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# **Electrical resistance variation during tensile and self-heating tests conducted on thermoplastic polymer-matrix composites**

**W. Harizi<sup>\*)</sup>, R. Azzouz, A.T. Martins, K. Hamdi, Z. Aboura, K. Khellil**

*Alliance Sorbonne Université, Université de Technologie de Compiègne, CNRS, Laboratoire  
de Mécanique Roberval FRE 2012, Dept IM, CS 60319, 60203 Compiègne Cedex, France*

<sup>\*)</sup> Corresponding author: [walid.harizi@utc.fr](mailto:walid.harizi@utc.fr)

## Abstract

The study investigates the electrical resistance response of polymer-matrix composites (PMC) with thermoplastic PPSU (polyphenylsulfone) resin reinforced by twill 2/2 carbon fabrics during tensile and self-heating tests. During the tensile tests, the electrical resistance increases at the beginning of the tests, but drops due to the fibers/matrix debonding and subsequently amplifies abruptly until the failure of the specimens owing to the breaking of the carbon fibers. The evaluation of the stability of measurements of temperature and electrical resistance was performed during the self-heating tests. The originality of the study lies in the assessment of the fatigue limit of thermoplastic composites using two macroscopic magnitudes: temperature and electrical resistance. These two quantities provide a fairly fast estimation of the fatigue limit with a slight percentage difference of 21%.

**Keywords:** Polymer-matrix composites; Carbon fibers; Electrical resistance; Tensile test; Self-heating test; Fatigue limit.

## 1. Introduction

Polymer-matrix composites (PMC) are widely used in several applications such as aerospace, naval, automotive, civil [1] and represent some alternatives to conventional materials like metals and ceramics [2]. This recognition is mainly due to their remarkable mechanical properties such as high tensile and impact strength-to-weight ratios, high stiffness-to-weight ratio, long fatigue life, corrosion and wear resistance, thermal insulation with glass fibers, electrical conductivity with carbon fibers, adding the raw material availability, a lower cost, both recyclability and biodegradability properties[1-3].

Despite the outstanding properties mentioned for the PMC composites, fatigue loadings may be responsible for many failures of structural components made by PMC even before reaching the Yield stress. Likewise, PMC materials are heterogeneous, anisotropic and their mechanical properties depend on their manufacturing process and stacking sequence, which justifies the lack of a specific model to estimate their fatigue life [4]. Fatigue limit consists in the high threshold value which a material subjected to cyclic loading is estimated to have an infinite life [5]. This limit is conventionally determined by the Wöhler (or S-N) curve and depends on several factors such as cyclic loading frequency, the loading ratio (minimal-to-maximal stresses ratio) and average stress. To perform a S-N curve, many specimens are tested under cyclic loading until failure at different average stress levels to acquire the number of load cycles to fatigue failure. S-N curve is extremely time-consuming because some specimens can endure for weeks and sometimes not even fail. Additionally, a large number of samples must be tested (a minimum of 20 specimens) to develop a reliable curve [6]. The mentioned drawbacks about the S-N method leads to the need for finding an alternative procedure faster and less complex than the previous to estimate fatigue limit. Many researchers such as *Locati* [7-8] and *Prot* [9-10] focused on the energy dissipation until *Catalbiano et al.* [11] followed by *Risitano and La Rosa* [12] suggested a thermographic

methodology to investigate the energy dissipation by observing the material's surface during cyclic loading. Thereafter, this methodology has been used to obtain a rapid estimation of the fatigue endurance limit and has proven its accuracy for the conventional and isotropic materials. Firstly, this technique was applied to stainless steel and metal alloys using the self-heating phenomenon to describe a model for the energy dissipation and estimate fatigue limit [13-18]. The research of this approach for other materials such as polymers occurred subsequently [19-20], such as the model developed by *Ovalle et al.* [19] for describing the self-heating of elastomeric materials, which was validated experimentally. The self-heating method has also been applied on thermosetting [21-22], ceramic-matrix composites [23] and especially thermoplastic composites [24] due to the fast growth in the application of these materials as structural components. The thermoplastic composites are still considered a challenge owing to their distinctive high viscoelasticity which can be affected by the average load, loading ratio and frequency of fatigue tests [25]. The viscoelasticity can generate a specific change in the mechanical behavior and complicates the development of an accurate model able to predict the fatigue behavior [26]. During the proposed self-heating tests the specimen is also subjected to cyclic loading. However, rather than applying a unique value of stress amplitude throughout the cyclic loading until the specimen failure for each batch of tests that regards the stress amplitude, a pre-defined successive series of cycles for different increments of average stresses are applied on a single sample until the failure, by keeping the same stress ratio and frequency [27-30]. These successive series of cycles of same load, designated as blocks, induce heating in the material by the energy released under tests, mainly in the first cycles until its stabilization through the block. Indeed, this variation of temperature is only visible if the heat is dissipated on the specimen's surface. At the end of each block, the sample is unloaded and maintained until it returns to its initial temperature. The variation of the stabilized temperature according to the maximum stress illustrates a

parabolic trend with two linear asymptotes whose intersection is the fatigue limit. It has been already proven that the sudden change of heating rate is associated to the excess of energy dissipation, which can be related to the viscous effect or the initiation of microdamages. These physical phenomena affect significantly the fatigue properties [31]. Some studies [13, 24] compared the endurance limit determined by the Wöhler method and the self-heating approach on the same material system. They reported a good agreement between the values found by the both methods and a close association involving the temperature variation and the fatigue limit under a cyclic loading [24]. Self-heating method presents the advantage to achieve a rapid estimation of the fatigue limit as well as due to the very limited number of test specimens required for performing the procedure. However, the measurements of the temperature variation by infrared thermography (IRT) method need to place the IR camera in front of the loaded structure and at some distance, this is not necessarily possible on some structures in operation and can generate more clutter. Furthermore, the thermal scene must be isolated using an obscuring insulator curtain to avoid external disturbance that can alter the temperature measurement [32-33]. Thus, recent studies have investigated the electrical resistance response of carbon fiber-reinforced polymer matrix composites under cyclic tests especially because the conductive nature of the carbon fibers. It was shown that electrical resistance change could be an indicator of health status of PMC structures [34-37]. So, the internal damage generated in the material can be detectable and defined by some macroscopic physical quantities such as temperature variation and electrical resistance change.

The originality of this paper consists in the using of electrical resistance measurements during self-heating tests on continuous carbon fiber polymer-matrix composites for a rapid estimation of their fatigue limit. After a presentation of the studied material and the experimental procedures used during tensile and self-heating tests (section 1), the results will

be presented and discussed in section 2 with a comparison of the two values of fatigue limit obtained using temperature and electrical resistance.

## 2. Materials and experimental procedures

### 2.1. Materials

The composite materials utilized in this study was manufactured with eight plies of twill 2/2 carbon fabrics (Tenax HTA 3K with 200 tex) and 16 sheets of PPSU (polyphenylsulfone) polymer (Radel R5000, Ajedium films) with a thickness of 125  $\mu\text{m}$ . The film stacking method and thermo-stamping process consolidated the materials with a stacking sequence of (0/90)<sub>4S</sub> (Fig. 1a, [37]). The thermo-stamping process subjected the preforms at 355°C under 30 bars during 25 minutes, in order to obtain, as far as possible, planar plates with a uniform thickness of  $3^{\pm 0.1}$  mm. The fiber volume and porosity fraction were of  $40^{\pm 2}\%$  and  $3^{\pm 1}\%$  respectively (Fig. 1b). All tensile specimens were cut from the same plate to reduce the variability of the material properties (five samples for the static tensile tests, six for self-heating tests and five specimens conducted on conventional fatigue test until  $2.10^6$  cycles to verify the fatigue limit evaluated by the self-heating method using stabilized temperature and electrical resistance) in the dimensions as detailed in Fig. 1c. Glass-epoxy tabs were bonded to the specimens employed for the mechanical tests as shown in Fig. 1c. They were built by a twill tape 2/2, with  $\pm 45$  oriented fibers, placed on the outer of the stack according to the ISO 527-4 standard [38].

[Insert Fig. 1]

### 2.2. Experimental tests

Static tensile tests and self-heating tests were carried out in this study.

#### 2.2.1. Tensile test

The tests were performed at a cross-head speed of 2 mm/min until the failure of the specimens using an electrical tensile machine (INSTRON 1186) with a load cell of 100kN. An extensometer with a gauge length of 10mm attached to the specimen surface measured the strain. Two broadband acoustic transducers (Micro-80, from Physical Acoustics Corporation) with a 40 dB pre-amplification were coupled with silicone grease on the same face of the samples at a distance of 110mm between their centers, symmetrically arranged about the center of the length of the specimen. The temperature variation on the specimen's surface under loading was acquired by an IR thermographic camera FLIR A320 with a pixel resolution of 640(H) x 480(V) and a thermal sensitivity of 0.05°C. The camera was positioned 75 cm from the specimen to monitor the thermal scene with a sampling frequency of 1 frame/s (Fig. 2). Moreover, the experimental setup was isolated using an obscuring curtain to avoid external disturbance that can alter the temperature measurement.

[Insert Fig. 2]

The real-time measurements of electrical resistance during all tensile tests employed the 4-wires sensing method by a digital multimeter KEYSIGHT B2901A. It consists of two wires (continuous yellow lines in Fig. 3) for the current-carrying and the second pair of wires (red dashed lines) for the voltage-sensing, which are responsible for the measurements of electrical resistance. The 4-wires method diminishes the disturbances created by the contact with the specimen at the moment of current injection (Fig. 3). The present work performed the electrical measurements with an imposed current of 10mA and measuring rate of 20 Hz for all specimens. The distance between the wires for the current supply and those for voltage-sensing are 70mm and 40mm respectively as shown in Fig. 3. It has been verified that the low intensity of 10mA does not cause heating of PPSU/carbon composite.

[Insert Fig. 3]

Electrical resistance variation  $\Delta R/R_0$  is calculated using Eq.(1) where  $R_0$  is the initial resistance before loading the specimens and  $R_i(t)$  is the instantaneous resistance obtained throughout the tests.

$$\frac{\Delta R}{R_0}(t) = \frac{[R_i(t) - R_0]}{R_0} \times 100 \text{ [%]} \quad (1)$$

### 2.2.2. Self-heating test

The tests were conducted on the servo-hydraulic fatigue testing machine Instron 1343 with a load cell of 250kN (Fig. 4a). The same apparatus employed for the characterization during tensile tests, which consists of the electrical measurements, IRT camera and extensometer were employed for the self-heating tests. The electrical measurements utilized the same method and distances between wires than those for the tensile tests, with a measuring rate of 0.5Hz. The IRT camera (FLIR A320) was placed at 75cm from the specimen with a sampling frequency of 0.1Hz (one frame every 10 seconds). The experimental device was also covered with an obscuring curtain. The test room is equipped with two air conditioners guaranteeing a fixed external temperature set at 20 °C for all tests. Because the heating of the hydraulic jaws during cyclic loading, the average temperature variation  $\Delta T(t)$  at the center of the specimens was promptly adjusted considering the instantaneous temperature of the two jaws using Eq.(2) [27-30].

[Insert Fig. 4]

$$\Delta T(t) = T(t) - T(0) - \frac{T_u(t) + T_l(t)}{2} + \frac{T_u(0) + T_l(0)}{2} \text{ [°C]} \quad (2)$$

Where  $T(0)$  is the initial average temperature in the central zone of the PMC specimen and,  $T_u(t)$  and  $T_l(t)$  are the instantaneous temperatures of the upper and lower jaws respectively (Fig. 4b). Eq.(2) allows obtaining the best stabilization of the measured temperature per block.

Fig. 5 schematizes the loading method utilized in the present study that composes of several blocks of cyclic loading. The values of the parameters concerning the loading blocks are shown in Table 1. The Self-heating tests consisted of cyclic tension-tension loading starting from a maximum load of 2550N (i.e., 34MPa, a value much lower than the Yield limit) that develops with incremental stress of 10MPa at every block until the failure of the specimen. At the beginning of each block, the specimen is loaded at a cross-head speed of 2mm/min until reaching the mean load ( $F_{mean}$ ). Then, 5000 cycles are applied with a frequency of 2 Hz and load ratio ( $R$ ) of 0.1. During this step, the stabilized temperature and stabilized resistance are monitored by IRT camera and the digital multimeter respectively. At the end of the block, the specimen is unloaded and maintained in this state during 6 minutes to regain its initial temperature. Afterward, the load increases again with the next block with the incremental addition of 10MPa to the ulterior maximum stress (Fig. 5 and table 1).

[Insert Fig. 5]

[Insert Table 1]

### 3. Results and discussions

#### 3.1. Tensile tests

The mean values of the tensile properties obtained from five PPSU/carbon composite specimens are exhibited in Table 2.

[Insert Table 2]

Fig. 6a presents the typical behavior of electrical resistance variation ( $\Delta R/R_0$ ), obtained from Eq.(1), temperature change  $T - T_0$  (the difference between the instantaneous temperature  $T$  and the initial temperature at zero stress  $T_0$ ) and stress *versus* time of the specimens under tensile tests. Fig. 6b assists the analysis with the curve of acoustic emission (AE) cumulative energy on the place of the temperature curve in Fig. 6a. The typical behavior of the specimens can be divided into 3 distinctive zones (highlighted in Fig. 6a), as follows:

[Insert Fig. 6]

☞ **ZONE I:** in this phase, the temperature variation presents a linear decreasing followed by a linear trend of the stress-time curve. The specimen shows a globally elastic behavior; this phase is explained by the theory of thermoelasticity [39]. At the same time, the resistance variation slightly increases, this can be attributed to the piezoresistive effect of the carbon fibers. It is exemplified in Eq.(3) by the relation between electrical resistance and the geometric properties of the specimen.

$$R = \frac{\rho L}{S} \quad (3)$$

where  $\rho$  is the electrical resistivity of the material,  $L$  is the length of the conductor (distance between the two points of measurement) and  $S$  is the cross-sectional area of sample ( $25 \times 3 \text{ mm}^2$ ) crossed by electric current. When the composite is loaded, the length ( $L$ ) increases and consequently the cross-section ( $S$ ) diminishes (Poisson effect), which leads to the growth of  $R$ .

☞ **ZONE II:** This phase is associated to the micro-damages developed in the specimen from the micro-defects generated during the manufacturing process (especially from porosities illustrated in Fig.1b), even if at the macroscopic point of view the material does not show any damage. In this stage, the temperature response loses its inverse linear proportionality with the applied stress. The temperature decreases non-linearly

until reaching a minimum value. This point corresponds to a balance between the energy dissipated for elastic deformation (cooling the specimen) and the energy absorbed by the composite to increase defects and damages (heating the specimen) [40]. The same behavior is noticed by the significant drop of electrical resistance variation throughout this phase until reaching its lowest value in the test. Indeed, during the two phases I and II, the damage mechanisms in the composites are essentially attributed to the matrix cracking and interfacial debonding between fibers and matrix. These behaviors are found by the lower value of AE cumulative absolute energy through the zone A (Fig. 6b) that regards to physical phenomena of inferior AE energy such as those mentioned. These two damage mechanisms are schematized in Fig. 7b. Matrix cracking and fiber/matrix debonding lead the carbon fibers (highly conductive) without coating created by their thermoplastic matrix PPSU (weakly non-conductive) and therefore, diminish the resistance to current flow through the carbon fibers. The material consequently becomes more conductive. It is noticeable that the real-time monitoring of electrical resistance is capable of indicate the health status of the material under tensile loading tests.

☞ **ZONE III:** The damages events regarding this zone consist especially of the fibers rupture that is characterized by the significant increase in the AE cumulative absolute energy during zone B in Fig. 6b. The fibers rupture develops continuously due to the increasing on loading until propagation finally becomes instable and therefore releases suddenly a considerable amount of heat. Fiber breakage (schematized in Fig. 7c) is also responsible to increase abruptly the electrical resistance. This behavior is caused by the reduction of electrical pathway, regarding the rupture of fibers, that consequently restricts considerably the flow of electrical current... The absolute total variation of  $\Delta R/R_0$  in the end test is evaluated to  $3.5^{\pm 0.2} \%$  (Fig. 6). Considering that

the reported damage in this event is a major deterioration for the composite that leads the specimen to failure, the monitoring of electrical resistance may also avoid catastrophic failure and may be supplementary to the others techniques such as acoustic emission and IRT employed in this work.

[Insert Fig. 7]

### 3.2. *Self-Heating tests*

Fig. 8 shows the typical response of  $\Delta R/R_0$  (calculated according Eq.(1) and illustrated in blue continuous line) and  $\Delta T$  (calculated according Eq.(2) and plotted in red color) during the cyclic mechanical loading carried out on the carbon fibers/PPSU composites. The zoom 1 on the two blocks 12 and 13 of Fig. 8 shows the interest to apply a smoothing on the temperature and electrical resistance curves in order to visualize their constant trends during each block. The smoothing operation is conducted using the “smoothing splines” option of Matlab® 2017b software.

Similarly, during each loading block, we programmed the acquisition of the 10 first cycles, then one every 10 cycles, then one every 100 cycles and finally one every 500 cycles, in order to improve the displaying of (load vs. time) curve (Zoom 2 in Fig. 8). The temperature measurement is conducted on the surface of the specimen and moreover at some distance while the electrical resistance is a volume measurement and is carried out in contact of material. The thermal measurement is performed throughout the entire test, from the beginning of loading until the failure of the specimen, while the electrical resistance stops before the specimen failure because its drastic increase that exceeds the limit of the digital multimeter. This response is mainly attributed to the fiber breakage, one of the major damages in the material, that becomes the specimen non-conductive due to the rupture of carbon fibers and therefore leads the electrical resistance towards infinite values.

[Insert Fig. 8]

During the first blocks, the temperature and electrical resistance return to their initial values when unloading the specimen. However, when the damage is accentuated, the composite fails to reach its initial values while maintained at zero load for 6 minutes. These two physical quantities have undergone smoothing to eliminate the fluctuations and to visualize the stabilization maintaining per block, in temperature (green curve in Fig. 8) and electrical resistance (purple curve in Fig. 8). The stabilization of electrical resistance is more evident during the first loading blocks than at the end of the test; this is mainly due to the development of damage in the material as well as the welding quality of the electrical wires which degrades throughout the test.

The stabilized mean temperature during each loading block was obtained for the six samples submitted to the self-heating tests. Fig. 9a exhibits the evolution of the stabilized mean temperature as a function of the maximum stress at every block. The intersection of the two linear regressions regarding the two distinct linear parts (mean\_part 1 and mean\_part 2) in the curve gives a rapid estimation of the fatigue limit of the composite at 273 MPa.

The same procedure was carried out for the electrical resistance data acquired for the 6 specimens to investigate the fatigue limit. The results of the stabilized resistance variation as a function of the maximum stress at each block are presented in Fig. 9b. The intersection of the two linear regressions results on the fatigue limit of 225 MPa. The originality of this study lies in the use of electrical resistance as a novel approach for estimating quickly the fatigue limit in comparison to the conventional method (S-N curve). This technique may be employed supplementary to IRT technique to establish the mentioned parameter. Additionally, the electrical resistance measurement may be applied to composite structures to monitor in-situ its health-status, which may avoid unexpected major damage.

[Insert Fig. 9]

Five specimens were loaded at  $\sigma_{max}=273$  MPa (the highest value from the two limits) on conventional fatigue tests ( $R=0.1$  and a frequency of 2 Hz) according to Table 3 and reached  $2.10^6$  cycles without breaking. This seems to confirm that the two values obtained by self-heating can estimate rapidly and with two alternative methods the fatigue limit of the thermoplastic PPSU/carbon composite material.

[Insert Table 3]

#### 4. Conclusions

In this study, woven carbon fiber reinforced the thermoplastic PPSU (polyphenylsulfone) matrix were subjected to tensile and self-heating tests. Tensile test results reveal that the electrical resistance response makes it possible to evaluate the health-state change of the stamped PMC material: at the beginning of the test, the electrical resistance increases with the stress increase then drops when interfacial debonding occurs between the carbon fibers and their thermoplastic matrix (the fibers are taking off from their matrix, so they become more conductive, therefore the resistance decreases). At the end, the resistance increases sharply when the carbon fibers are broken. Damage mechanisms identification was easy based on both the temperature variation profile and especially the acoustic activity recorded during the tensile test.

In self-heating, the goal was to use the conductive effect of this type of PMC material in order to propose a new methodology for rapid estimation of their fatigue limit. Thus, the block-stabilized electrical resistance was noted, and the stabilized resistance variation as a function of the maximum stress was plotted. The fatigue limit was estimated and compared with that found using the conventional stabilized temperature variation. Both measurements

were conducted simultaneously during the self-heating tests. The two methodologies are based on two different physical magnitudes, the first (the electrical resistance) is conducted in contact with the material but the second (the surface temperature) is carried at some distance of the surface of the loaded structure. For this reason, the two estimated fatigue limits, evaluated using the temperature and electrical resistance respectively, present a gap of 21%. However, the particularity of the electrical measurement lies in the fact that it is governed by the carbon fibers (current conductors) which form part of the material. For this, a simple real-time measurement of the variation of its electrical resistance as a function of the monotonic cyclic loading can give a quick estimate of the fatigue limit of the structure which they are part.

To reduce the difference between these two limits, adjustment works on both measurement techniques must be made: improvement of the welding quality of the electric wires, coloring the hydraulic jaws in black, use of the thermocouples for a temperature measurement in contact...

Finally, the originality of this study lies in two major points:

- ☞ The use of the electrical resistance variation to evaluate the health-state of the stamped thermoplastic PMC material.
- ☞ The proposal of a new physical quantity (the electrical resistance measurement during the self-heating tests) for the rapid estimation of the fatigue limit of thermoplastic PMC which present more and more a very considerable stake given the high viscosity of their thermoplastic matrices.

## **Data availability**

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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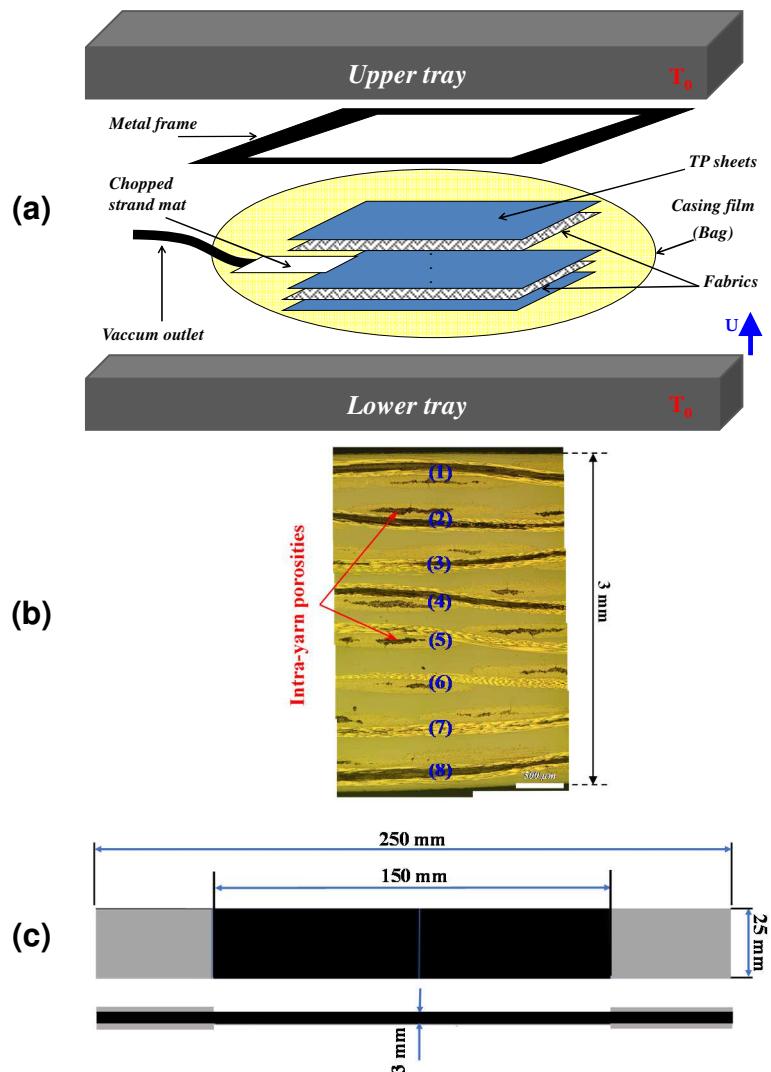
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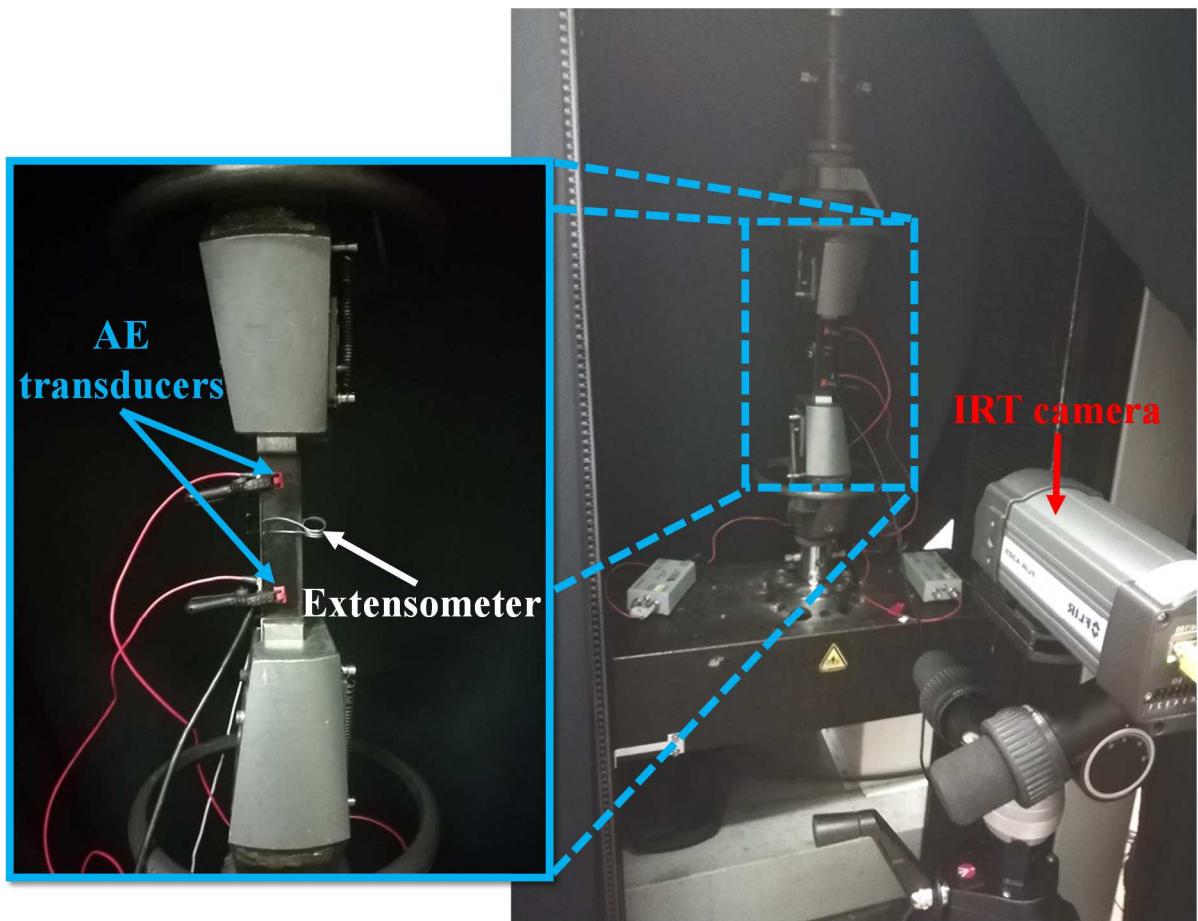
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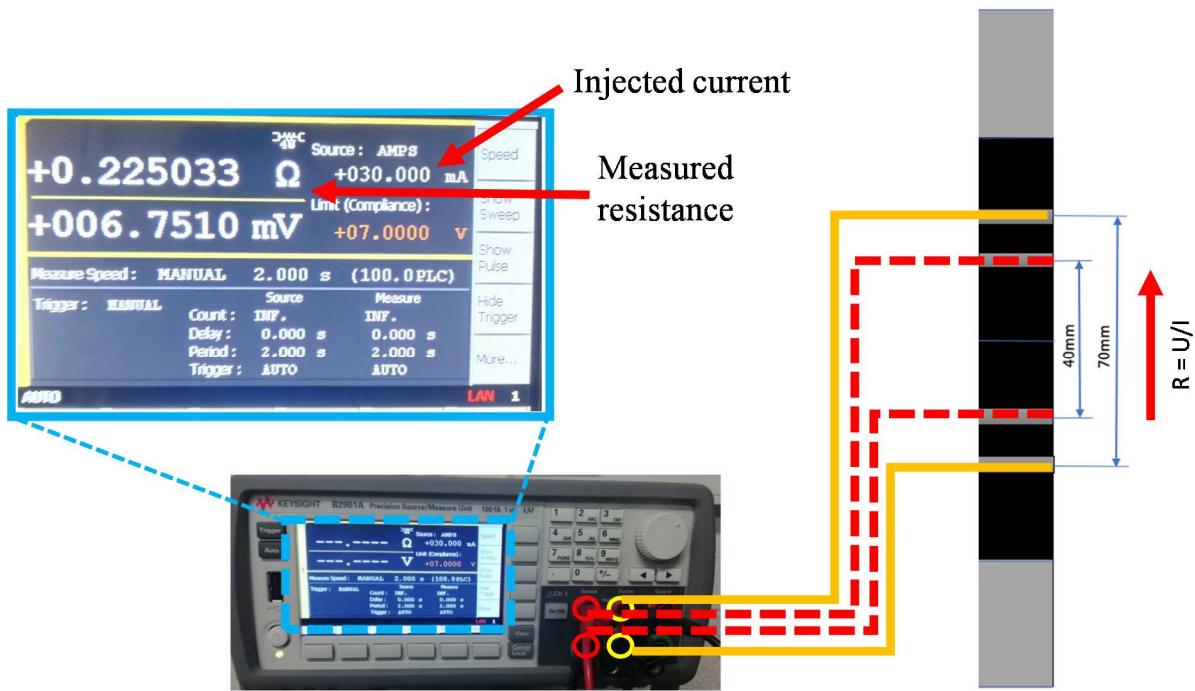
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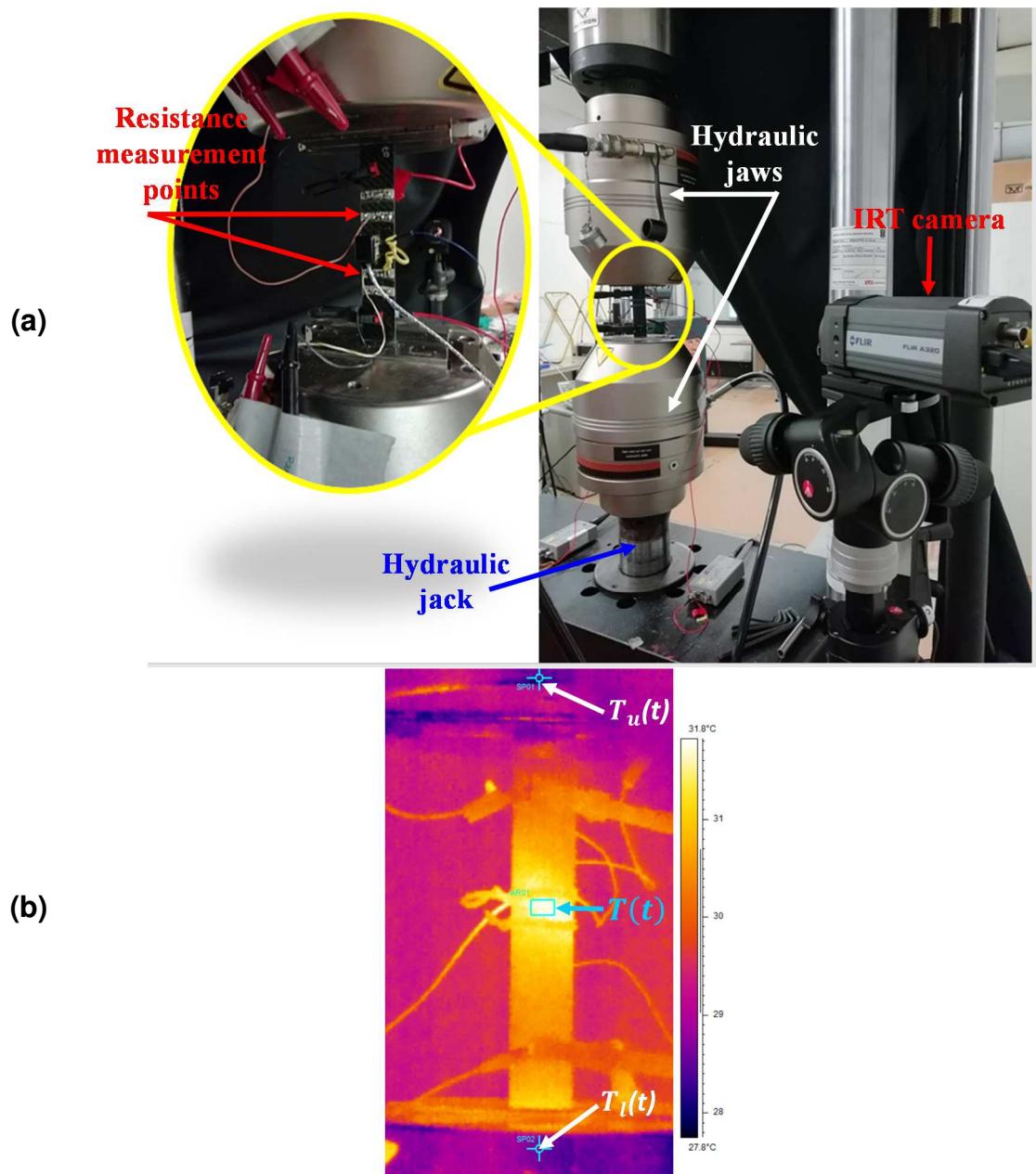
**Figure 1.** (a) thermo-stamping process [32]; (b) optical micrographic observation in the thickness of PMC material; (c) dimensions of the tensile specimens



**Figure 2.** Experimental devices used in the tensile tests.

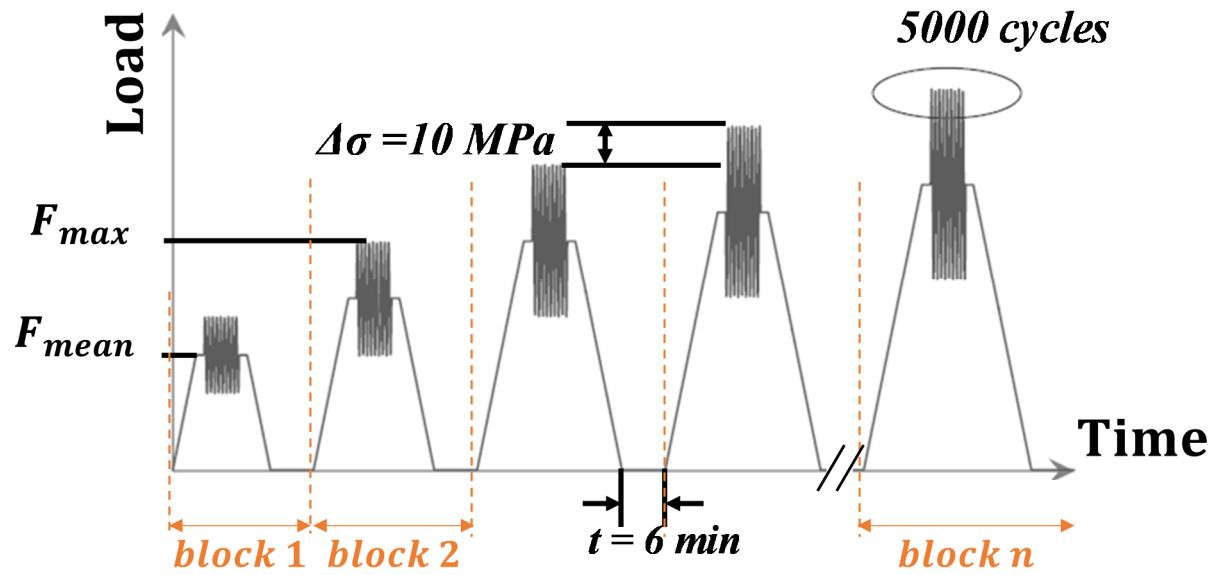


**Figure 3.** The 4-wires sensing method used for electrical resistance measurements.

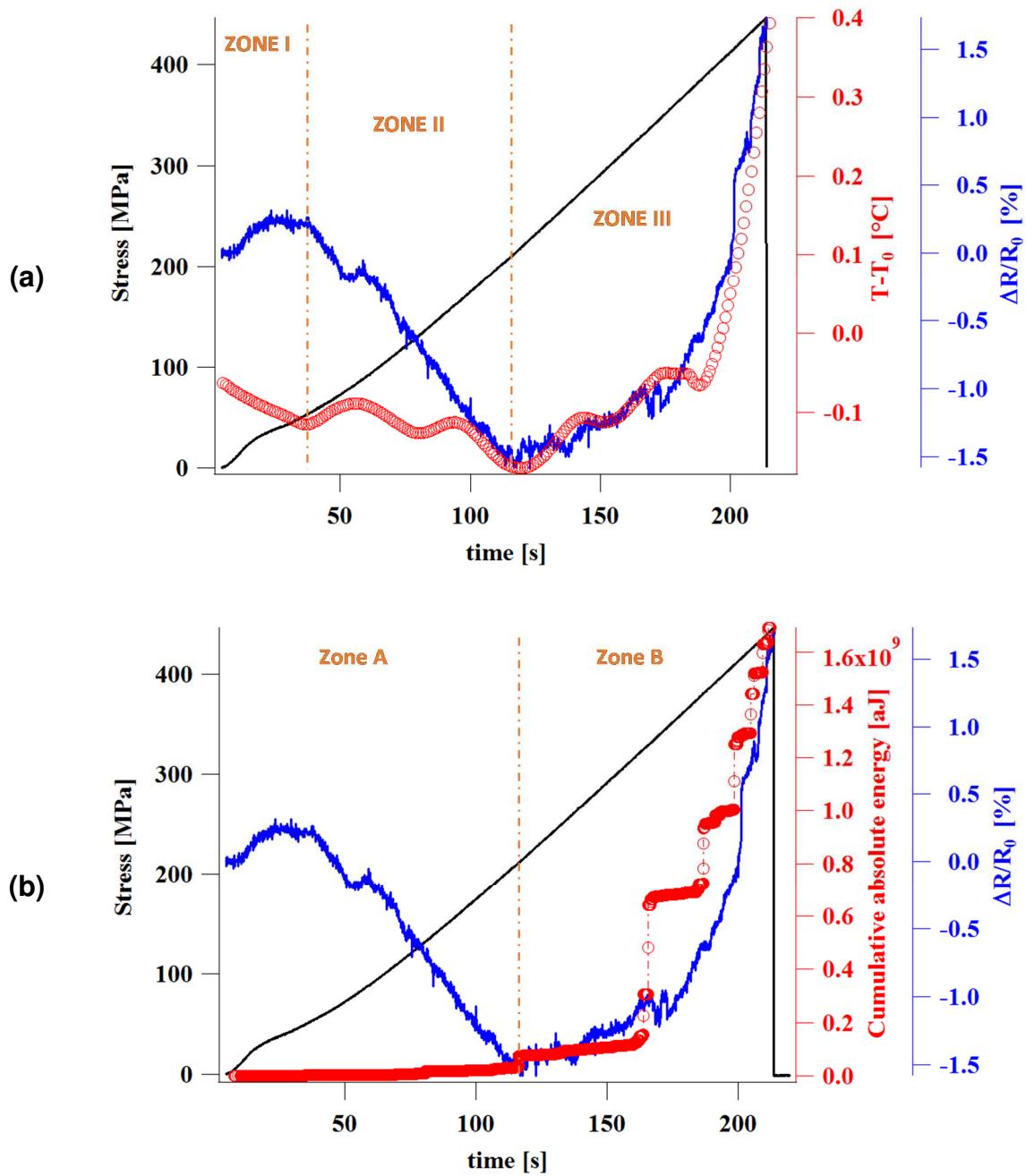


**Figure 4.** (a) Experimental setup during the self-heating test; (b) IR temperature measurement

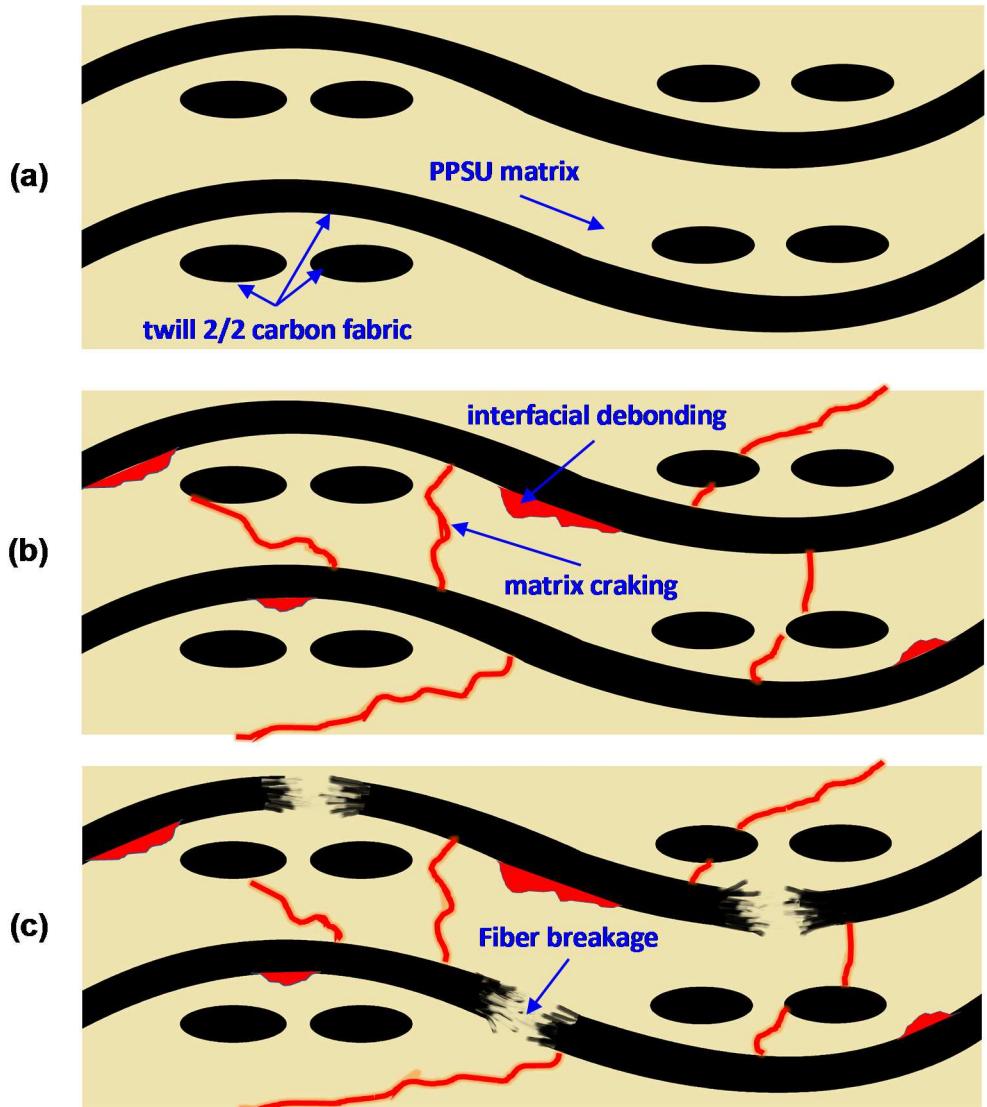
using IRT camera



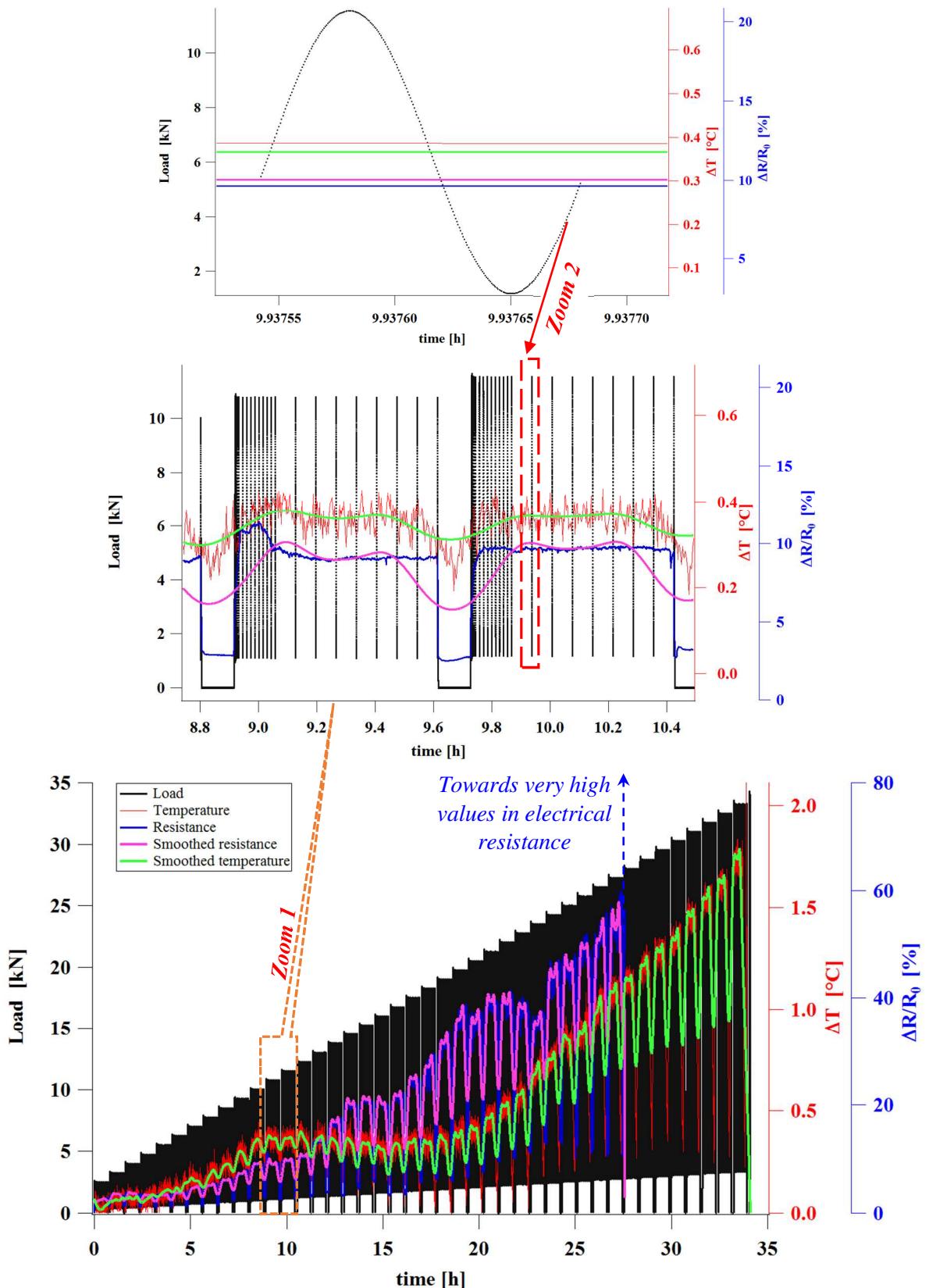
**Figure 5.** Schematic of the loading method utilized during the self-heating tests.



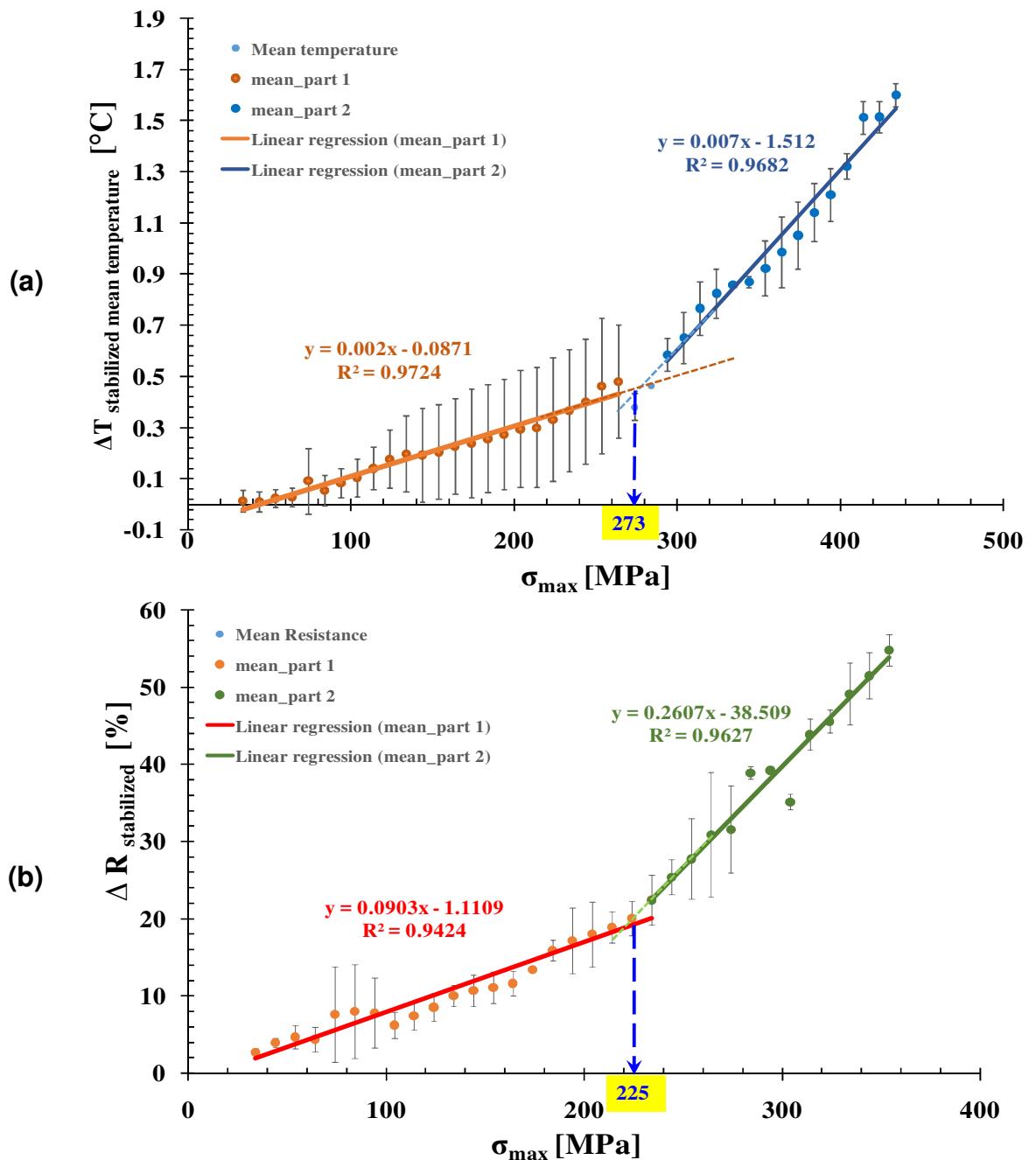
**Figure 6.** Typical behavior of (a) temperature and electrical resistance variations and, (b) AE cumulative absolute energy during the tensile test.



**Figure 7.** Schematic illustrations of damage mechanisms of the specimens during the tensile test: (a) initial state before loading; (b) matrix cracking and fiber/matrix debonding that lead to the drop of the resistance and (c) fiber breakage, source of the electrical resistance increase.



**Figure 8.** Typical curves of temperature ( $\Delta T$ ), electrical resistance ( $\Delta R/R_0$ ) and load versus time for the carbon fibers/PPSU subjected to self-heating test.



**Figure 9.** Fatigue limit evaluation using (a) temperature and (b) electrical resistance

variations

Block	$\sigma_{max}$ (MPa)	$F_{max}$ (N)	$F_{mean}$ (N)	$F_a$ (N)
1	34	2550	1403	1148
2	44	3300	1815	1485
3	54	4050	2228	1823
4	64	4800	2640	2160
:	:	:	:	:
Specimen's failure				

**Table 1.** Mechanical loading values in self-heating test

Young's modulus $E$ (GPa)	Tensile strength $\sigma_R$ (MPa)	Tensile elongation $\varepsilon_R$ (%)
48 $\pm$ 2	452 $\pm$ 36	0.95 $\pm$ 0.12

**Table 2.** Tensile properties of the specimens

$\sigma_{max}$ (MPa)	$F_{max}$ (N)	$F_{mean}$ (N)	$F_a$ (N)	<i>Number of cycles</i>
273	20475	11261	9214	<b>&gt;2.10<sup>6</sup></b>

**Table 3.** Parameters of conventional fatigue test