ACCELERATED ESTIMATION OF FATIGUE PERFORMANCES OF THERMOPLASTIC COMPOSITE MATERIALS BY SELF-HEATING MONITORING

Laura Muller[a], Jean-Michel Roche[a], Antoine Hurmane[a], Catherine Peyrac[b], Laurent Gornet[c]

[a]DMAS, ONERA, Université Paris Saclay, F-92322 Châtillon - France
laura.muller@onera.fr
[b]CETIM, FCM, 52 avenue Félix Louat, 60300 Senlis, France
catherine.peyrac@cetim.fr
[c]Ecole Centrale de Nantes, GeM, UMR CNRS 6183, 1 rue de la Noë, 44321 Nantes, France
laurent.gornet@ec-nantes.fr

Keywords: thermoplastic matrix, carbon fibers, self-heating, thermoelasticity, fatigue

Abstract
Self-heating tests are an alternative experimental approach to classic fatigue tests in order to estimate the fatigue limit of materials. This experimental procedure is based on the monitoring of the self-heating of the studied material under short cyclic loading blocks. The aim is to correlate a change of the thermal behaviour of the material with the appearance of fatigue damage in the material. Acoustic emission monitoring and microscopic damage inspection could be used to confirm the accuracy of the self-heating test method. In this paper, self-heating tests are applied to a balanced 2D-woven carbon/thermoplastic composite material. In addition, quasi-static tensile tests are performed to characterize the mechanical properties of material, as well as to monitor the evolution of and the thermal field. The thermal behaviours of the material under tensile static loads and during self-heating tests are compared and found to be quite similar which opens very promising prospects as for the determination of the fatigue limit of thermoplastic composites.

1. Introduction

Over the years, composite materials are more and more used for primary structural components. This phenomenon is due to their high strength and stiffness to weight ratios. Under these conditions, it has become very important to study their static and fatigue behaviour: indeed, structural components are designed with high safety factors, as the fatigue behaviour of the used composite material is not completely known and understood [1]. So far, the main method used in the industry to study the fatigue behavior of a given material is to build its Wöhler curve, also called S/N curve, which provides the number of cycles to failure (N) for each load level (S). Yet, the constitution of the Wöhler curve requires a huge number of samples and several months of test.

An alternative to the conventional, time-consuming fatigue tests is the self-heating tests which are a much faster step-wise loading method: instead of cycling each sample until failure for several given load values, several blocks with a limited number of cycles for each block are applied on the same sample, for increasing load values (**Figure 1**). During each block of mechanical cycles, the surface temperature of the sample increases until a stabilized temperature field. An theoretical average temperature evolution during one block is given in **Figure 2**. The self-heating is first due first to the resin viscosity and then, after a critical point, to any kind of micro damage growth induced by the cyclic loading. The field can be monitored by an infrared camera or thermocouples in several points, and compared block after block. The evolution of the average stabilized heating with the maximal stress value of each block shows two tendencies: for the lowest load values, the stabilized heating is almost constant from one block to another. Then, after a particular load value, the stabilized heating strongly increases block after block. This change of the thermal behaviour may indicate an alteration of the viscous properties of the resin and/or the initiation of fatigue damage (**Figure 3**). This approach has been proven efficient for metallic materials [2]–[6] and successfully applied to some composites [7], [8] : the specific load value between the two thermal tendencies during the self-heating tests is related to the fatigue limit determined on the Wöhler curve. It seems that the self-heating monitoring can indeed enable a reliable monitoring of fatigue damage.

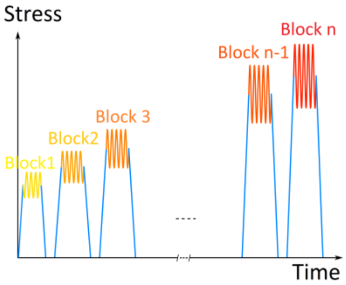

Figure 1 : Schematic representation of self-heating tests

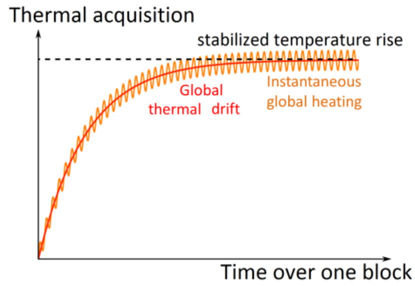

Figure 2 : Temperature rise over one block

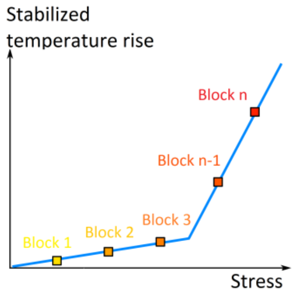

Figure 3 : Self-heating curve

In this context, the purpose of the work is to study the thermal behavior of a balanced 2D-woven carbon/thermoplastic composite material for quasi-static and dynamic loads. The present paper is focused on the analysis of a quasi-static test campaign. The stakes are twofold:

(1) Such test is needed to determine the mechanical properties in three main directions (weft, warp and shear). The fiber orientation at 0° allows characterizing the longitudinal behavior of elementary ply and the ± 45°, the plane shear behavior.

(2) It is also relevant to monitor the thermal behavior of the material under static loading in order to identify additional thermal indicators and compare them the self-heating parameters.

Another key-point of the study is to assess the possible effect of moisture, which is a first-order parameter for thermoplastic materials and well-known for carbon epoxy composites [9]. Consequently, the influence of moisture on the mechanical behavior has been careflully studied.

2. Quasi-static tensile test campaign

1.1 Material and conditioning

The studied material is a balanced plain woven carbon/thermoplastic composite. The thermoplastic matrix is a semi-crystalline polymer, the polyamide 6.6, also called PA66. This resin has the particularity to be very sensitive to moisture absorption : the polyamid can absorb 2% to 5% moisture, depending on the relative humidity [10], [11]. This important sensitivity to moisture has a direct impact on the mechanical properties (like the Young modulus or the ultimate tensile strength) [7], [12]. To guarantee the consistency of the experimental campaigns, samples have to be conditioned with a controlled relative humidity (RH). In this study, the samples are conditioned at ambient temperature, at three different RH values : RH25%, RH50% and RH85%. The conditioning is ensured by desiccators, in which the conditioned air is generated by saturate salted solutions [13] and regularly controlled. The tests are carried out when the sample has reached a moisture balance, which means that its mass is stabilized.

1.2 Quasi-static tensile tests

1.2.1 Experimental procedure

The quasi-static tensile tests are carried out on a ZWICK testing machine, equipped with mechanic grips and a 150 kN load cell. The applied speed load is fixed to 1 MPa/s. Samples are rectangular (250x25mm²) along three directions: 0°, 90°, 45°. Three samples per orientation are tested. The temperature field and specimen displacement are monitored respectively by an infrared camera and optical cameras for Digital Image Correlation (DIC). Their specifications are detailed in [14]. Two acoustic sensors are also used to record each acoustic emission occurring during the test. Another sample is positionned near the tested sample, to monitor the ambient temperature during the tests.

1.2.2 Influence of the relative humidity on the mechanical behaviour

As the moisture is absorbed by the resin part of the composite material, its influence on the mechanical behaviour is more obvious on samples with fibers oriented at $\pm 45°$ relatively to the applied load. Samples are tested up to failure, and the measured displacement fields lead to calculated strain fields. Two samples are tested for each relative humidity condition. As the section shrinks by striction during the test, the actual stress should be calculated as the test goes on. The evolution of the applied force with the strain is then plotted, to compare, at the same given load, the deformations of the specimens in function of RH (**Figure 4**). **Figure 5** represents a focus on the beginning of the curves presented in **Figure 4** to clarify the analysis of the influence of the RH factor.

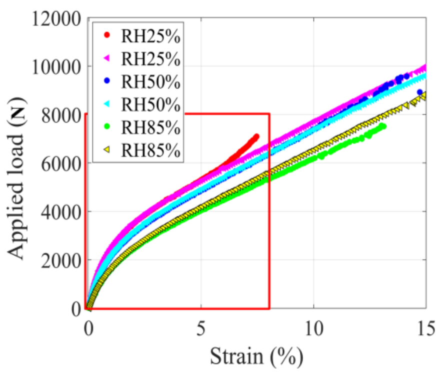

Figure 4 : Stress-strain curves for specimens oriented at 45°, with two samples tested per humidity level

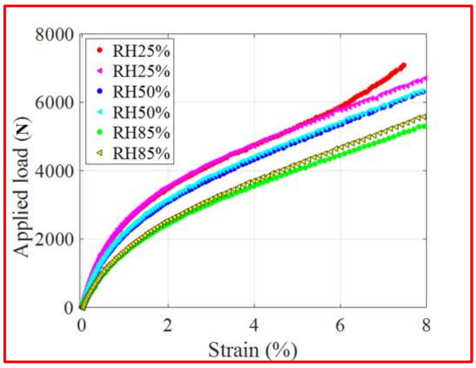

Figure 5 : Focus over the beginning of the applied load -strain curves

Two observations can be made : first, specimens with the same conditioning present a high reproductible behaviour, their strain curves are overlaid. Second, the behaviours are different depending on the RH factor : the strain is less important for low RH specimens. This phenomemon is not major for conditioning between 25% and 50% humidity, and more obvious for specimens conditioned at a 85% relative humidity. Moreover, if the failure load is divided with the failure section to determine the actual stress at failure (axial Cauchy stress componante applied), as summarized in **Table 1**, it appears that the higher the relative humidity is, the lower the failure actual stress is.

The conclusion of this part of the study is that the material has a different behaviour depending on its humidity conditioning, with a higher strength and lower strain for low HR than for high RH. However, this RH-dependency is tempered by the presence of carbon fibres, which are not sensitive to moisture. For the rest of the study, the samples are conditioned at RH50, to approach the ambient conditions.

Table 1 : Ultimate tensile load and actual stress of the [(±45°)] samples, at RH25, RH50 and RH85

Relative Humidity	Ultimate load (N)	Ultimate actual stress (MPa)
RH25	13,414	305
RH25	13,410	300
RH50	12,674	283
RH50	13,300	310
RH85	12,790	292
RH85	11,903	278

1.2.3 Mechanical properties at RH50%

Mechanical characterization involves the determination of the failure stress, the Shear's and Young's moduli and the Poisson ratio along the warp, weft, and shear directions of the material. Only warp and weft plane properties are measured. Assuming that the material strength is equivalent to a laminate composite strength, the Young modulus is experimentally determined according to the EN 2561-91 norm, i.e. by calculating the slope of $\sigma(\varepsilon_{longi})$ on a stress range between 10% and 50% of failure stress (black curve in **Figure 6**). The Poisson's ratio is determined from the slope $\varepsilon_{transv}(\varepsilon_{longi})$ on the same interval (10% to 50% of failure stress) as for Young's modulus. As shown in **Figure 6**, the properties at 0° and 90° are similar, due to the balanced nature of the woven composite. **Table 2** summarizes the properties of samples conditioned at RH50, for both relevant orientations (0° and 45°), by averaging the values obtained for each sample.

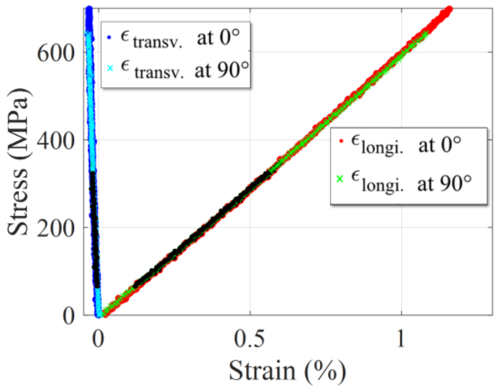

Figure 6 : Stress-strain curve, for the longitudinal and transversal strains for samples at 0° and 90°, and the domains used for the evaluation of the Young modulus and the Poisson's ratio (black)

Table 2: Elastic properties and ultimate tensile stress of the studied carbon T700/PA66 thermoplastic composite material at RH50

Orientation	Ultimate tensile stress	Young modulus	Poisson's ratio
0° (warp)	675 MPa	60 GPa	0.03
45° (shear)	295 MPa		

1.3 Thermal behaviour

1.3.1 Theoretical background

Several authors [1], [15]–[18] have been studying the thermal behaviour of plastic and thermost composite materials during a static tensile test. It is noted that temperature variations follow a three-step pattern:

- The first regime (I) corresponds to the elastic mechanical domain of the material, and the temperature linearly decreases with the increasing stress.

- The second regime (II) corresponds to the anelastic mechanical behaviour, the temperature decreases non-linearly until a minimum.
- The third regime (III) is characterized by the non-linear increase of the temperature, due to viscosity and damage apparition, until the sample breaks.

The linear variation of the heating ΔT in the first domain (I) is related, as described in (1), to the stress level increase $\Delta\sigma$ by thermoelastic effect [19], [20]:

$$\Delta T = -T_0 \, K_m \, \Delta\sigma \quad (1)$$

T_0 is the initial average temperature of the sample, K_m is the thermoelastic coefficient defined as follows:

$$\mathrm{K_m} = \frac{\alpha}{\rho c_p} \quad (2)$$

α is the thermal expansion coefficient in the loading direction, ρ the mass density and c_p the specific heat of the material. According to this law, the end of linearity of the temperature behaviour marks the end of the elastic domain. This change has been correlated with the appearance of micro-damage in [16], [17], on thermoset composites. Then, the transition between the second and the third thermal regimes is marked by a thermal minimum. This transition was correlated with the appearance of macro damage in [16], [17], which would generate enough heat to balance the cooling of the specimen. It was then highlighted that the changes of thermal behaviour could be correlated to the appearance of different types of damage (micro, macro). The authors also noted that for a thermoset composite the stress related to the first thermal transition corresponds to the fatigue limit, which is definitely the most promising conclusion and motivation for the present thermal study on thermoplastic composite materials.

Therefore, the following sections of the paper are dedicated to the assessment of the threshold stress values associated with the transition from one regime to another (the stress value related to the end of the elastic domain and the stress value related to the thermal minimum), and to correlate, if possible, these particular stress values to physical phenomenon (such as viscosity and first micro damages) potentially linked to fatigue initiation.

1.3.2 Results and discussion

For a 0° specimen, the evolution of the average heating is in good agreement with the literature description, as shown in **Figure 7**. To determine the two particular stress values mentioned before, the noise due to the infrared camera needs to be filtered. To do this, a Gaussian filter is applied, as shown in **Figure 8**. Once the thermal signal is filtered, the $\ell 1$ trend filtering, a statistical regression method called "segmented regression", is applied : the variables (the thermal curve) are segmented and a regression analysis is performed on each segment [21], [22]. The thermal curve is interpolated as a succession of linear regressions (**Figure 9**), and the number of segments is defined by the $\ell 1$ trend filtering so that the correlation coefficient between the curve and the line is minimized. The accuracy of this minimization can be adjusted according to the $\ell 1$ parameter λ, which is a regulation parameter used to control a trade-off between the experimental curve segmentation and the residue size.

The junctions between two straight lines are highlighted by dots (**Figure 9**). The two particular values that need to be identified are the one associated with the end of the linear domain, i.e. the end of the first line; and the one corresponding to the minimal thermal value. In order to estimate a margin of error, a circular permutation of the thermal signal is applied, while maintaining the general trend of the curve: this is to redistribute the noise. Then, the $\ell 1$ trend-filtering is applied for each circular permutation step, and the new values of the junctions are stored. The process is repeated a hundred times, and the set of stored values for each junction is used to assess a margin of error (**Figure 10**). The particular stress values are summarized in **Table 3**.

Table 3 : Particular stress values for 0° specimens under quasi-static loads

	End of linearity	Thermal minimum
Particular stress values at 0°	80 MPa [60 – 90] MPa	330 MPa [290 – 360] MPa

The next step of the work consists in connecting these particular stress values to physical phenomena, especially to damage apparition. Two acoustic sensors were used to record each acoustic event occurring during the test. The evolution of the cumulative acoustic energy with the applied stress enables to identify a damage threshold, from which it starts increasing, as shown in **Figure 11**. Even if some isolated events appear from 120 MPa, the regular occurrence of damage, increasing the cumulative energy, has been determined shortly before 400 MPa for 0° specimens, corresponding to their thermal minimum. As the damage mechanism in a composite is assumed to be the same during a static or a fatigue test, the damage threshold assessed from static testing might be a good approximation of the fatigue limit. The fatigue limit is considered to correspond to the threshold stress from which damage occurs.

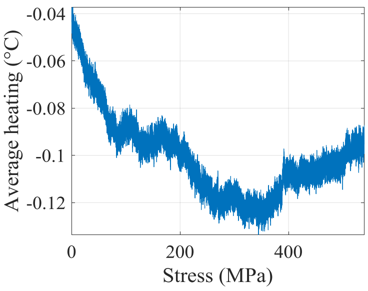

Figure 7 : Evolution of the spatial average heating of the sample with the stress during a static test

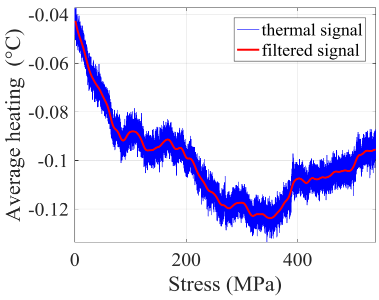

Figure 8 : Gaussian filtering of the thermal signal

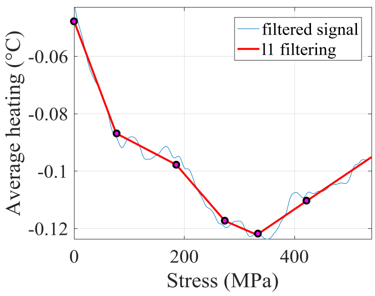

Figure 9 : Application of the $\ell 1$ trend filtering on the filtered signal

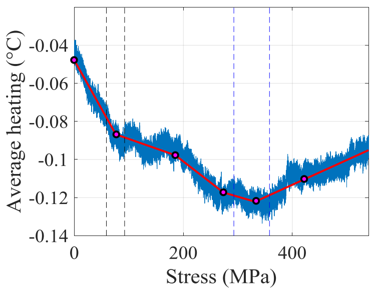

Figure 10 : Incertitude range for the two particular stress values

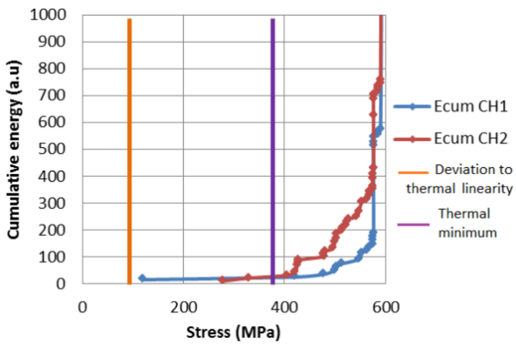

Figure 11 : Cumulative energy measured by two acoustic sensors during a quasi-static tensile test

As a conclusion, the literature on the thermal behaviour of thermoset composite materials under a static tensile test identified three thermal trends. Based on microscopic observations, variations in thermal change are related to the occurrence of damage, and the stress associated with the first thermal change is related to the fatigue limit, meaning that under this stress or under this limit, the material does not present micro damage. In our study, these three thermal trends also exist, however, it is more difficult to correlate thermal changes with microscopic observations because too little damage appears. The acoustic emission is then used to detect the appearance of the first damage events, which rather seem to concur with the second thermal transition: thus, the equivalence at the fatigue limit would be more at the thermal minimum. As for the first thermal change, it might be explained by the viscosity of the material, which is more important in a thermoplastic material than in a thermoset material. To confirm this hypothesis, it will be necessary to analyse the thermal behaviour of the material on specimens at 45°, for which the matrix is heavily stressed.

3. Self-heating tests campaign

3.1 Contribution of literature

Self-heating tests on woven carbon/thermoplastic composite material have already been carried out by [23] on a MTS 880-100kN machine equipped with hydraulic grips for rectangular specimens of nominal dimensions 250x30 mm, with a $[0]_8$ stacking sequence. **Figure 12Erreur ! Source du renvoi introuvable.** shows the final curve : three thermal trends are highlighted and the particular load values associated with the changes of thermal behavior are given in **Table 4**.

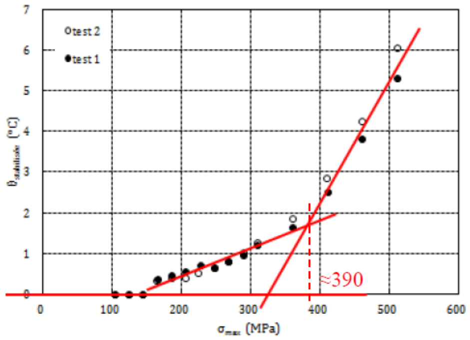

Figure 12 : Self-heating curve obtained by [23] on woven carbon/thermoplastic composite

Table 4 : Particular stress values for 0° specimens during self-heating tests

	1st intersection	2nd intersection
Particular stress values at 0°	≈150 MPa	≈390 MPa

It is very promising to note that these values are similar to those recorded during quasi-static tests (**Table 3**). It seems that viscosity effects are responsible for the first temperature variation, which tends to be validated by the acoustic emission analysis; as for the second intersection, it corresponds to first significant acoustic emission. The coupling of acoustic emission to heat transfert analysis highlights that the fatigue limit could be identified with quasi-static tensile tests or self-heating tests, which is a significant breakthrough as it would considerably reduce the duration and the cost of the experimental campaigns.

Conclusion

This study was focused on the thermal analysis of quasi-static tensile tests in order to predict the fatigue limit of a woven thermoplastic composite material.
The mechanical behaviour of the matrix (tested on 45°-specimens) has been shown to have a more ductile behavior (high deformation, low breaking stress) for specimens conditioned at high relative humidity. Then, for the specimens tested in the warp and weft directions, the properties are similar, due to the balanced nature of the woven composite. That justifies the study of only the warp and the shear directions in order to reduce the number of fatigue tests.
The thermal behavior of 0°-specimens has been shown to present three distinct domains, that might be compared to the three thermal trends detected on self-heating curves (**Figure 6**, **Figure 12**). This correlation is verified, analysed, and connected to physical phenomenon (variation of the matrix viscosity, beginning of damage).
The main conclusion of the paper is that the monitoring and analysis of the thermal behavior during quasi-static tensile tests might give an accurate approximation of the fatigue limit (beginning of micro damage). It has also been highlighted that the static test provides a conservative, accurate approximation of the fatigue limit by a combined heat transfer and acoustic analysis.

References

[1] C. Colombo, F. Libonati, F.Pezzani, A. Salerno, and L. Vergani, 'Fatigue behaviour of a GFRP laminate by thermographic measurements', *Procedia Eng.*, vol. 10, pp. 3518–3527, 2011.

[2] M. P. Luong, 'Infrared thermographic scanning of fatigue in metals', *Nucl. Eng. Des.*, vol. 158, no. 2–3, pp. 363–376, 1995.

[3] G. La Rosa and A. Risitano, 'Thermographic methodology for rapid determination of the fatigue limit of materials and mechanical components', *Int. J. Fatigue*, vol. 22, no. 1, pp. 65–73, 2000.

[4] C. Doudard, S. Calloch, F. Hild, and S. Roux, 'Identification of heat source fields from infrared thermography: Determination of "self-heating" in a dual-phase steel by using a dog bone sample', *Mech. Mater.*, vol. 42, no. 1, pp. 55–62, Jan. 2010.

[5] R. Munier, C. Doudard, S. Calloch, and B. Weber, 'Identification of the micro-plasticity mechanisms at the origin of self-heating under cyclic loading with low stress amplitude', *Int. J. Fatigue*, vol. 103, pp. 122–135, Oct. 2017.

[6] M. Poncelet, C. Doudard, S. Calloch, F. Hild, B. Weber, and A. Galtier, 'Prediction of self-heating measurements under proportional and non-proportional multiaxial cyclic loadings', *Comptes Rendus Mécanique*, vol. 335, no. 2, pp. 81–86, Feb. 2007.

[7] L. Gornet, O. Westphal, A. Krasnobrizha, P. Rozycki, C. Peyrac, and F. Lefèbvre, 'Rapid determination of the high cycle fatigue limit of a woven carbon fibe thermoplastic matric', *JNC 19*, 2015.

[8] L. Jégou, Y. Marco, V. Le Saux, and S. Calloch, 'Fast prediction of the Wöhler curve from heat build-up measurements on Short Fiber Reinforced Plastic', *Int. J. Fatigue*, vol. 47, pp. 259–267, Feb. 2013.

[9] J. Jedidi, F. Jacquemin, and A. Vautrin, 'Accelerated hygrothermal cyclical tests for carbon/epoxy laminates', *Compos. Part Appl. Sci. Manuf.*, vol. 37, no. 4, pp. 636–645, Apr. 2006.

[10] A. Malpot, F. Touchard, and S. Bergamo, 'Effect of relative humidity on mechanical properties of a woven thermoplastic composite for automotive application', *Polym. Test.*, vol. 48, pp. 160–168, 2015.

[11] M. Broudin *et al.*, 'Water diffusivity in PA66: Experimental characterization and modeling based on free volume theory', *Eur. Polym. J.*, vol. 67, pp. 326–334, Jun. 2015.

[12] A. Benaarbia, A. Chrysochoos, and G. Robert, 'Influence of relative humidity and loading frequency on the PA6.6 cyclic thermomechanical behavior: Part I. mechanical and thermal aspects', *Polym. Test.*, vol. 40, pp. 290–298, Dec. 2014.

[13] L. Greenspan, 'Humidity Fixed Points of Binary Saturated Aqueous Solutions', *Phys. Chem.*, vol. 81 A, 1977.

[14] L. Muller *et al.*, 'Experimental monitoring of the self-heating properties of thermoplastic composite materials', *Procedia Eng.*, vol. 213, pp. 183–191, 2018.

[15] C. Clienti, G. Fargione, G. La Rosa, A. Risitano, and G. Risitano, 'A first approach to the analysis of fatigue parameters by thermal variations in static tests on plastics', *Eng. Fract. Mech.*, vol. 77, no. 11, pp. 2158–2167, Jul. 2010.

[16] L. Vergani, C. Colombo, and F. Libonati, 'A review of thermographic techniques for damage investigation in composites', *Frat. Ed Integrità Strutt.*, 2014.

[17] F. Libonati and L. Vergani, 'Damage assessment of composite materials by means of thermographic analyses', *Compos. Part B Eng.*, vol. 50, pp. 82–90, Jul. 2013.

[18] L. Gornet *et al.*, 'Propriétés mécaniques en fatigue à grands nombres de cycles des composites carbone époxy', *Rev. Compos. Matér. Avancés*, vol. 25, no. 2, pp. 181–200, 2015.

[19] W.-T. Kim, M.-Y. Choi, Y.-H. Huh, and S.-J. Eom, 'Measurement of thermal stress and prediction of fatigue for STS using Lock-in thermography', *Proc 12th -PCNDT*, 2006.

[20] A. Salerno, A. Costa, and G. Fantoni, 'Calibration of the thermoelastic constants for quantitative thermoelastic stress analysis on composites', *Rev. Sci. Instrum.*, vol. 80, no. 3, p. 034904, Mar. 2009.

[21] S.-J. Kim, K. Koh, S. Boyd, and D. Gorinevsky, 'l1 Trend Filtering', *SIAM Rev.*, vol. 51, no. 2, pp. 339–360, May 2009.

[22] A. Moghtaderi, P. Borgnat, and P. Flandrin, 'Trend filtering : Empirical Mode Decompositions versus l1 and Hodrick-Prescott', *Adv. Adapt. Data Anal.*, vol. 03, no. 01n02, pp. 41–61, Apr. 2011.

[23] C. Peyrac, T. Jollivet, N. Leray, F. Lefebvre, O. Westphal, and L. Gornet, 'Self-heating Method for Fatigue Limit Determination on Thermoplastic Composites', *Procedia Eng.*, vol. 133, pp. 129–135, 2015.