

Characterization of the mechanical properties of a polyurethane adhesive: Tensile strength, fatigue and fracture tests

Henrique M. F. Oliveira

Department of Mechanical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, 4200-465 PORTO, Portugal (up202004505@edu.fe.up.pt) ORCID 0009-0001-5109-7872

Maria J. P. Ribas

Department of Mechanical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, 4200-465 PORTO, Portugal (up201806409@edu.fe.up.pt) ORCID 0009-0004-9689-0489

Alireza Akhavan-Safar

Institute of Science and Innovation in Mechanical Engineering (INEGI), Rua Dr. Roberto Frias, 4200-465 PORTO, Portugal (aakhavan-safar@inegi.up.pt) ORCID 0000-0002-7168-7079

Ricardo J. C. Carbas

Institute of Science and Innovation in Mechanical Engineering (INEGI), Rua Dr. Roberto Frias, 4200-465 PORTO, Portugal (rcarbas@fe.up.pt) ORCID 0000-0002-1933-0865

Eduardo A. S. Marques

Institute of Science and Innovation in Mechanical Engineering (INEGI), Rua Dr. Roberto Frias, 4200-465 PORTO, Portugal (emarques@fe.up.pt) ORCID 0000-0002-2750-8184

Sabine Wenig

Sika Automotive AG, Kreuzlingerstrasse 35, 8590 Romanshorn, Switzerland (Wenig.sabine@ch.sika.com) ORCID 0009-0002-8356-6378

Lucas F.M. da Silva

Department of Mechanical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, 4200-465 PORTO, Portugal (lucas@fe.up.pt) ORCID 0000-0003-3272-4591

Author Keywords	Abstract
Adhesive, polyurethane, fracture energy, fatigue, threshold energy	The aim of this paper is to study the response of a polyurethane-based adhesive when subjected to different loading conditions. To achieve this, double cantilever beam (DCB) and dogbone specimens were manufactured, and tensile strength, fracture and fatigue fracture tests were performed. The results of the tensile tests revealed a tensile strength of 14.5 MPa and an elastic modulus of 0.27 GPa for the studied polyurethane adhesive. The analysis of the fatigue tests showed that fatigue crack growth starts to stabilize after 20% of the test is done, and that the adhesive used is more suitable for static loading conditions than for cyclic loading conditions, since the value of the average G_{th} , 0.2 N/mm, is much lower than the value of the average GI_c , 4 N/mm.
Type: Research article ∂ Open Access ☑ Peer Reviewed ⓒ € CC BY	

1. Introduction

Adhesive bonding has seen an exponential growth over the last years (Laurén 2021), becoming an alternative, or, in some cases, replacing traditional bonding methods like welding and riveting (de Oliveira et al. 2022; Zamani et al. 2019). The fact that it offers a more uniform stress distribution along the bondline, allows the bonding of different materials, and is financially more appealing, are characteristics that have contributed to the rise of adhesive joints in several industries (Sousa et al. 2022). Among different types of adhesives, the use of polyurethane has grown in the last years (Leitsch et al. 2016), since it showcases excellent variety in its properties, both physical and chemical (Ebnesajjad et al. 2015). This means that it can be used in a wide range of applications, such as automotive, apparel and medical industries. At the same time, variables like strain rate or the service temperature have a big impact on the behavior of these adhesives (Banea and daSilva 2010; Li and Wang 2016). Therefore, it is important to understand how they behave before different loading conditions and mode mixities, since, in real-life applications, most of the adhesives at service are subjected to mixed mode conditions (Chaves et al. 2014; Monteiro et al. 2020).

Fatigue loading is a prevalent condition experienced by adhesive joints in service. In the analysis of bonded structures under fatigue, two distinct strategies have been employed: the stress-life (S-N) approach and fatigue crack growth analysis. Fatigue crack growth in adhesive joints has been widely investigated over the years, indicating the relevance of the topic. (Rocha et al. 2020a) investigated fatigue behavior of one epoxy-based adhesive, when subjected to mixed mode loading conditions, to perform a numerical simulation of it. Mixed mode was split into the corresponding components of modes I and II. Results showed that this approach can be applied to estimate the fatigue life of adhesive joints. The comparison between fatigue fracture behavior of different materials has also been studied before, showing the impact that the selection of the adhesive has on the joint's fatigue life. (Rocha et al. 2020b) studied the fatigue fracture behavior of an epoxy-based, acrylic, and a rubber-like adhesive, testing double cantilever beam (DCB) specimens under several mode mixities and different load levels. Results showed that the crack propagation life is higher for the acrylic adhesive, the variation of the threshold energy with load level is superior for the epoxy-based one, and that for pure mode II, the normalized threshold of the rubber-like adhesive was higher than the epoxy-based one, while for pure mode I conditions, the opposite occurred.

When it comes to polyurethane adhesives, the investigation that has been done so far is significantly lower compared to the previous topic. (Perez et al. 2022) investigated the loading rate and temperature interaction on a polyurethane adhesive, which was ductile and used in the automotive industry, as well as their effects on its mode I fracture response. To achieve that, DCB specimens were manufactured and tested under three different speed levels and temperatures. The results showed that increasing the loading rate from quasi-static (0.2 mm/min) to 6000 mm/min significantly increases the maximum strength of the specimens, especially for DCBs tested at room temperature, where the fracture energy increased by a factor of 3.5. Also in that topic, (Santos et al. 2023) characterized a structural polyurethane adhesive suited for automotive applications in terms of its fatigue behavior in pure mode I. Two testing methods were used, load control and displacement control, and their results were compared. The threshold energy release rate was calculated for different test temperatures. Results showed that the last methodology is superior to a constant load control one, when testing for the threshold energy release rate, and the differences observed between the two methodologies were exacerbated by the effect of temperature on the adhesive.

Despite the studies mentioned before, fatigue crack growth in polyurethane-bonded joints is a subject with much more to be investigated. Therefore, the main aim of this work is to deal with that research gap, studying the impact that different loading conditions have on the behavior of polyurethane-bonded joints.

2. Experimental Details

2.1. Materials

For the tensile strength test, dogbone specimens, made with a two-component polyurethanebased adhesive, were used. According to the data provided by the manufacturer, this adhesive has a curing period of seven days at room temperature. For the fatigue and fracture tests, the joints were made of high strength steel PM300, and a primer was used.

2.2. Specimens' geometry and manufacturing

As said before, dogbone specimens were made of a polyurethane-based adhesive and, for its manufacturing, bulk plates were produced and cut into a dobgbone shape, according to the standard BS 278. Their dimensions can be seen in Figure 1. A silicon frame, with a thickness of 2 mm was used, to help controlling the thickness of the specimen. To avoid the formation of voids, the mold, with the adhesive in it, would be put in a hydraulic press for 24 hours.

In order to perform fracture and fatigue fracture tests, DCB specimens were manufactured, and, after that, submitted to mode I loading conditions.

Before that, the surfaces of the DCBs were sandblasted, to eliminate iron oxides and improve the adhesion, and cleaned with acetone, causing their degreasing. A layer of primer was also applied, 24 hours prior to the adhesive application, in order to improve the adhesion between substrate and adhesive, as well as preventing the specimens from oxidation.

In addition, to ensure the presence of an initial crack, razor blades, with a thickness of 0.1 mm each, were placed between steel spacers, coated with release agent. This procedure did not follow any standard, and no bonding was ensured. The total thickness of this set and the precrack were 2 mm and 0.1 mm, respectively. The length of the pre-crack had a value of 45 mm, counted from the center of the DCB holes. On the other end of the joint, a spacer made of aluminum was placed to control the thickness of the bondline, which was also 2 mm. This thickness was chosen since higher values are of less interest for practical applications, while lower ones present an inferior G_{Ic} (Banea et al. 2015). The dimensions of the substrates can be seen in Figure 2.



Figure 2: Geometry of the DCB specimen.

2.3. Test approach

For the tensile test, an INSTRON 3367 machine, with a load cell capacity of 30 kN, was used with a displacement rate of 1.0 mm/min. To measure strain, an extensometer was placed on the specimen before the start of the test and removed from it when the strain value reached around 100% (due to the limitations of the extensometer used). Three tests were conducted in order to characterize the tensile properties of the adhesive, by obtaining the average Young's modulus, maximum tensile strain and tensile stress.

The fracture test was performed using standard DCBs, as previously mentioned, in the INSTRON 3367 electro-mechanical machine. The standard procedure followed was ISO 25217. Three specimens were subjected to mode I quasi-static loading conditions, with a loading rate of 0.2 mm. The test was performed at room temperature (23°C).

Fatigue fracture test was performed using the INSTRON 8801 servo-hydraulic machine. Similarly to the fracture test, the fatigue test was performed at room temperature and the DCBs were subjected to mode I fatigue loading conditions at a frequency of 12 Hz. Therefore, two DCBs were selected and tested with a displacement control method, which prevents the DCB from failing at the end of the test and allows the achievement of the threshold (Bittencourt et al. 2022; Azari et al. 2010). By using this method, the maximum and minimum displacements remained constant during the test, so that it was possible to observe the minimum and maximum loads. With those two values, it was possible to obtain the fatigue load ratio, *R*, by dividing them, which had a value of approximately 0.25. The crack growth length in each cycle was measured via compliance-based beam method.

3. Results and Discussion

3.1. Tensile bulk tests

For the execution of the tensile strength test, three specimens were tested. With the results provided by the strength test, it was possible to obtain the tensile stress-strain curve. Only one sample is represented in Figure 3.

After obtaining the maximum strain, maximum stress (ultimate strength), and Young's modulus of each one, the average values were calculated, as shown in Table 1.



Figure 3: Tensile stress-strain curve of the Bulk test.

	Maximum tensile stress (MPa)	Strain at failure (%)	Young's modulus <i>, E</i> (GPa)
Average	14.5	88.2	0.2655
Standard Deviation	0.5	0.1	0.0185

 Table 1: Results of the strength test.

3.2. Fracture test

Regarding the quasi-static fracture test, the load-displacement and the *R*-curve of one selected sample can be seen in Figure 4 and Figure 5.



Figure 5: R-curve of the fracture test.

To obtain Figure 5, a data reduction scheme was used, more precisely compliance-based beam method, as well as equations. By analyzing it, it is possible to conclude that the value of G_{Ic} , for quasi-static conditions, is 4 N/mm.

3.3. Fatigue fracture test

Two specimens were tested, but only one sample is represented in the curves. A representative Paris law curve is shown in Figure 6, and Figure 7 shows the variation in maximum energy as a function of loading cycles for the respective test. The choice of the Paris law curve was made based on a previous study (Rocha et al. 2020b). By using G_{max} for the x-axis, it was possible to obtain the threshold values more directly. By analyzing Figure 6, the *m* value of the adhesive used in both tests was obtained. This value is numerically equal to the slope of the graph, in the zone where G_I is increasing. In addition, it allowed the understanding of their fatigue crack growth. In Table 2, the average values of *m*, obtained with the power law relation of the stable crack growth phase, and the threshold strain-energy release rate, G_{th} , considered as the condition where the rate of crack growth is less than 1E-6 mm/cycle, are represented.



Figure 6: da/dN - Gmax curve of the fatigue test.



Figure 7: Gmax - Normalized Cycles curves of the fatigue test.

By analyzing Figure 7, it is possible to see that the value of G_{max} begins to steady when normalized cycles' value is 0.2. The normalized cycles were normalized by the maximum number of cycles obtained in each test. Therefore, it is possible to conclude that fatigue crack growth begins to steady after 20% of the test is done, which suggests that the energy reached its threshold value (Pirondi and Moroni 2010; Sekiguchi et al. 2023).

	m	Threshold strain energy release rate, G _{th} (N/mm)
Average	4.0	0.2
Standard Deviation	0.2	0.1

 Table 2: Results of the fatigue test.

Lastly, after the analysis and comparison of both G_{Ic} quasi-static value, 4 N/mm, and the average G_{th} , 0.2, represented in Table 2, it is possible to see that the value of G_{th} is significantly lower than the value of G_{Ic} . This gives an indication about the behavior of the adhesive before cyclic and static loading conditions.

4. Conclusions

After conducting three types of tests on adhesive joints, namely tensile strength and mode I fracture tests under quasi-static and fatigue loading conditions, conclusions can be drawn. Tensile strength tests showed that the experimental value of the adhesive's Young's modulus is 265.5 MPa, while the values of its maximum tensile stress and strain are 14.5 MPa and 88.2%, respectively. Fatigue fracture tests results showed that the energy reaches its threshold value after 20% of the test is done. In addition, the value of the average G_{th} is much lower than the value of G_{Ic} for quasi-static conditions, which allows to conclude that the adhesive used is more prone to the initiation and propagation of cracks under cyclic loading than under static loading.

References

- Azari, S., M. Papini, J. A. Schroeder, and J. K. Spelt. 2010. "Fatigue threshold behavior of adhesive joints". *International Journal of Adhesion and Adhesives* 30, no. 3: 145-159. https://doi.org/10.1016/j.ijadhadh.2009.12.004.
- Banea, M. D., and L. F. M da Silva. 2010 "The effect of temperature on the mechanical properties of adhesives for the automotive industry". *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications* 224, no.2: 51-62. https://doi.org/10.1243/14644207JMDA283.
- Banea, M. D., L. F. M. da Silva, and R. D. S. G. Campilho. 2015. "The effect of adhesive thickness on the mechanical behavior of a structural polyurethane adhesive". *The Journal of Adhesion* 91 no.5: 331-346. https://doi.org/10.1080/00218464.2014.903802.
- Bittencourt, M. H. S., A. Akhavan-Safar, D. R. C. O. A. Santos, S. Wenig, and L. F. M. daSilva. 2022. "Fatigue threshold analysis of adhesives: displacement control vs. load control strategy". *Journal on Mechanics of Solids* 1 no.1: 9-14. https://doi.org/10.24840/2975-8262_001-001_001843.

- Chaves, F. J. P., L. F. M. da Silva, M. F. S. F. de Moura, D. A. Dillard , and V. H. C. Esteves. 2014. "Fracture mechanics tests in adhesively bonded joints: a literature review". *The Journal of Adhesion* 90 no.12: 955-992. https://doi.org/10.1080/00218464.2013.859075.
- de Oliveira, L. Á., M. M. Vieira, J. C. dos Santos, R. T. S. Freire, M. L. P. Tonatto, T. H. Panzera, P. Zamani, and F. Scarpa. 2022. "An investigation on the mechanical behaviour of sandwich composite structures with circular honeycomb bamboo core". *Discover Mechanical Engineering* 1: 7. https://doi.org/10.1007/s44245-022-00006-z.
- Ebnesajjad, S., and A. H. Landrock. 2015. "Chapter 4 Classification of Adhesives and Compounds". In *Adhesives Technology Handbook (Third Edition)*, 67-83. William Andrew. https://doi.org/10.1016/B978-0-323-35595-7.00004-8.
- Laurén, S. 2021. "Cohesive vs. adhesive failure in adhesive bonding." Assessed May 12, 2023. https://www.biolinscientific.com/blog/cohesive-vs.-adhesive-failure-in-adhesivebonding.
- Leitsch, E. K., W. H. Heath, and J. M. Torkelson. 2016. "Polyurethane/polyhydroxyurethane hybrid polymers and their applications as adhesive bonding agents". *International Journal of Adhesion and Adhesives* 64: 1-8. https://doi.org/10.1016/j.ijadhadh.2015.09.001.
- Li, P., and Z. Wang. 2016. "Experimental characterization and modified constitutive modeling of the strain rate dependent compressive behavior of adhesives". *Macromolecular Materials and Engineering* 301, no. 5: 577-585. https://doi.org/10.1002/mame.201500415.
- Monteiro, J., A. Akhavan-Safar, R. J. C. Carbas, E. A. S. Marques, R. Goyal, M. El-Zein, and L. F. M. da Silva. 2020. "Influence of mode mixity and loading conditions on the fatigue crack growth behaviour of an epoxy adhesive". *Fatigue & Fracture of Engineering Materials & Structures* 43, no. 2: 308-316. https://doi.org/10.1111/ffe.13125.
- Perez, M., A. Akhavan-Safar, R. J. C. Carbas, E. A. S. Marques, S. Wenig, and L. F. M. da Silva. 2022. "Loading rate and temperature interaction effects on the mode I fracture response of a ductile polyurethane adhesive used in the automotive industry". *Materials* 15, no. 24: 8948. https://doi.org/10.3390/ma15248948.
- Pirondi, A., and F. Moroni. 2010. "A progressive damage model for the prediction of fatigue crack growth in bonded joints". *The Journal of Adhesion* 86, no. 5-6: 501-521. https://doi.org/10.1080/00218464.2010.484305.
- Rocha, A. V. M., A. Akhavan-Safar, R. J. C. Carbas, E.A.S. Marques, R. Goyal, M. El-Zein, and L.F.M. da Silva. 2020a. "Numerical analysis of mixed-mode fatigue crack growth of adhesive joints using CZM". *Theoretical and Applied Fracture Mechanics* 106: 102493. https://doi.org/10.1016/j.tafmec.2020.102493.
- Rocha, Ana VM, A. Akhavan-Safar, R. J. C. Carbas, E. A. S. Marques, R. Goyal, M. El-Zein, L. F. M. da Silva. 2020b. "Fatigue crack growth analysis of different adhesive systems: Effects of mode mixity and load level". *Fatigue & Fracture of Engineering Materials & Structures* 43, no. 2: 330-341. https://doi.org/10.1111/ffe.13145.
- Santos, D., A. Akhavan-Safar, R.J.C. Carbas, E.A.S. Marques, S. Wenig, and L.F.M. da Silva. 2023.
 "Load-control vs. displacement-control strategy in fatigue threshold analysis of adhesives: Effects of temperature". *Engineering Fracture Mechanics* 284: 109255. https://doi.org/10.1016/j.engfracmech.2023.109255.
- Sekiguchi, Y., K. Houjou, K. Shimamoto, and C. Sato. 2023. "Two-parameter analysis of fatigue crack growth behavior in structural acrylic adhesive joints". *Fatigue & Fracture of Engineering Materials & Structures* 46, no. 3: 909-923. https://doi.org/10.1111/ffe.13908.

- Sousa, F., A. Akhavan-Safar, R. Goyal, and L. F. M. da Silva. 2022. "Fatigue life estimation of adhesive joints at different mode mixities". *The Journal of Adhesion* 98: 1-23. https://doi.org/10.1080/00218464.2020.1804376.
- Zamani, Pedram, A. Jaamialahmadi, L. F. M. da Silva, and K. Farhangdoost. 2019 "An investigation on fatigue life evaluation and crack initiation of Al-GFRP bonded lap joints under four-point bending". *Composite Structures* 229: 111433. https://doi.org/10.1016/j.compstruct.2019.111433.