zafeiropoulos (2011), Das et al. (2014)). Size effects have also been observed in epoxy matrix materials (Odom et al. (1992), Seo et al. (2005)). In addition to defects in the resins and fibers, composite materials also have defects in the microstructure that are contributing to the size effect. These weaknesses are heterogeneities in the packing of the fibers, resin rich regions, voids, delaminations, broken or misaligned fibers, fibers debonded from the matrix or cracks due to shrinkage during cure. Furthermore, Vallée et al. (2006); (2011) have investigated failure in composite and timber materials using probabilistic methods and results can be considered as representative in a wider sense.

The basis of the Weibull distribution is formed by the weakest link theory, this is, it assumes that the material is built up of smaller elements linked together in a chain and that failure of the material as a whole occurs when one of these elements fail. If the coupon is idealized as being constituted by n elements from a finite element model, then specimen survival, following the weakest link theory, depends on simultaneous non-failure of all elements.

3.1. Selection of the best-fit failure criterion

Different failure criteria are considered as potential candidates for a best fit to the adhesive material. Failure curves are calculated for the different criteria taking the 50% of probability of survival for strength prediction. Fig. 6 (a) shows the map where pure tensile data are marked on the y axis, pure shear data on the x axis and the combined tensile-shear data are marked between the axes depending on the ratio. The experimental data are obtained from the different tests performed on bulk adhesive coupons. On the other hand, the different lines represent the probability of survival of 50% of the coupons for the different failure criteria. This map shows that some failure criteria match the experimental data much better than others. Anyway, the Root Mean Square Error (RMSE) method is used to choose the most suitable failure criterion for further analysis. For this specific case the failure criterion named isotropic parabolic failure surface criterion (IPFSC) is found to match the data most closely (Stassi d'Alia (1967). Fig. 6 (b) shows the map where different probabilities of survival (95%, 50% and 5%) are calculated. One of the main advantages of this methodology is that the design engineer can define the percentage of the probability of survival that he/she wants to use for further calculations.

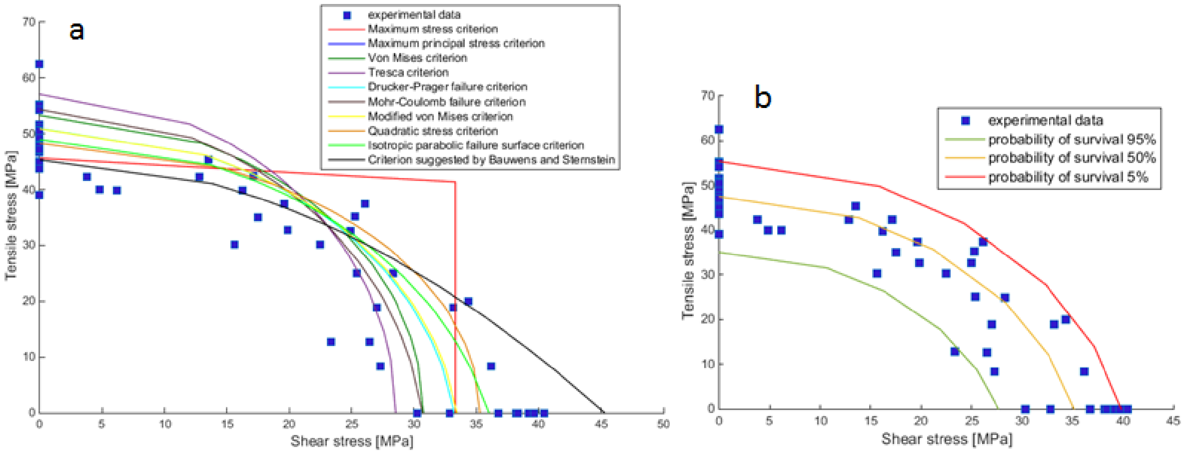

Fig. 6. (a) Failure criteria and adhesive experimental data; (b) Probability of survival curves for the isotropic parabolic failure criterion.

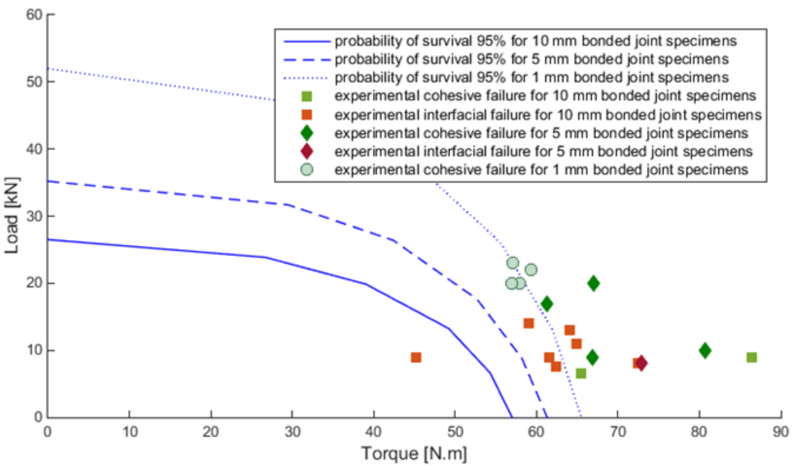

Fig. 7. Probability of survival curves for the bonded joints and obtained experimental data.

The same procedure can be applied to the bonded joint specimens taking into account that now the volumes are different. The stress state and volume of each element of the adhesive material from the FE model are considered to perform this analysis. Fig. 7 illustrates the probability of survival curves (at 95%) for each bonded joint window size. The experimental data obtained for the different specimens are plotted too. As the number of test specimens is relatively low, a significant degree of scatter is observed on the data points. The experimental campaign should be extended to a larger number of samples to be able to extract definitive conclusions.

Last, a final remark should be done to the subcomponent analysis. A specific test structure is designed and manufactured as a C-beam to reproduce load transfer phenomena as they occur in real blades. An experimental test campaign is conducted using different data acquisition principles and sensors to monitor structural behavior. Results from a finite element model are compared to experimental results and satisfactory results are obtained.

4. Fatigue analysis

Accurate prediction of fatigue life is a challenge due to the complicated nature of fatigue crack initiation and propagation, geometry of bonded joints, and complex material behavior under loading and unloading regimes. A particularly important aspect in fatigue analysis is the occurrence of stress concentrations. Local stress peaks which do not affect quasi-static strength may drastically reduce the fatigue life of cyclically loaded structures. Joints inevitably exhibit stress concentrations, and for this reason they need to be investigated in detail. It is well known that fatigue phenomena exhibit a high degree of scatter, especially in high cycle fatigue (Vassilopoulos (2015)). In most of the models used for the derivation of S-N curves, the probability of failure is not explicitly included, this is why Castillo and Fernandez-Canteli (2009) proposed the fatigue Weibull regression model. As a result, they developed a free software program ProFatigue (Fernández-Canteli et al. (2014)) which is used in this paper.

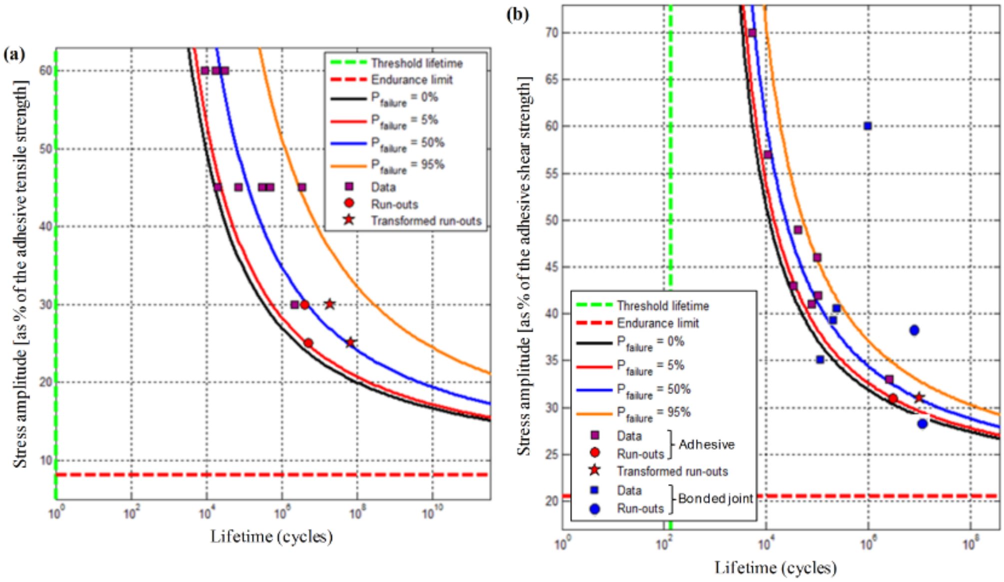

Fig. 8. S-N curves for (a) pure tensile fatigue tests; (b) pure torsion fatigue tests.

Fig. 8 shows the results obtained using ProFatigue for tensile fatigue data Fig. 8(a) and torsion data Fig. 8(b). A graphical representation of the different percentile curves representing the relationship between lifetime N (logarithmic axis) and stress range $\Delta\sigma$ (linear axis) is illustrated in these figures. These curves give an idea of the shape of fatigue curves at different probabilities of failure. In the case of bonded joint samples, due to the limited set of experimental data, it is not possible to do a thorough probabilistic analysis. For that reason, the bonded joint data are plotted in the curves obtained for bulk adhesive torsion fatigue tests. As the bonded joint specimens failure was within the adhesive, and as these data points (bonded joint results) are above the curves extracted for bulk adhesive coupons, it is concluded that bulk adhesive fatigue probabilistic curves can be used bonded joint results, as bonded joint results are above the pure adhesive results.

5. Conclusions

This paper describes an experimental campaign for the identification of the static and cyclic strength of adhesively bonded joints on glass fiber reinforced composite materials. This campaign focuses on joint thicknesses up to 10 mm and on multi-axial stress states, with one component of normal stress and one component of shear stress. Material properties are obtained in uni-axial loading, this is, in tension and in shear. As these loads occur simultaneously, tests are done at different tensile/shear ratios. In addition, fatigue testing is done in tension and in torsion. On the other hand, an intermediate specimen is designed to specifically analyze blade bonded joints. This specimen is subjected to similar multi-axial stress state, where tensile and shear loads are given at the same directions as in blade bonded joints.

The most appropriate failure criteria are identified for this material. The different criteria are not applied deterministically, as the mechanical strength of brittle materials exhibits a size effect. Due to randomly distributed flaws in the body, the probability that a flaw leads to failure increases with increasing specimen size. Therefore, this effect is present in both pure adhesive and bonded joint specimens. The Weibull distribution is found to be well suited to represent adhesive material intrinsic strength. Once the most suitable failure criterion is identified, probability curves at different percentages are calculated. The main conclusion of this analysis is that with the employed methodology, it is possible to identify the best probability curve for each specific case.

Fatigue results are analyzed in a probabilistic sense using the software called Profatigue. Probability failure curves at 0%, 5%, 50% and 95% are extracted. For the case of bonded joint fatigue results, in order to be able to extract reliable conclusions, the experimental fatigue campaign needs to be extended to establish a larger data set which allows for a more realistic prediction. Nevertheless, the probability curves of pure adhesive material show a good potential in the bonded joint specimens.

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