Fatigue properties determination of carbon fiber reinforced epoxy composite by self-heating measurements

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Abstract:

Polymers are one of the most important types of engineering materials used with reinforcing fibers in various industries in order to improve the mechanical properties. One of the most widely used composites is the carbon fiber reinforced plastic owning excellent mechanical properties such as high specific stiffness and specific strength. The fatigue performances imposed by standards have to be performed by means of experimental campaigns in laboratory on specimens. Classical procedures to evaluate the fatigue limit of material involve expensive and time-consuming tests because of the high number of specimens being tested. In the last decades, with an aim to reduce testing times and costs of fatigue tests, different techniques and methods have been proposed in order to study the various damage phenomena rapidly and consistently. Infrared thermography is considered as a promising method to investigate the fatigue behavior focusing on the metals and fiber reinforced plastic. The identification procedure of this method is based on the use of self-heating measurements and validate by comparing the prediction of the S-N curves given by the model and experimental fatigue results. In this study the thermographic technique based on surface temperature variation was used to assess the fatigue behavior of carbon fiber reinforced epoxy. To fabricate the composite samples, four layers of woven carbon fibers with an equal weight ratio of epoxy resin were used. The quasi-static tensile tests were conducted to determine the stress levels in classical and self-heating fatigue tests. The load-controlled fatigue tests were conducted with a stress ratio of 0.1 and loading frequency of 10 Hz. In fiber reinforced epoxy under cyclic loading the main mechanisms causing energy dissipation are attributed to the matrix cracking, fibers fracture, and interface cracking/friction among others. According to the fatigue tests analysis (evolution of the hysteresis area and Young's modulus) and the *results obtained from self-heating tests, the energy dissipation mechanisms were discussed. It is worth noting that the obtained results from self-heating measurements can propose a good agreement with those attained by the classical methods. In other words an empirical relation could be considered between self-heating and classical fatigue results. Moreover with comparing the dissipated energies and stabilized temperatures in the self-heating measurements, there was a direct relation between them. Finite element simulation using commercially available ABAQUS software was also performed to model the temperature variations in the specimens subjected to cyclic loadings.*

Key words: Polymer composites, Carbon fibers, Self- heating measurement, Fatigue assessment, Staircase method, Finite Element Simulation

1 Introduction

Polymers are one of the most important types of engineering materials used with reinforcing fibers in various industries in order to improve the mechanical properties. One of the most widely used composites is the carbon fiber reinforced plastic (CFRP) owning outstanding mechanical properties such as high specific stiffness and specific strength [1]. In addition, epoxy resin is extensively used as a matrix in fiber reinforced plastics (FRP) due to its high strength and modulus, low contraction, excellent heat resistance, and high chemical and corrosive resistance [2]. Fatigue could be one of the primary reasons for failure in structural materials such as CFRPs. The nature of fatigue damage in CFRPs is complicated and is totally different from those of isotropic materials. The damage states are mostly related to the heterogeneity and anisotropy which leads to the formation of different stress levels [3]. To evaluate the fatigue limit of the material, classical procedures involve expensive and time-consuming tests because of the high number of specimens being tested. In the last decade, in order to study the various damage phenomena rapidly and with an aim to reduce testing times and costs of fatigue tests, different techniques such as, X-ray radiography, acoustic emission, X-ray micro-computed tomography and infrared thermography techniques (IRT) have been applied. Infrared thermography is considered as a promising method to investigate the fatigue behavior, focusing on the metals and fiber reinforced plastic (FRP). In the case of cyclic loading, the energy dissipation leads to a self-heating phenomenon. In other words, the main part of the mechanical energy dissipates in the form of heat [4]. On the other hand, during a cyclic loading and depending on stress level and loading frequency, the self-heating effect can be a very dangerous phenomenon which occurs in polymeric composites. This effect might intensify a degradation process and leads to a failure of composite [5]. Moreover thanks to the thermal maps from thermographic techniques it is possible to monitor the damage evolution during the tensile test.

In the case of cyclic loading in polymer matrix composite (PMC) materials, the main mechanisms causing damage and energy dissipation are attributed to the matrix cracking, fiber fracture, and interface cracking/friction among others [6]. To assess the damage state in PMC materials during the fatigue tests, the evolution of the hysteresis area and modulus could be considered. under cyclic loading, a three-stage modulus or stiffness degradation is reported [7]. Moreover, a typical three-stage trend is reported in surface temperature measurement. During stage I, the temperature variation is due to the occurrence of micro-cracks in multiple locations in the matrix, frictions (fibers/fibers and fibers/matrix), debonding at the weak interface between fibers and matrix, and breaking of some fibers with low strength. This stage typically consists of 10–20% of the entire lifespan of the specimen. In stage II, the temperature reaches a balance that is due to the saturation in the crack density and damage. The existing cracks grow toward the fiber/matrix interface where cracks cannot cross the high strength fiber and begin to bifurcate in two directions. This event results in the initiation and propagation of fibers/matrix interfacial debonding and delamination. This stage consists of 70–75% of the entire lifespan of the specimen. Stage III is the last stage of damage growth which leads to a sudden increase in temperature. In this stage, all the damage modes would be developing rapidly in a fast-decreasing stiffness of the laminate. As the stress state reaches a critical value, fracture of the fibers and laminate would be initiated. In addition, this stage consists of 10–20% of the entire lifespan of the specimen. The severity of each stage and the total amount of damage depend on laminate stacking, material properties and loading conditions [6].

This study is focused on the self-heating response and the fatigue properties of carbon fiber reinforced epoxy. More precisely, this work aims at understanding the fatigue behavior of carbon fiber reinforced epoxy via self-heating method and studying the energy dissipation in different stages of self-heating test. According to the fatigue tests analysis (evolution of the hysteresis area and Young's modulus) and the results obtained from self-heating tests, the energy dissipation mechanisms could be investigated. The results obtained provide us to make a contribution towards determining the relation between the results obtained from self-heating tests and those obtained from classical fatigue method.

2 Experimental procedure

2.1 Studied material

Standard composite samples were fabricated by hand lay-up method [8]. To fabricate the composite samples the Epon 828 epoxy resin with a polyamine hardener (from Mokarrar Engineering Materials Co.) was used as the matrix. Four layers of woven carbon fibers (from Mokarrar Engineering Materials Co.) with an areal density of 200 g/m^2 and equal weight ratio of epoxy resin were used. Moreover, a flat fixture was used to prepare the composite panel for the sake of applying uniform pressure to them. The samples were cured at room temperature for one day and post-cured at 80 ˚C for 120 min. Finally, according to standard ASTM D 3039, the prepared composite flat panels were cut by the abrasive water jet cutting technique with the dimensions of 200 mm × 25 mm × 2.2 mm (length × width × thickness). To avoid gripping induced failure at the ends of the samples, all of them were end tabbed with 2 mm thickness carbon fiber reinforced epoxy end-tabs, leaving a gauge length of 100 mm.

2.2 Experimental setup

The general testing set up is presented in Fig. 1. To determine the ultimate tensile strength (UTS) and assess the mechanical behavior of composite samples, the quasi-static tensile tests were carried out at room temperature using INSTRON servo-hydraulic testing machine with the capacity of 100kN testing machine. The tensile test was conducted according to ASTM D3039, with a displacement rate of 10^{-3} mm/s (10^{-5} 1/s). The tensile strain was measured using a digital image correlation (DIC) system and extensometer with 50 mm gage length. GOM 5M (DIC system) with 2D or 3D displacement measuring results was utilized for in-situ measurement of surface strain and surface displacement of composite samples. This system has a strain accuracy of 0.01% and a strain measuring range of 0.02% up to >100%. In addition, the cameras of this system have a resolution of 2448×2050 pixels. The surface displacements of the composite specimen were deduced by analyzing the random speckles pattern, which was created with black and white paints. Images were acquired with a frequency of one image per second and the post-processing of the obtained results was carried out by ARAMIS v.6.3.1 software.

The load-controlled fatigue tests were performed at room temperature, by means of an INSTRON servo-hydraulic testing machine with the capacity of 100kN. This test was conducted with a stress ratio of 0.1 and a loading frequency of 10 Hz. The staircase method was utilized with stress amplitude levels starting from 120 MPa and the stress increment of 5 MPa. The infrared thermography technique was employed to analyze the energy dissipation mechanisms in self-heating test and determine the high cycle fatigue strength (HCFS) of the carbon fiber reinforced epoxy composite. An 8300HP series infrared camera (from Infra Tec) with a maximum frequency of 200 Hz was used to *in-situ* measurement of temperature evolution at the surface of the composite sample. In order to avoid the influence of the environment on the measurements and provide a relatively thermal insulated system, special attentions were considered (Fig. 1b).

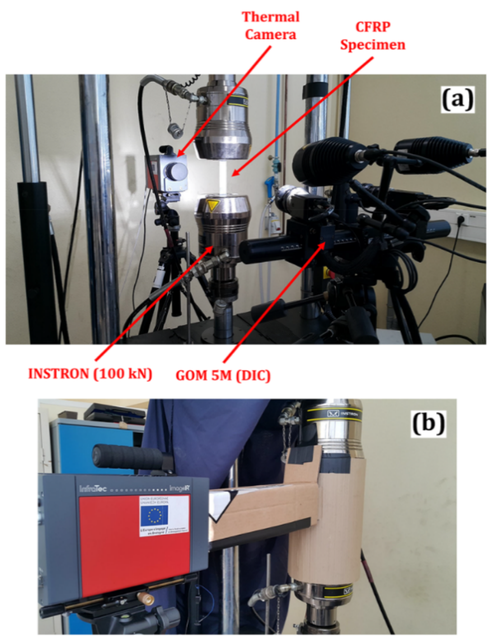

Fig. 1. Experimental set up: (a) view on the clamped sample with measurement devices, and (b) special attention for avoiding the influence of the environment on the measurements.

2.3 Self-heating test

A self-heating test is the realizing of a successive series of cyclic loadings (or loading blocks) for the same specimen, with a constant stress ratio and increasing stress amplitude, as illustrated in Fig. 2. Each loading block consists of two parts. In the first part, a certain number of loading cycles is applied and temperature variations are recorded. In the second one, the cyclic loading is stopped in order to thoroughly cool down the specimen up to the initial temperature (T_0). The evolution of the temperature during one loading step is shown in Fig. 2. The self-heating curve can be obtained by considering three different parts of temperature evolution which is recorded during each loading block; (I) primary temperature rising rate, (II) the stabilized temperature and (III) the cooling rate after a sudden interruption of the cycling.

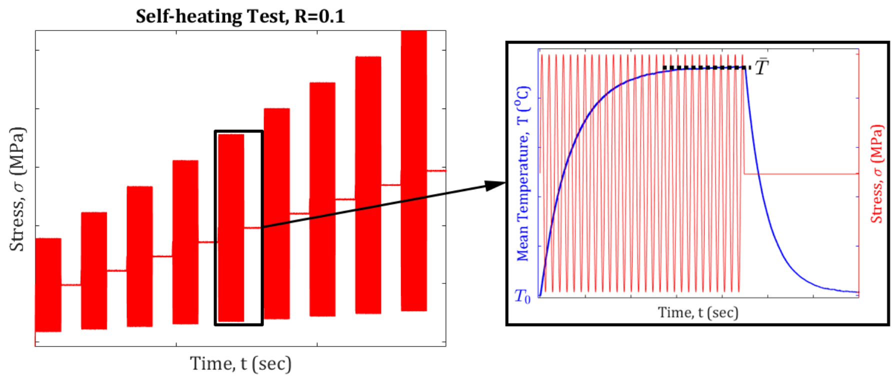

Fig. 2. Successive series of cyclic loadings with constant stress ratio and increasing stress amplitudes.

In this study, the self-heating test was performed with the same parameters of the traditional fatigue test (i.e., R=-1 and f=10 Hz). Table 1 shows the details of load levels in 18 steps. The stress amplitudes were chosen between 70 MPa and 220 MPa. For each block, 9000 cycles were applied and at the end of each step, the test was paused for 10 minutes in order to cool down the specimen. The stabilized temperature was considered to plot the self-heating curve.

Table 1. Stress levels in self-heating test

Step Number	σ_{max} [MPa]	σ_{Amp} [MPa]
1	155.55	70
2	177.77	80
3	200	90
4	222.22	100
5	244.44	110
6	266.66	120
7	288.88	130
8	311.11	140
9	333.33	150
10	344.44	155
11	355.55	160
12	366.66	165
13	377.77	170
14	400	180
15	422.22	190
16	444.44	200
17	466.66	210
18	488.88	220

3 Results and discussions

3.1 Mechanical behavior

In order to get additional information and analyze the damage phenomenon from an energetic point of view, the thermal camera was utilized to monitor all the tensile tests. In addition, understanding the mechanical behavior of the material was necessary to determine the load levels in self-heating measurements. Fig. 3 shows the temperature and stress trend for the longitudinal specimens with [0˚] stacking sequences of glass fibers [9]. As can be seen in this figure, the thermal profile can be characterized by three distinct stages. The first stage of the thermal profile is approximately linear. This linear temperature decrease is due to the thermoelastic effect that implies the elastic behavior of the material with no created microscopic cracks. A variation in the linear regime of thermal trend indicates the end of the elastic area of the material. According to stress-strain curve (Fig. 4), in the case of our material the elastic behavior is up to 360 MPa and stress value of damage initiation (σ_D) is 360 MPa that showed a good agreement with result obtained from temperature measurement. After damage stress (σ_D) the microscopic damages initiate and a balance between thermoelastic effect and dissipated energy due to small micro-damages could be considered. A mild temperature evolution in the second stage supports this hypothesis. A sudden increase in temperature is visible in the last stage of the thermal trend which is due to release of stored energy and macroscopically damage propagation.

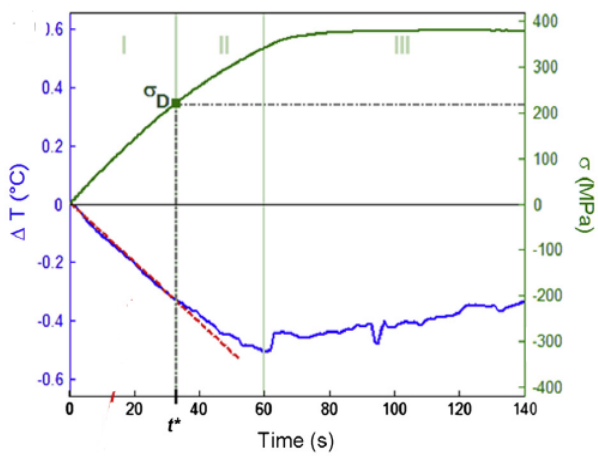

Fig.3. Temperature and stress trend during tensile test [9].

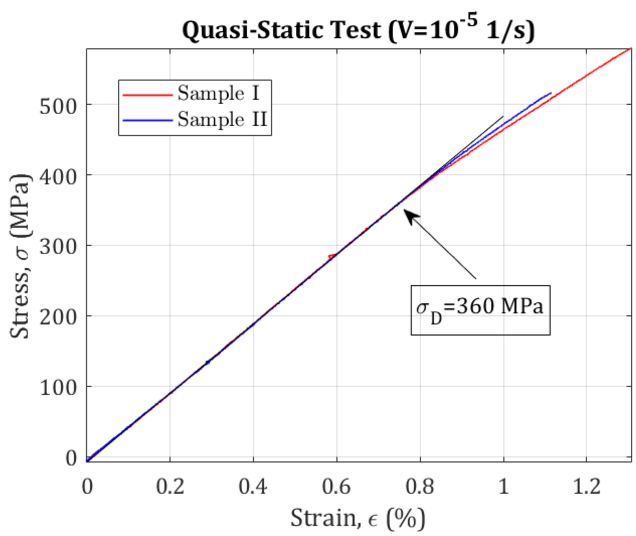

Fig. 4. Stress-strain curves obtained from quasi-static tests.

By applying the loading–unloading test, the hysteresis area can be considered to identify the damage progression which the area bound by the hysteresis loop shows a loss of stiffness in the respective cycle [10]. The results obtained from the loading–unloading quasi-static tests with certain maximum stress levels exhibited negligible hysteresis area and consequently little energy dissipation under low stress levels (σ_{Max}<σ_D). In fact, the slight area of the hysteresis loop showed the elastic behavior and an insignificant viscous effect of the material. Unlike in higher stress level ($\sigma_{Max}<\sigma_D$) the large hysteresis area implied the higher dissipated energy and viscoelastic behavior of the material. The variation of hysteretic behavior of the material was due to the creation of various damage phenomena in carbon-reinforced epoxy such as matrix cracking, matrix deformation, debonding and delamination. These phenomena degrade the elastic properties of the material in stress levels higher than damage stress. These results of the mechanical behavior of carbon fibers/epoxy composite show a good agreement with those obtained from the thermographic analysis.

3.2 Self-heating measurement

Using stabilized temperature from self-heating measurements, the temperature evolution is plotted as a function of the stress amplitude in logarithmic scale (Fig. 5), which three regimes of temperature evolution are visible.

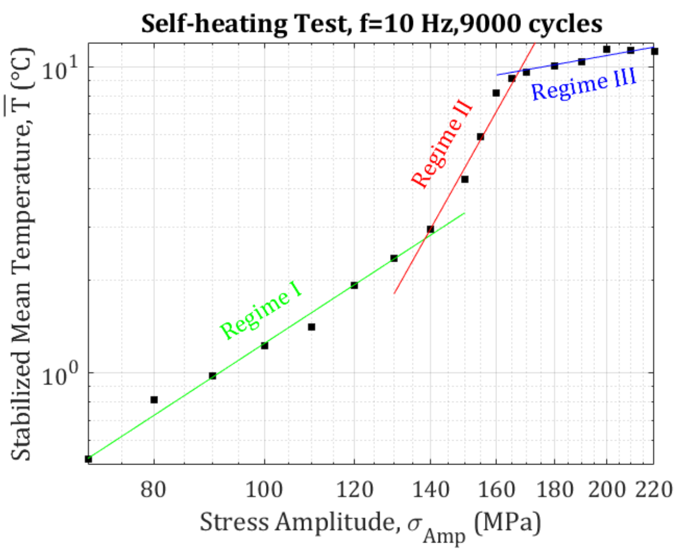

Fig. 5. Self-heating curve in logarithmic scale.

The intrinsic energy dissipation and temperature evolution in composite materials during cyclic loading is attributed to the local matrix deformation and the damage effect. During regime I (Fig. 5), the temperature increase could be due to the initiation of micro-cracks in the locations with stress concentration. In the case of regime II, with increasing the stress amplitude the temperature raises with a higher rate comparing to the first regime. It implies that a transition in the damage state and damage propagation on different size scales has occurred. However, under these stress levels, the critical cracks and critical damage state have not attained and the residual strength of the composite sample will not be higher than the maximum applied stress. Therefore, the fatigue failure cannot occur under these stress levels. In the third regime (III), comparing to the second regime, a decrease in the rate of temperature increase occurs. This implies that the rate of energy dissipation is decreased which is a result of reducing in damage progress rate. As it is visible at the third regime, the mean stabilized temperature is in the range of 8-11 ˚C (the spots with significantly higher temperature can be existed). The temperature elevation can lead to the softening and degradation of the polymer matrix, which would cause the local viscous energy dissipation. In addition, temperature elevation impresses the dominated energy dissipation mechanisms. Hence, it can be concluded that the temperature elevation in the third regime is mainly due to local viscous heating of the polymer matrix and partially due to damage initiation and propagation. In the case of our material, the second transition point (the stress amplitude of 167 MPa) was considered as the fatigue limit. For the stress amplitude above 167 MPa, the damage state attains the critical limit and can cause failure in the particular number of cycles.

3.3 Classical fatigue tests

In order to ratify the results of self-heating measurements, the classical fatigue test was carried out using the staircase method. Staircase method was applied to obtain the mean fatigue limit of studied material. As the results of the staircase method revealed, there is a fatigue limit for the carbon fiber reinforced epoxy composite. From the Dixon-Mood method [11] and using the following equations, the mean fatigue limit and the standard deviation were calculated.

Define, $A = \sum_{i=0}^{i_{max}} m_i$, $B = \sum_{i=0}^{i_{max}} i m_i$, $C = \sum_{i=0}^{i_{max}} i^2 m_i$ (1)

$$\mu = S_0 + d.\left(\frac{B}{A} \pm \frac{1}{2}\right) \quad (2)$$

$$\sigma = 1.62.d\left(\frac{A.C - B^2}{A^2} + 0.029\right) \text{ if } \frac{A.C - B^2}{A^2} \geq 0.3 \quad (3)$$

or, $\sigma = 0.53.d$ if $\frac{A.C - B^2}{A^2} < 0.3$ (4)

Where, μ is mean fatigue limit, σ is standard deviation, S_0 is the lowest stress level for the less frequent occurrence, d is the stress increment, i is the stress level numbering and m_i is the number of samples at stress level i. The summations required for the Dixon-Mood equations are given in Table 2.

Table 2. Summations for illustration of the Dixon-Mood method.

i	**m_i**	**$i \times m_i$**	**$i^2 \times m_i$**
0	1	0	0
1	2	2	2
2	1	2	4
Σ	4	4	6
	A	**B**	**C**

In the case of our study, S_0 and d were 160 MPa and 5 MPa, respectively. So the mean fatigue limit and the standard deviation were calculated 162.5 MPA and 4.28 respectively. This evaluated value is in complete agreement with those obtained from the self-heating measurement.

A plot of successive stress-strain curves for the cycles during the various stages of fatigue test for the specimen loaded at 150 MPa stress amplitude is shown in Fig. 6. As indicated by the areas within the stress-strain ellipses, the specimen shows relatively higher energy dissipation during the 10^5th cycles. The large area of stress-strain ellipses shows a higher damage amount and viscous effect during cycling. Hence it can be deduced that at middle stages (10^5th cycles) the amount of damage is relatively higher than the primary and final stages. This fact could be confirmed by the strain ratcheting occurred during cycling which is mainly due to damage development. As illustrated in the plot of Fig. 6 the strain ratcheting at the middle stages (between 10^4th and 10^5th cycles) is notable with compare to the values at the terminal stages (between 10^6th and 2×10^6th). It implies that the higher amount of damage has occurred at the middle stages.

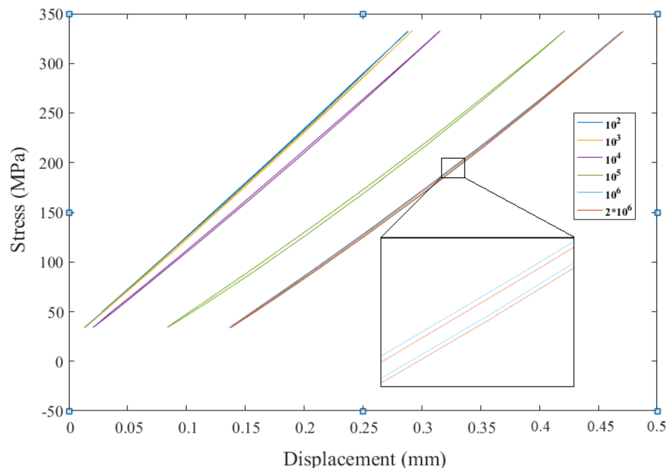

Fig. 6. Stress-strain response during fatigue test with stress amplitude of 150 MPa.

4 Modeling the self-heating phenomenon using FE simulation

A finite element simulations using ABAQUS software was performed in order to simulate the self-healing phenomenon in the specimens. Using a thermo-mechanical coupled analysis, the generated temperature in the specimens as a function of the applied stress can be obtained. Experimental tensile properties of the composite specimens were used as the input material data for the analysis. In order to validate the model, the obtained numerical results were compared with the experimental data. Employing the verified model, a parametric study on the effective parameter can be performed.

In this regards, a representative volume element (RVE) which consists of two phases (inclusion and elastic surrounding matrix) was considered. Coupled thermo-elastic analysis was conducted on the specimens for the each load amplitude. The typical variation of the temperature distribution in the specimens is shown in Fig. 7. The temperature in the specimen increases as a function of the applied stress and time. For the given RVE, the variation of the temperature with time is demonstrated in Fig. 8. According to Fig. 8, after an initial rise, the temperature reaches a stable value during the time. This stabilized temperature can be obtained for each case according to the stress amplitudes and frequencies.

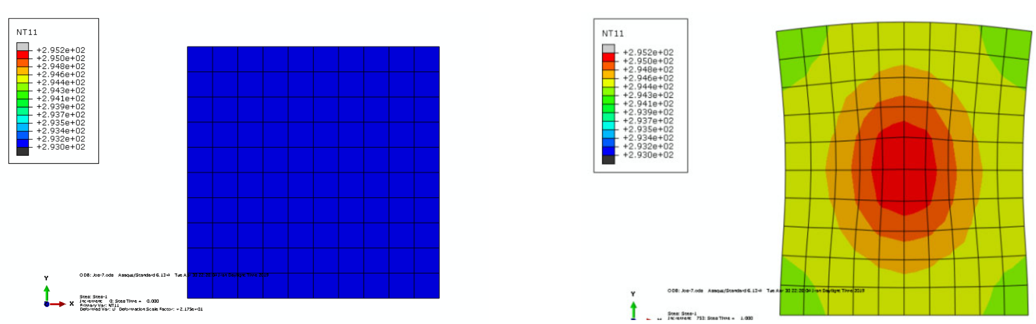

Fig. 7. Temperature distribution within the RVE a) before applied load and b) after application of the stress.

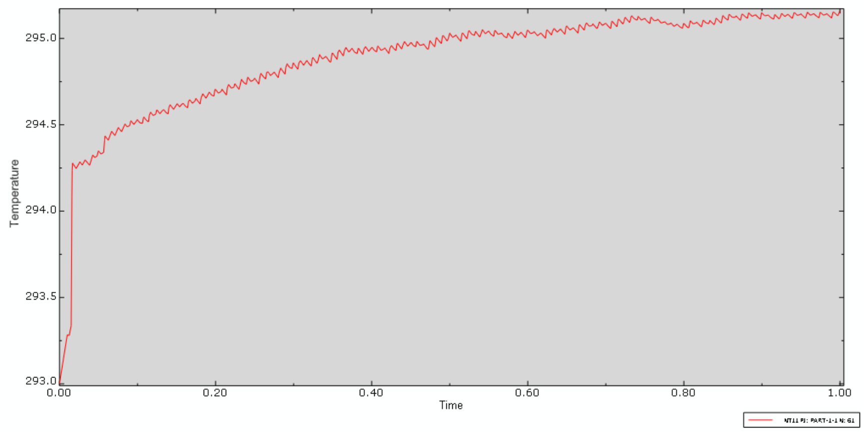

Fig. 8. Variation of the generated temperature with time in RVE.

5 Conclusions

In the present study, the self-heating response and the fatigue properties of carbon fiber reinforced epoxy were experimentally studied. According to the fatigue tests analysis (evolution of the hysteresis area and Young's modulus) and the results obtained from self-heating tests, the energy dissipation mechanisms were investigated and the relation between the results obtained from self-heating tests and those obtained from classical fatigue tests was empirically obtained. The salient observations from these experiments can be summarized as follows:

1- In the case of quasi-static tests, the elastic behavior was up to 360 MPa and stress value of damage initiation (σ_D) was 360 MPa that showed a good agreement with the results obtained from temperature measurements.
2- The self-heating measurements demonstrated three regimes of temperature evolution which the second transition point was considered as the fatigue limit. The fatigue limit obtained from the self-heating measurement was in complete agreement with those evaluated from classical fatigue tests.
3- Classical fatigue results showed that at middle stages of fatigue test, the amount of damage is relatively higher than the primary and final stages.

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