EFFECT OF TEMPERATURE ON DAMAGE MECHANISMS IN AN ACRYLIC-MATRIX AND GLASS-FIBER-REINFORCED COMPOSITE UNDER MONOTONIC TENSILE AND FATIGUE LOADINGS

E. Boissin[1], C. Bois[1], J.-C. Wahl[1] and T. Palin-Luc[2]

[1]I2M, IUT de Bordeaux, CNRS, University of Bordeaux, 15 rue de Naudet 33170 Gradignan, France
Email: eileen.boissin@u-bordeaux.fr, christophe.bois@u-bordeaux.fr, jean-christophe.wahl@u-bordeaux.fr
[2]I2M, Arts et Métiers Paristech, CNRS, Esplanade des Arts et Métiers 33405 Talence, France
Email: thierry.palin-luc@ensam.eu

Keywords: temperature, damage, laminate, acrylic matrix, static, fatigue

Abstract
Effect of temperature on the damage scenario in an acrylic-matrix and glass-fiber reinforced composite has been studied under monotonic quasi-static and fatigue loadings. Results highlights a significant effect of temperature on the damage scenario for both loading types, this one being different at 15°C and 40°C. Observed damage scenarios have also been compared regarding the loading type, showing that the damage scenario at 40°C is different whether the loading is quasi-static or cyclic.

1. Introduction

Wind energy industry, as well as automotive or maritime industries, mostly uses thermoset-matrix composites for their parts manufacturing, but these composites are hardly recyclable. In order to reduce the environmental footprint, new polymer matrices have been developed in the last years to replace commonly used thermoset resins, including partly or fully bio-sourced thermoset resins, biodegradable polymer matrices but also acrylic thermoplastic matrices. These latter are gifted with better recycling and repair opportunities but also with reduced manufacturing costs as their polymerization is performed at room temperature, unlike other commonly used polymer matrices [1].

During its twenty years in service, a turbine blade suffers from 10^8 to 10^9 fatigue cycles [2] and is exposed to temperatures from -20°C to 60°C, in dry and wet environments. These cyclic loadings can lead to a significant deterioration of the composite mechanical properties [3–5], due to damages occurrence: fiber-matrix debonding, cracking and delamination. These damages occur at different scales, can interact with each other and lead to premature fiber breakings. Thus, the composite final fracture is due to a combination of these damage mechanisms [5]. However, there is no or few data on the behavior, the damage scenario and the sensitivity to temperature of acrylic-matrix composites under monotonic quasi-static or fatigue loadings.

2. Effect of temperature on polymer composites

In general, studies dealing with the effect of temperature on the mechanical properties of polymers highlight a stiffness and strength reduction with an increase in ultimate strain when temperature increases towards the glass transition temperature (T_g) [6–9]. These changes may be observed in a range of temperature well below the glass transition temperature as for PMMA (T_g=105°C) where they can be observed between 4 and 60°C [7, 8]. However, in the case of polymer composites, it is more complicated. First of all, polymer composites under monotonic tensile loadings in a fiber-dominant direction (i.e. fiber and loading directions are the same) do not show any significant temperature-dependent behavior [10–12]. On the contrary, off-axis behavior of polymer composite is generally strongly temperature-dependent, since the load is applied in a matrix-dominated direction [11], [12]. In general, a mechanical behavior similar to the neat polymers one is observed [12, 13]. However, the level of temperature sensitivity also depends on the reinforcement nature, the fiber sizing quality and the thermal stress induced by the manufacturing process [14, 15]. Thus, it is difficult to estimate the level of temperature sensitivity of a composite system which has never been tested before.

Furthermore, testing temperature may also affect damage scenario in polymer composites [16, 17]. Indeed, changes in matrix and fiber-matrix interface mechanical properties may lead to new stress distributions into the laminate at the micro-scale, which may lead to the observation of a different damage scenario. Thus, the damage initiation threshold and failure occurrence can be delayed [16], and some damage mechanisms can become more predominant, such as cracking or matrix plasticity [10, 17]. However, the lack of studies with in-situ damage observations makes difficult to clearly understand the effect of temperature on the damage scenario in polymer laminate composites, whereas understanding the mechanical properties deterioration during structure service lifetime is essential to be able to model it and thus, reduce costs and duration of fatigue experimental campaigns. It may also help to find a way to improve the fatigue strength of polymer laminate composites.

3. Material

The studied material is an acrylic-matrix and glass-fiber-reinforced laminate composite. Two kinds of reinforcements, whose fiber sizing is optimised to be combined with an acrylic resin, were used: unidirectional plies G-PLY™ UD960 CT3,8 (UD) and biaxial non crimped fabrics G-PLY™ BXS1000 C3,4 (BXS). The laminate composite was fabricated by infusion and fiber fraction volume was evaluated at 55% for BXS panels and at 61.5% for UD panels. Plus, observations of panel coupons using an optic microscope showed a void rate lower than 3%. The glass transition temperature of the Elium® acrylic resin was provided by the manufacturer and is about 109°C, with a maximum temperature service fixed at 85°C.

Damage mechanisms have been observed under monotonic quasi-static and fatigue loadings on several cross-ply laminates, which allow focusing on the observation of cracking, since the predominant damage mechanism in these laminates is transverse cracking.

4. Effect of temperature on damage mechanisms under monotonic tensile loadings

4.1. Testing method

Tests were performed on a MTS Systems Alliance RF/100 electromechanical testing machine and the temperature was controlled thanks to an environmental chamber set around the specimen and the clamping jaws. The temperature was measured using four thermocouple sensors (one on each machine clamping jaw and two on specimen gage length). Plus, a moisture content sensor has also been placed in the environmental chamber to measure the relative humidity variation all the tensile tests long.

The environmental chamber was conceived with a sliding door allowing setting an optical microscope to observe damages on the polished edge of tested specimens, and moving it in the three space directions to cover the whole observed area. Thus, damage observations were performed in-situ: specimens were loaded step by step at different total strain levels to create damages, and then slightly unloaded to prevent potential evolutions due to viscous phenomena during the scan of the observed area. The latter was achieved via a camera mounted on the microscope, which takes pictures with an overlapping and merges them in order to rebuild the whole observed area on dimensions reaching several centimeters long over all the specimen thickness [5, 18, 19]. This method allows considerably reducing the test duration and preventing potential placement mistakes since it is not necessary to unmount the specimen after each strain level. Furthermore, it also eases damage detection since damages are more open when specimens are tensile loaded.

All tests were performed at 0.5mm/min and a 50mm-gage-length extensometer was used for strain measurements. A 30mm-long observation area was considered, which was long enough to be representative of the whole specimen while keeping decent test duration. Three specimens were tested per temperature for BXS [$(0/90)_4$] specimens, two per temperature for BXS [$(0/90)_2]_s$ specimens and one at 40°C and two at 15°C for UD [$0/90_3/0$] specimens.

4.2. Results

First, it has to be noted that moisture conditions were the same for all specimens, which allowed comparing the temperature effect without undergoing a coupling with a potential moisture content effect. No significant impact of temperature on the mechanical behavior of these laminations was found regarding the strain-stress curves. This can be explained by the fact that the fiber mechanical behavior is dominant in laminates with 0° plies, i.e. fibers in the load direction. This is supported by the determination of each specimen’s stiffness (Table 1) where no significant effect of the temperature is observed regardless the lamination.

However, results showed a different damage scenario at 15°C than at 40°C regardless the considered lamination. Indeed, at 15°C, several transverse cracks are observed on the specimen polished edge, in 90° plies (Figure 1(a)), whereas at 40°C, there is none (Figure 1(b)). Moreover, at 10°C, only cracking is observed at fiber strand ends (Figure 2(a)), whereas at 40°C, small cracks surrounded by a dark area are observed, which seems to imply a greater capability of plastic deformation of the matrix (Figure 2(b)).

Table 1. Longitudinal modulus of BXS [(0/90)]4, BXS [(0/90)2]S and UD [$0/90_3/0$] specimens at 15°C and 40°C

	Lamination	15°C	40°C	Difference (%)
Longitudinal modulus (GPa) *(Number of tested specimens)*	BXS $[(0/90)]_4$	23,6 ± 1,7 *(3)*	23,2 ± 0,7 *(3)*	-1,7
	BXS $[(0/90)_2]_S$	22,9 ± 0,6 *(2)*	20,9 ± 2,5 *(2)*	-8,7
	UD [$0/90_3/0$]	21,9 ± 1,7 *(2)*	23,7 ± 1,9 *(1)*	+8,2

X-ray micro-tomography was used on a volume of the observation area of two BXS $[(0/90)_2]_s$ specimens, the first tested at 15°C and the second at 40°C, with the aim of supporting that damages observed on specimen edges were the same in the specimen cores. Specimens were loaded under a static force in order to open damages and make them detectable at the maximum resolution allowed by the specimen loading system. Thus, at the micro-tomograph room temperature, considered specimens were loaded at 0.6% of total longitudinal strain (i.e. around 11000N), while the allowed maximum resolution was 5.5x5.5x5.5 μm^3. The maximum size of the observed volume per scan was around 0.5cm^3. In the case of the specimen tested at 15°C, tranverse cracks and matrix damages were observed in the specimen core, while in the case of the specimen tested at 40°C, only matrix damages were found, supporting results from damage observation on the polished edge of specimens.

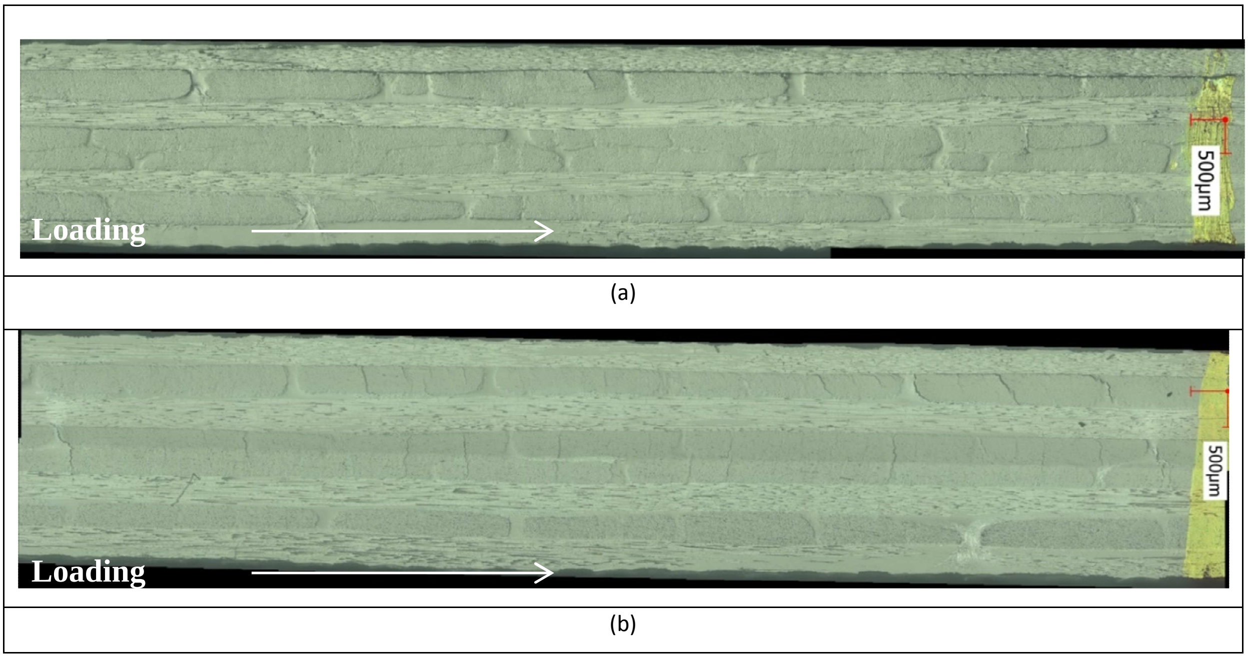

Figure 1. Observation area at 2.4% of total longitudinal strain of a BXS $[(0/90)_2]_s$ specimen edge tested under quasi-static tensile load at (a) 40°C with no transverse cracks and (b) 15°C with numerous transverse cracks

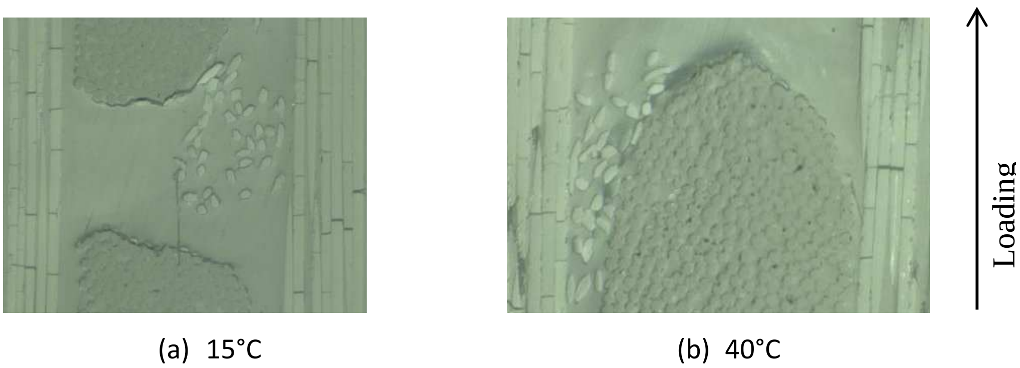

Figure 2. Difference of damage on fiber strand ends between a specimen tested at 15°C and one tested at 40°C, at 1.8% of total longitudinal strain

5. Effect of temperature under fatigue loadings

In the case of fatigue loadings, BXS $[(0/90)_2]_S$ specimens were tested at 15, 25 and 40°C, using a environmental chamber on a MTS servo-hydraulic machine. All tests were carried out in tension R=0,1 under force control with a sinusoidal signal at a frequency of 3Hz. The frequency was taken as high as possible while keeping specimen self-heating under 5°C. The maximum and minimum values per cycle of both stress and total strain werer recorded all the test long.

5.1. Effect of temperature on the fatigue life and the stiffness loss

As described in part 1.2., dumbbell-shaped specimens were used for the determination of fatigue life and stiffness loss. The number of specimens tested per load level at each temperature is shown in Table 2, where σ_{rup} is the ultimate tensile strength determined during the previous monotonic quasi-static tensile tests, i.e. 600MPa. Test frequency and duration are also shown in Table 2. Temperature was measured via four thermocouple sensors and strain was measured using a 20mm-gage-length extensometer. Only two specimens did not break in the gage length but in one of their tabs, both tested at 40°C, one at a maximum stress of 300MPa, and one at a maximum stress of 240MPa. The S-N curve, shown in Figure 3, did not highlighted a significant effect of testing temperature on the fatigue life of BXS $[(0/90)_2]_S$ specimens, regardless the fatigue load level.

Table 2. Number of tested specimens per load level at each temperature with σ_{rup} the ultimate tensile strength and related test frequency and duration (load ratio R = 0.1)

Load level	15°C	25°C	40°C	Frequency (Hz)	Test duration (h)
σ_{max} = 50% σ_{rup} = 300 MPa	2	-	2	3	0.5-2.5
σ_{max} = 40% σ_{rup} = 240 MPa	2	2	2	3	4-10
σ_{max} = 30% σ_{rup} = 180 MPa	2	1	1	3	30-55
σ_{max} = 20% σ_{rup} = 120 MPa	-	1	1	5	200-300

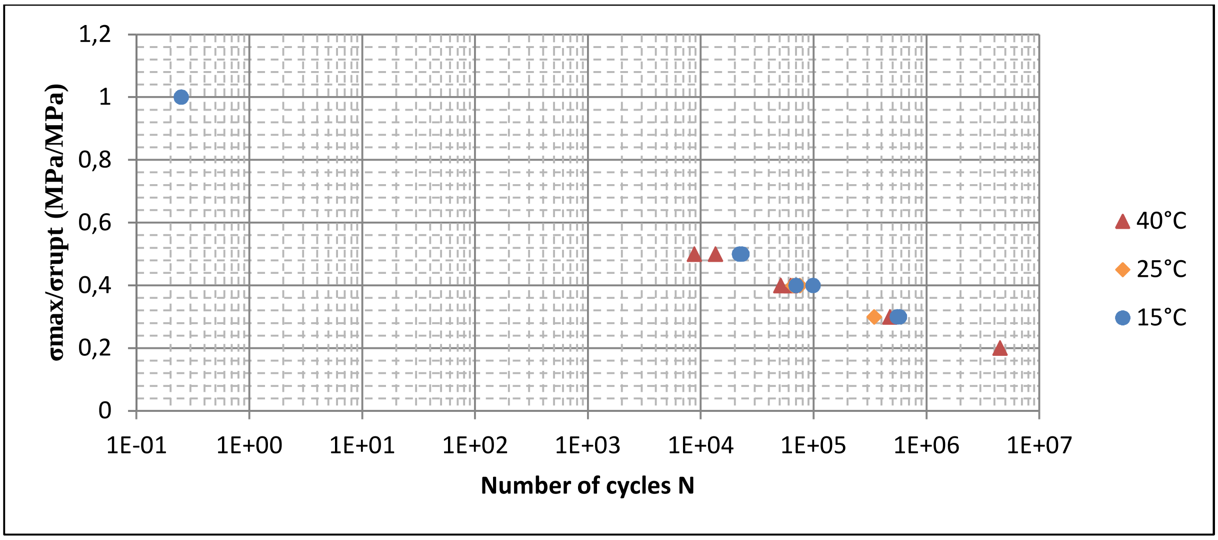

Figure 3. Fatigue life of BXS $[(0/90)_2]_S$ specimens tested at 15, 25 and 40°C at different load levels, at R=0.1

Stiffness loss of the specimens were determined by calculating the secant modulus E_s defined by Eq. (1) at each fatigue cycle, with σ_{max} and σ_{min} respectively the maximum and minimum stress and ε_{max} and ε_{min} respectively the maximum and minimum stress of each fatigue cycle.

$$E_s = \frac{\sigma_{max} - \sigma_{min}}{\varepsilon_{max} - \varepsilon_{min}} \quad (1)$$

Results at each load level did not highlighted a significant effect of the testing temperature on stiffness loss of BXS $[(0/90)_2]_S$ specimens regardless the fatigue load level, as shown in Figure 4 in the case of σ_{max} = 50% σ_{rup} = 240MPa.

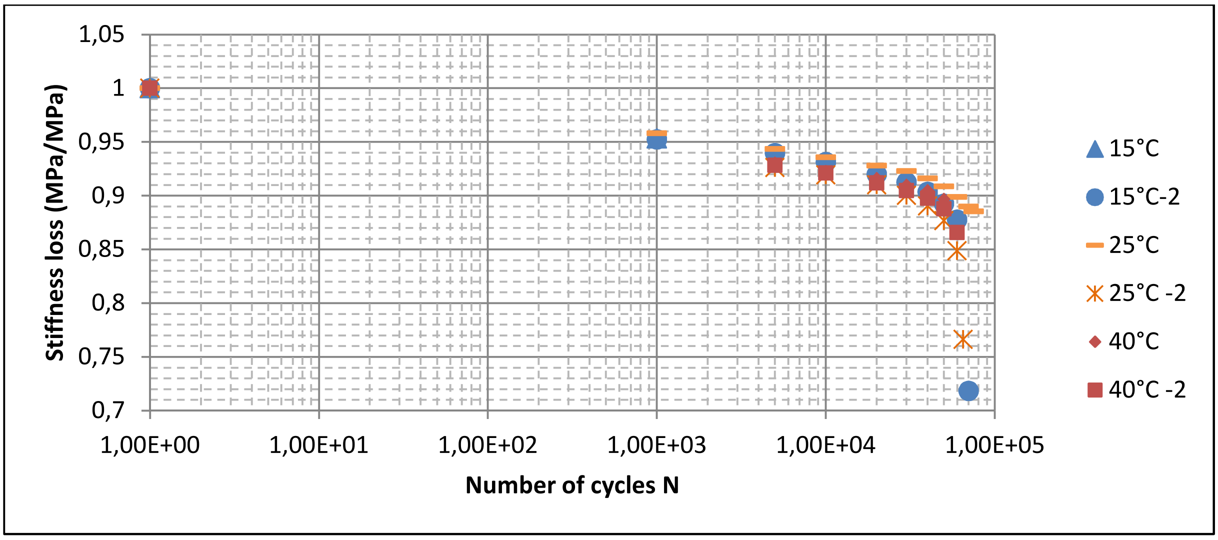

Figure 4. Stiffness loss against number of fatigue cycles at 15°C, 25°C and 40°C at R=0.1 for σ_{max} = 40% σ_{rup} = 240MPa for BXS $[(0/90)_2]_s$ specimens

5.2. Effect of temperature on the damage scenario

In order to ease polishing and damage observation of specimens, 250x25mm² coupons were used instead of dumbbell-shaped specimens. A 30mm-long area was observed on specimen edges at different points of the fatigue life, using the same microscopic observation device than the one used during quasi-stactic tensile tests. In order to be able to see damages by opening them, a tensile load corresponding to a 0.6%-strain was apply on specimens during the observation phase. It has to be noted that all the specimens tested at 40°C and 15°C broke into one of their tabs before reaching 50% of their theorical fatigue lifetime (i. e. around 60 000 cycles).

Transverse cracks were observed before breakage at 15 and 25°C, but also at 40°C whereas no tranverse cracks were observed under a quasi-static tensile load. This is consistent with the fact that fatigue tests encourage crack propagation since the maximum applied stress is lowest than the ultimate tensile strength and the load is cyclic. In order to compare fatigue damage scenario at each temperature, the evolution of the crack density along the fatigue lifetime was determined for all specimens by dividing the number of observed tranverse cracks by the observation area length and then compared (Figure 5).

Results show that, the threshold for first observable transverse crack is delayed at 40°C with respect to those at 15°C and 25°C. For a given number of cycles, it leads to a lower lineic crack density along the observed area on specimen edges at 40°C than at 15°C and 25°C regarding the fatigue lifetime. Since all the specimens tested at 40°C and 15°C broke into one of their tabs before reaching 50% of their theorical fatigue lifetime, no conclusion on the temperature effect can be drawned over this point.

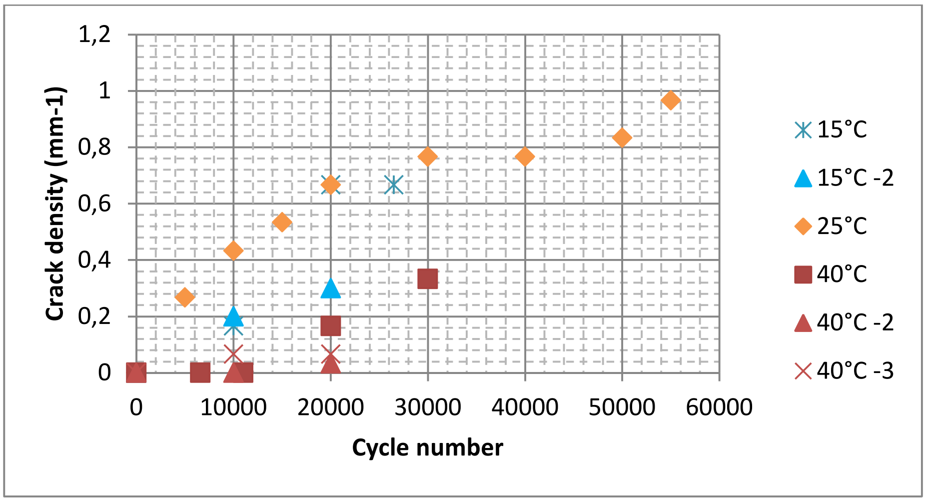

Figure 5. Evolution of the lineic crack density versus the number of cycles on BXS $[(0/90)_2]_S$ specimens under fatigue loading 5r= 0.1) at 15, 25 and 40°C, for σ_{max} = 40% σ_{rup} = 240MPa

6. Conclusion and prospects

Effect of temperature on damage scenario under quasi-static tensile and fatigue loadings was investigated on an acrylic-matrix and gless-fiber-reinforced laminate. Under monotonic quasi-static tensile load, the observed damage scenario is different at 15°C and 40°C. Indeed, at 15°C, several transverse cracks are observed from a 0.4% total strain, whereas at 40°C, there is none until the rupture. This can be explained by a greater ductility of the resin at 40°C than at 15°C. These observations have been confirmed by X-ray micro-tomography results, were the same damages are observed in the specimen cores.

Under fatigue, no significant temperature effect was found on the fatigue lifetime and the rigidiy loss of BXS $[(0/90)_2]_S$ specimens between 15 and 40°C which can be explained by the fact that the load is applied in a fiber-dominant direction. Plus, transverse cracks were observed on all specimens regardless the testing temperature. However, the threshold for transverse crack occurrence seems delayed at 40°C with respect to those at 15°C and at 25°C, leading to a lower lineic crack density along the observed area on specimen edges at 40°C than at 15°C and 25°C until 50% of the theorical fatigue lifetime. However, no conclusion on the temperature effect can be drawn over 50% of their theorical lifetime, since all the specimens tested at 40°C and 15°C broke into one of their tabs before reaching this lifetime. Thus, additional fatigue tests on specimens must be carried out to overcome this lack of datas.

Acknowledgments

The authors thank the Nouvelle Aquitaine region, the ADEME and all the partners of the EFFIWIND project for their support. The EFFIWIND project has been chosen by the regional council and ADEME for a funding as part of the Investissements d'Avenir program.

References

[1] C. Bathias. *Matériaux Composites*. Dunod, 2005.

[2] J. F. Mandell, R. M. Reed, and D. D. Samborsky. *Fatigue of fiberglass wind turbine blade materials*. Sandia National Laboratories, 1992.
[3] J. R. Calcaterra, and S. Mall. Strength degradation during fatigue of unidirectional and cross-ply SCS-6/Ti-15-3 composites. *International Journal of Fatigue*, 21:215‑223, 1999.
[4] G. N. Praveen, and J. N. Reddy. Transverse matrix cracks in cross-ply laminates: Stress transfer, stiffness reduction and crack opening profiles. *Acta Mechanica*, 130: 227‑248, 1998.
[5] N. Revest. Comportement en fatigue de pièces épaisses en matériaux composites. PhD thesis, Ecole nationale supérieure des Mines de Paris, 2011.
[6] V. Jardret, and P. Morel. Viscoelastic effects on the scratch resistance of polymers: relationship between mechanical properties and scratch properties at various temperatures. *Progress in Organic Coatings*, 48:322‑331, 2003.
[7] E. Riande, R. Diaz-Calleja, M. Prolongo, R. Masegosa, and C. Salom. *Polymer Viscoelasticity: Stress and Strain in Practice*. CRC Press, 1999.
[8] E. H. Andrews. *Fracture Mechanics of Polymers*. London: Oliver and Boyd, 1968.
[9] T. Crochon. Modelling of the non linear viscoelastic behaviour of a polyimide matrix. PhD thesis, Department of Mechanical Engineering, Ecole Polytechnique, Montréal, Canada, 2012.
[10] J. Montesano, Z. Fawaz, C. Poon, and K. Behdinan. A microscopic investigation of failure mechanisms in a triaxially braided polyimide composite at room and elevated temperatures. *Materials & Design*, 53:1026‑1036, 2014.
[11] J. Montesano, M. Selezneva, Z. Fawaz, C. Poon, and K. Behdinan. Elevated temperature off-axis fatigue behavior of an eight-harness satin woven carbon-fiber/bismaleimide laminate. *Composites Part A: Applied Science and Manufacturing*, 43:1454‑1466, 2012.
[12] M. W. Hyer, C. T. Herakovich, S. M. Milkovich, and J. S. Short. Temperature dependence of mechanical and thermal expansion properties of T300/5208 graphite/epoxy. *Composites*, 14:276–280, 1983.
[13] H. Mivehchi and A. Varvani-Farahani. The effect of temperature on fatigue strength and cumulative fatigue damage of FRP composites. *Procedia Engineering*, 2:2011‑2020, 2010.
[14] L. Sorrentino, D. S. de Vasconcellos, M. D'Auria, F. Sarasini, and J. Tirillò. Effect of temperature on static and low velocity impact properties of thermoplastic composites. *Composites Part B: Engineering*, 113:100‑110, 2017.
[15] S. Sethi, D. K. Rathore, and B. C. Ray. Effects of temperature and loading speed on interface-dominated strength in fibre/polymer composites: An evaluation for in-situ environment. *Materials & Design*, 65:617‑626, 2015.
[16] K. Vallons. The behaviour of carbon fibre - epoxy NCF composites under various mechanical loading conditions. PhD thesis, Katholieke Universiteit Leuven, Leuven, 2009.
[17] A. El Mourid, R. Ganesan, M. Brochu, and M. Lévesque. Effect of temperature on the failure modes of a triaxially braided polymer matrix composite. *International Journal of Solids and Structures*, 97-98:1‑15, 2016.
[18] C. Bois, J.-C. Malenfant, J.-C. Wahl, and M. Danis. A multiscale damage and crack opening model for the prediction of flow path in laminated composite. *Composites Science and Technology*, 97:81‑89, 2014.
[19] H. Laeuffer, C. Bois, J.-C. Wahl, N. Perry, and F. Lavelle. Damage and permeability of composite laminates. *Proceedings of the 20th International Conference on Composite Materials, Copenhagen, Denmark*, July 19-24 2015.