Mechanical behaviour at elevated temperatures of GFRP pultruded composite profiles

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M.Sc. Dissertation in Civil Engineering
Extended Abstract

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Summary: This paper presents results of an experimental and analytical study about the mechanical behaviour at elevated temperatures of glass fibre reinforced polymer (GFRP) pultruded profiles made of polyester resin and E-glass fibres. The paper first describes results of DMA and DSC/TGA tests that were used to evaluate the glass transition and decomposition processes of the GFRP pultruded material. It then describes results of an extensive study about the tensile, shear, and compressive responses of the GFRP material at temperatures varying from 20°C to 250°C, namely the load-deflection curves, the stiffness, the failure modes and the ultimate strength. The final part of this paper assesses the accuracy of different empirical and phenomenological models published in the literature, as well as an empirical model proposed in this study, to reproduce the variation pattern as a function of temperature, that was verified for the mechanical properties that were studied. In the modeling performed, it was found that all models under analysis can adjust with greater or lesser accuracy the variation pattern obtained from experimental tests for the different properties in study. The phenomenological model presented the worst fitting, while the empirical models resulted on very similar adjustments.

Keywords: GFRP profiles, fire, elevated temperature, tension, shear, compression, DMA, DSC.

1. Introduction

Glass Fibre Reinforced Pultruded profiles (GFRP profiles) are made of composite materials, consisting of glass fibres embedded in a polymeric matrix (usually polyester or vinylester) and are included in the group of fibre reinforced plastic materials, also known as FRP materials. The use of these materials in civil engineering applications has grown significantly in the last two decades, owing to their main advantages over traditional materials, namely, high strength, lightness and corrosion resistance.

Inevitably, with the growth of the number of the application of this material in the civil engineering field [1], new design issues and challenges are inevitably encountered. Among these issues, are the legitimate concerns regarding the performance of FRP materials when exposed to high temperatures, especially in building applications, where the safety of people and property are at stake. In fact, the loss of FRP’s properties when subjected to high temperatures is well known, namely when the glass transition temperature (Tg) of the resins is approached, typically in the range of 60-170°C. Furthermore, when exposed to temperatures about 300-500°C, the organic matrix of these materials decomposes, releasing smoke and toxic volatiles [2].

Although this problem is of great importance, there is relatively limited knowledge about the response of organic matrix composites at elevated temperatures. On the other hand, in order to enable the structural use of GFRP profiles in building applications, fire design methods are still to be developed. One step towards such development involves the careful description of GFRP
material mechanical behavior at elevated temperature, where duly validated curves expressing the degradation of mechanical performance as a function of temperature are needed. However, in order to develop such mechanical degradation curves, a wealth of experimental data is needed and, presently only a limited number of studies are available in the literature.

Therefore, this paper presents further research about the mechanical response at elevated temperature of glass fiber reinforced polymer (GFRP) pultruded profiles with the objective of contributing to the development of material degradation curves to be used in fire design. The glass transition and decomposition process of the GFRP pultruded material used are first analysed by means of DMA and DCS tests, respectively. Subsequently, the tensile, shear and compressive responses of the GFRP material at temperatures varying from 20°C to 250°C are evaluated in terms of load-deflection curves, stiffness, failure modes and ultimate strength. In order to complete this study, the results obtained herein were compared with the results reported by other authors, namely Keller and Bai [3], Robert and Benmokrane [4] and Wong et al. [5].

The final part of this paper assesses the accuracy of different empirical models and one phenomenological model suggested in the literature to estimate the variation with temperature of the tensile, shear and compressive strengths of the GFRP pultruded material. A new model based on Gompertz [6] statistical distribution is proposed and its accuracy is compared with that of the remaining models. The experimental part of the study aimed at complementing existing literature, whereas the modeling was performed with the purpose of assessing the reliability of models suggested earlier, thereby validating design tools that may allow for simple and quick characterization of the mechanical properties variation with temperature.

2. Experimental procedure

2.1 Materials

The GFRP pultruded material used in this experimental campaign was produced by company Fiberline DK and consists of alternating layers of unidirectional E-glass fibre rovings and strand mats embedded in an isopthalic polyester resin matrix. The two following cross-sections were used in the experiments: a rectangular plate (500 mm wide and 10 mm thick), which was used in the tension and shear tests, and an I-section profile (120 mm (height) × 60 mm (width) × 6 mm (flange and web thickness)) that was used in the compressive tests. The inorganic content of those cross-sections, determined from burn-off-tests, is 70% and 69% respectively.

2.2 Thermal analyses

2.2.1 Dynamic mechanical analysis (DMA)

The dynamic mechanical analyses (DMA) were performed as specified in the ISO 6721 [7] standard, on specimens sawed from the web of the I-section profile with 40 mm (length, in pultrusion direction) × 15 mm (width) × 3 mm (thickness).
In these tests, coupon specimens are subjected to a sinusoidal mechanical oscillation at a fixed frequency and, while temperature increases at a constant rate, the amplitudes of the load and deformation cycles and the phase angles between these cycles are measured. DMA provides a quantitative determination of the mechanical properties of a sample under an oscillating load and as a function of temperature, time and frequency. These experiments were performed in the GFRP pultruded material in order to determine the glass transition temperature ($T_g$), which marks the transition from a glassy state to a rubbery solid state, and is associated with a considerable reduction of the mechanical properties, namely the stiffness and strength.

Experiments were performed on a Q800 dynamic mechanical analyser from TA Instruments, with capacity of 18 N and resolution of $1 \times 10^{-4}$ N, in a single-cantilever test setup. In this test setup the sample is clamped on one side and flexed in the other. The specimens (one for each heating rate) were scanned from 30°C to 250°C (higher than the $T_g$, but lower than the $T_d$) at six different heating rates (0.5°C/min, 1°C/min, 2°C/min, 4°C/min, 6°C/min and 8°C/min) and four dynamic oscillation frequencies ($f_1=1$ Hz, $f_2=3$ Hz, $f_3=5$ Hz e $f_4=10$ Hz).

2.2.2 Thermogravimetric analysis and differential scanning calorimetry (DSC/TGA)

The thermogravimetric analysis and differential scanning calorimetry experiments were performed in the GFRP material of the flat profile in accordance with the ISO 11357 standard [8], in order to determine the mass variation and the energy changes suffered by the materials as a function of temperature and time.

While TGA analysis measures the specimen’s mass variation, the DSC analysis measures the energy changes suffered by the material as a function of time and temperature. In this analysis one test specimen and one reference specimen are used, the latter for equipment calibration. Both specimens are placed inside the oven, over platinum containers, and then the oven is heated at a controlled heating rate in two atmospheres (air and nitrogen). In these tests the temperature inside the oven is uniform, with equal heat fluxes being submitted to both test specimens. If the specimens’ thermal capacity differs or if the test specimen exhibits changes in heat absorption/loss due to chemical reactions or phase transitions, the consequent flow difference creates thermal gradients that are registered by the equipment. So, DSC/TGA analysis allows to determine the decomposition temperature ($T_d$), from the mass and heat flow changes with the increasing temperature.

Experiments were performed on a SDT2960 Simultaneous differential scanning calorimeter and thermogravimetric analyser from TA Instruments, from room temperature to 600°C in air and nitrogen atmospheres, at heating rates of 5, 10, 15 and 20°C/min. Specimens were produced by sawing the flat plate profiles into small parallelepiped specimens with approximately 10 mg. The time, the temperature, the mass of the specimens and the heat flow during the tests were measured and registered.

2.3 Mechanical tests

2.3.1 Specimens preparation and instrumentation

The tests specimens were obtained by sawing the pultruded profiles with the dimensions listed in Table 1.
Table 1: Specimen’s dimensions (l-length, w-wide, t-thick).

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Dimensions (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile</td>
<td>l: 1800, w: 20, t: 10</td>
</tr>
<tr>
<td>Shear</td>
<td>l: 800, w: 25, t: 10</td>
</tr>
<tr>
<td>Compressive</td>
<td>l: 50, w: 120, t: 6</td>
</tr>
</tbody>
</table>

Regarding the axial deformation, in order to assess the stress-strain relationship, some specimens tested at ambient temperature were monitored with general purpose strain gauges from TML, model FLK-6-11 (with an electrical resistance of 120 Ohm). For specimens tested at elevated temperature, strain gauges also from TML, model BFLA-3-11 (also with an electrical resistance of 120 Ohm), were used.

In order to measure the temperatures in the specimens, thermocouples type K were used. In order not to jeopardize the structural integrity of the specimens to be loaded in the tensile and shear experiments, it was decided to produce “dummy” specimens (Figure 1), with three thermocouples installed at different heights at a depth of 5 mm (half of the specimen thickness), for the control and monitoring of test specimen temperatures, which were also positioned inside the furnace. Temperatures were measured at a height corresponding to the center of the tested (loaded) specimen and, in addition, in the upper and bottom part of the dummy specimen. For the compressive experiments, due to the test setup, the thermocouples were fixed in the center of the specimen’s web (Figure 2), at a depth of 3 mm, which corresponds to half of the specimen thickness.

2.3.2 Tensile tests at elevated temperature

Tensile tests were performed on specimens 1800 mm long obtained by sawing the flat profile with a circular sawing machine. In these tests, the central length (1100 mm) of the specimens was first heated up at an approximate rate of 7.5°C/min to a predefined temperature (20°C, 60°C, 90°C, 120°C, 150°C, 200°C, 220°C) using a Shimadzu thermal chamber with inner dimensions of 1100 x 280 x 340 mm. When the predefined temperature was reached the specimens were monotonically loaded until failure using a Schenck 500 universal testing machine, with a load capacity of 500 kN. Tests were carried out as suggested in ISO 527-1,4 [9] under displacement control at an approximate speed of 2.0 mm/min. An average of 3 specimens was tested for each temperature and, the clamps of the machine were kept at ambient temperature to prevent premature failure mechanisms. Figure 3 shows the setup used in these tests.
2.3.2 Shear tests at elevated temperature

In-plane shear tests were carried out by means of 10°C off-axis tensile experiments. GFRP specimens were prepared by sawing the rectangular flat profile with a length of 800 mm and a cross-section of 25 mm (width) × 10 mm (thickness), as specified in ISO 527 – 1,4 [9].

Figure 4 illustrates a general view of the test setup and equipment used in the experimental tests that included an Instron universal testing machine and an electrical split furnace attached to it.

The central length of the specimens (150 mm long) was first heated up to a predefined temperature (20°C, 60°C, 90°C, 120°C, 150°C, 200°C, 250°C) at an approximate rate of 6°C/min using a split furnace from Termolab, with an inner diameter of 150 mm and height of 450 mm. The temperature of this oven is defined by means of an electronic controller of Shimaden, model FP21. The remaining length of the specimens was insulated with ceramic wool (with an approximate thickness of 15 mm). After heating, specimens were monotonically loaded until failure using an Instron universal testing machine, with a load capacity of 250 kN (Figure 4). Tests were carried out under displacement control at an approximate speed of 2.0 mm/min. The clamps of the machine, in which the extremities of the laminates were gripped, were kept at ambient temperature to prevent premature failure mechanisms. For each temperature series at least five specimens were tested.

2.3.3 Compressive tests at elevated temperature

The specimens used in the compressive tests, were obtained by sawing the I-section profile with a tape sawing machine in 50 mm long specimens. Figure 5 illustrates a general view of the test setup that included, among other components, the split furnace used in the shear tests, the compression test machine and the grooved steel plates that fixed the specimens during loading.
In these tests the specimens were positioned in between cylindrical steel blocks, with a height of 280 mm and a diameter of 150 mm. The upper block was bolted to the reaction frame of the test machine, while the lower one was positioned on top of a load cell from Microtest, model MT-CLC/3000, with a capacity of 3000 kN. These blocks were covered by thick steel plates that materialized the supports of the specimens. These 20 mm thick plates had 5 mm deep grooves mimicking the geometry of the cross section (Figure 5 (c) and (d)). The plates were machined in order to fit the test specimens, keeping them in position and preventing the rotation of their extremities. The width of the grooves was slightly thicker than that of the I-section walls in order to provide some geometrical tolerance and avoid crushing mechanisms during loading due to local misalignments.

The specimens were first heated up at an average rate of 8°C/min to predefined temperature (20°C, 60°C, 90°C, 120°C, 150°C, 200°C, 250°C). Subsequently, specimens were monotonically loaded until failure using an Inerpar 3000 compression test machine with a load capacity of 3000 kN and a pressure unit of Walter+Bai. Tests were carried out under load control at an approximate speed of 4.0 kN/s. The applied load was measured using a load cell which, as already mentioned, was positioned below the bottom steel cylindrical block. The vertical deflection of the test specimen was measured using a high-temperature LVDT from RDP, model LIN252A, with a stroke of ± 25 mm and a precision of 0.01 mm, which is able to measure deflections up to temperatures of about 220°C. The LVDT was fixed to a steel tube, welded to the bottom cylindrical block, and its piston contacted with a steel plate welded to the top cylindrical block. In these tests at least five specimens were tested for each temperature.

Unlike the tensile and shear tests (in which the clamps were kept at ambient temperature), in the compressive tests the supports of the specimens were also heated during the tests, owing to the test setup, in particular, due to the small length of the test specimens. Therefore, in order to prevent premature crushing failure mechanisms at the extremities of the specimens, before each test, the grooved steel plates where specimens were supported were cooled by using freeze pack coolers.
3. Experimental results and discussion

3.1 DMA and DSC/TGA tests

DMA results showed the typical considerable peaks in the loss modulus and loss factor (tangent delta) curves, and a considerable drop in the storage modulus curves. Those curves provide estimates of the $T_g$, the highest being obtained from the tangent delta curves, the lowest from the storage modulus curves, with intermediate estimates being provided by the loss modulus curves. As expected, $T_g$ estimates increased with the heating rate and the oscillation frequency. At a frequency of 1 Hz, $T_g$ estimates from the loss modulus curves varied between 97°C and 143°C for the different heating rates. As an example, figure 6 shows results obtained for heating rates of 5°C/min and 8°C/min, including the variation of the storage modulus retention (compared to ambient temperature) and of the degree of glass transition ($\alpha_g$, obtained from the variation of the storage modulus).

Figure 6: Variation with temperature of storage modulus and degree of glass transition temperature for heating rates of 6°C/min and 8°C/min.

Figure 7: Variation with temperature of remaining mass and decomposition degree for heating rate of 5°C/min.
Regarding to DSC/TGA tests, figure 7 plots the remaining mass curve (in air atmosphere) obtained for heating rates of 5°C/min and, the decomposition degree ($\alpha_d$) obtained from the variation of the mass with the temperature. The temperature of decomposition ($T_d$), determined as the middle temperature in air atmosphere (for 5°C/min), was set as $T_d=350^\circ$C, which corresponds to 87.5% of remaining mass.

3.1 Tensile tests at elevated temperature

Figure 8 presents the load-displacement curves for one representative specimen of each one of the target heating temperatures. Each representative specimen corresponds to an intermediate curve obtained within each series.

From the load-displacement curves presented it can be stated that for all temperatures, the material presents a linear response until failure, in particular for temperatures up to 150°C, as for higher temperatures tensile stiffness was progressively reduced prior to failure.

In figure 9 are represented the variation of the tensile strength and stiffness as a function of temperature. It can be seen that the tensile strength presents an almost linear reduction from ambient temperature up to 220°C, for which the material retains about 54% of the ambient temperature strength. In turn, the tensile stiffness did not suffer any remarkable changes up to a temperature of 150°C and for 220°C in maintained about 88% of the ambient temperature value. With these results, it can be seen that the glass transition process seems to affect tensile strength much more than stiffness.

Since during the specimens heating the clamps remained at ambient temperature, failure always occurred in the gage (heated) region due to the tensile rupture of the glass fibres, as illustrated in Figure 10. For temperatures to 150°C, the failure mode was similar to that observed at ambient temperature: delamination and tensile rupture of the fibres without any clear visual influence of resin softening and decomposition. For temperature equal to and above 200°C, the tensile rupture of the fibres was somewhat affected by resin softening and decomposition: in fact, although failure was also governed by the fibres tensile strength, it was possible to identify a high volume of broken and tensioned loose fibres prior to failure. As a consequence, at higher temperatures, the stress distribution among the glass fibres prior to failure became less uniform. This also explains why tensile strength is more affected by high temperature than stiffness.
The experimental results of strength obtained in the present study were compared with the results reported by Keller and Bai [3] and Robert and Benmokrane [4] for pultruded laminates and bars, respectively. Figure 11 plots results obtained in those two studies together with results obtained in this study and, it can be seen that the results obtained are in a very good agreement with those reported by Robert and Benmokrane. Both data sets show considerable tensile strength reduction at elevated temperature, namely across the glass-transition of the polymer matrix. In what concerns the comparison of the experimental data reported herein with that obtained by Keller and Bai, tensile strength results are in fairly good agreement for temperatures up to 100°C. But, for higher temperatures, this agreement does not occur, with tensile strengths reported by Keller and Bai being considerably lower than those obtained in the present study. Such difference is justified with the premature failure mechanisms at the grips that were observed by those authors, which were prevented in the present study by keeping the machine clamps at ambient temperature.

3.2 Shear tests at elevated temperature

Figure 12 shows the load-displacement curves obtained for one representative specimen of each one of the target heating temperatures. Each representative specimen corresponds to an intermediate curve obtained within each series. Analyzing each curve that was obtained, it is possible to verify that material presents a linear response until failure, with only a slight stiffness reduction prior to failure.

Figure 13 plots the shear strength and the stiffness as a function of temperature. From the analysis of this figure, it can be seen that the shear strength decreases with temperature, with a very significant reduction occurring between temperatures of 90°C and 150°C, which can be attributed to the glass transition process undergone by the polyester resin that constitutes the matrix of the GFRP composite. At higher temperatures, namely at 250°C, the shear strength is reduced to almost 11% of the value obtained at ambient temperature, which is a very significant loss of this property. The stiffness suffers a slight reduction from 20°C up to 90°C and then, between temperature of 90°C and 150°C, stiffness suffers a step reduction and it then tends to stabilize up to 250°C. In this case, these variations are also naturally due to the glass transition process undertaken by the resin matrix in the gauge region of the specimen. With this regard, it is worth mentioning that most of the length of the test specimen is
thermally insulated and therefore is not expected to contribute to the overall stiffness reduction. In addition, the tensile load is applied at an angle of only 10° with the pultrusion direction, which means that a considerable part of the solicitation is carried out in tension and, for this type of loading, the glass fibres retain a considerable fraction of ambient temperature stiffness. For these reasons the relative reduction of stiffness is considerably smaller than that exhibited by shear strength. In fact, at 250°C residual stiffness is 56% (compared to ambient temperature), while residual shear strength is only about 11%.

The typical failure mode observed in the shear test is illustrated in figure 14. As in the tensile tests, failure always occurred in the gage region, but now the failure surfaces were oriented roughly at a 10° off-axis, parallel to the pultrusion (rovings) direction. In this shear failure mode, the longitudinal fibres did not break but the superficial mats were torn.

The values of residual shear strength obtained in the present study were compared with the results reported by Keller and Bai [3] and Robert and Benmokrane [4]. Figure 15 represents those results and it can be seen that the results obtained herein has a good agreement with the results reported by Keller and Bai. On the other hand, the same analysis can’t be made as to about the results reported by Robert and Benmokrane, that although showing also a considerable decrease with the temperature, present a rather different pattern of variation compared to those obtained here. In fact, the shear strength of the FRP bars tested by those authors suffered only a slight reduction for temperatures up to 150°C (less than 10%), and

![Figure 12: Load-displacement curves for representative specimens of all tested temperatures (shear).](image12)

![Figure 13: Comparison between the normalized values of the shear strength and stiffness.](image13)

![Figure 14: Typical failure mode for the shear tests.](image14)

![Figure 15: Comparison of the residual shear strength variation with temperature reported here and in different studies.](image15)
then the shear strength suffered a major reduction from 150°C to 250°C and in then stabilized up to 320°C. One believes that the reasons for this different behavior are related with the different test setup used by those authors.

### 3.3 Compressive test at elevated temperature

Figure 16 presents the load-displacement curves for one representative specimen of each one of the objective heating temperatures. Each representative specimen corresponds to an intermediate curve obtained within each series. The load-deflection curves measured for all temperatures tested exhibit an initial non-linear branch that is more or less pronounced and basically corresponds to the adjustments of the test setup, including those between the test specimen an the grooved end plates. Subsequently, the behavior is approximately linear up to failure, although for higher temperatures progressive stiffness reduction can be identified in the load-deflection curves.

![Figure 16: Load-displacement curves for representative specimens of all tested temperatures (compressive).](image)

Compressive strength and the stiffness as a function of temperature are represented in figure 17. Stiffness was obtained by the slope of the linear branch of the load-deflection curves. Results plotted in figure 17 show that the compressive properties are remarkably reduced with temperature, with compressive strength reduction being more pronounced than that exhibited by the stiffness. For a temperature as low as 60°C, that can be easily attained in outdoor applications or in structures of roofs, compressive strength is reduced more than 30% compared to ambient temperature strength. For a temperature of 90°C, compressive strength is already less than half of the ambient temperature strength. At the highest temperature tested of 250°C, the compressive strength is reduced to about 5% of the ambient temperature strength, which corresponds to very significant strength degradation, probably stemming from the low height of the specimens and the fact that even for high temperatures the material has limited compressibility.

The typical failure modes are represented in figure 18. Failure at ambient temperature (Figure 18 (a)) was caused by crushing and wrinkling of the GFRP material with ply delamination and, was more concentrated in the contact zone of specimen with the grooved steel plates. For elevated temperature (Figure 18(b)), the crushing of the GFRP material at the contact areas with the grooved plates became less frequent. For those higher temperatures failure became
more concentrated near the mid-height of the specimens and seems to have been triggered by the resin softening due to the glass transition process, which caused a kinking type of rupture. The residual compressive strength obtained in the present study was compared with the results reported by Keller and Bai [3] and Wong et al. [5] for GFRP pultruded tubes and C-channel columns, respectively. Figure 19 shows that the results obtained in the present study in terms of compressive strength as a function of temperature present a fairly good agreement with those reported earlier by Keller and Bai and by Wong et al. In fact, with the exception of Wong et al.’s results for 90°C, which were somehow reasonably higher than those obtained here, being also above the fitting curve to the results of Keller and Bai.

Figure 18: Typical failure modes for the compressive tests at (a) ambient temperature and (b) high temperatures.

Figure 19: Comparison of the residual compressive strength variation with temperature reported here and in different studies.

4. Modeling the mechanical properties degradation

4.1 Description of models and parameter estimation

The experimental results described earlier were used to assess the accuracy of models suggested in the literature to describe or simulate the mechanical degradation of the GFRP material at elevated temperature, namely the strength, the elastic modulus and distortion modulus. The accuracy of a new descriptive model, based on Gompertz distribution, is also assessed.

The models analysed include a phenomenological or semi-empirical model recently proposed by Keller and Bai [10,11] and, descriptive relaxation models, namely those presented by Gibson et al. [12], Mahieux et al. [13] and Wang et al. [14] that encompass curve fitting procedures.

In the phenomenological model proposed by Keller and Bai [10,11], the variation of the mechanical property with temperature is suggested to be calculated according to the following equation,

\[
P(T) = P_g \times (1 - \alpha_g) + P_l \times \alpha_g \times (1 - \alpha_d) + P_d \times \alpha_g \times \alpha_d
\]

where \( P_g, P_l \) and \( P_d \) are the mechanical properties in the glassy, leathery and decomposed states, respectively and, \( \alpha_g \) and \( \alpha_d \) are the glass transition and decomposition degrees, respectively, which are both computed based on kinetic theory [11] with the results from DMA and TGA tests. Therefore, the mechanical properties at a given temperature can be
determined by adopting an appropriate distribution function that weights the contribution of
the material response at those three different “states”.

According to Gibson et al.[12], the variation of a mechanical property with temperature can be computed based on the following equation (one relaxation),

$$P(T) = P_u - \frac{P_u - P_r}{2} \times (1 + \tanh[k'(T - T_{g,\text{mech}})])$$  \hspace{1cm} (2)

in which $k'$ and $T_{g,\text{mech}}$ are parameters obtained by fitting the experimental data and, $P_u$ is the property at ambient temperature and $P_r$ is the mechanical property after glass transition (but before decomposition), corresponding to the property in the glassy and leathery states, respectively. It is important to mention that $T_{g,\text{mech}}$ is not necessarily equal to $T_g$ (obtained from DMA or DSC analysis).

Mahieux et al.[13] suggested the following functional relationship based on Weibull distribution to compute the properties degradation in function of temperature (in Kelvin),

$$P(T) = P_r + (P_u - P_r) \times \exp[-(T/T_o)^m]$$  \hspace{1cm} (3)

in which, $T_o$ is the relaxation temperature and $m$ is the weibull exponent, both parameters being numerically fitted to the experimental data.

Wang et al.[14] recently proposed the following model, originally developed for metals subjected to high temperatures, which the authors successfully applied to describe the tensile strength of carbon fibre reinforced polymer (CFRP) pultruded laminates.

$$P(T) = P_u \times \left[ A - \frac{(T - B)^n}{C} \right]$$  \hspace{1cm} (4)

In this model the coefficients $A, B, C$ and $n$ are the adjustment result of the equation to the experimental data for different temperature ranges.

Finally, the following new model, which is based on Gompertz statistical distribution [6], is suggested as an alternative to the previous models,

$$P(T) = \left( 1 - Ae^{Be^{C\times T}} \right) \times (P_u - P_r) + P_r$$  \hspace{1cm} (5)

where coefficients $B$ and $C$ are fitting parameters that adjust the equation to the experimental data.

### 4.2 Results and discussion

The modeling curves were estimated based on the experimental results described in section 3. For Keller and Bai’s model [10,11], properties at 20°C and 250°C were considered respectively for the glassy and leathery states (based on DMA results plotted in Figure 6). For the decomposed state strength retention of 0.5, 0.0 and 0.0 were assumed for tension, shear and compression, respectively, while for the modulus was considered retention of 0 for all loads types.
For empirical models, parameters were estimated using a standard procedure that minimizes the mean square errors to the test data. In particular, for Wang et al.’s model [14], were considered three adjustment study cases: (i) a single adjustment temperature range, equal to the range of temperatures that was considered in the experimental campaign; (ii) two adjustment temperature ranges, intermediate situation among cases (i) and (iii) and case (iii) three adjustment temperature ranges: 20°C to 90°C, 90°C to 150°C and 150° to 250°C. The objective of these study cases was identifying the best that reproduce the variation pattern of each property studied.

The results of the modeling performed are described below.

4.2.1 Strength

The variation with temperature of the normalized strength obtained for tensile, shear and compressive tests are plotted in figures 20 to 22, together with the different modeling curves obtained from each model studied. It these figures it can be seen that, with the exception of the model proposed by Keller and Bai [10,11], the other models provided reasonably accurate estimates of tensile, shear and compressive strength data obtained in the present study. In fact, the estimates obtained with phenomenological model proposed by Keller and Bai tended to overestimate GFRP material strength for all types of loading, particularly for tension in the brink of glass transition (at 60°C and 90°C) and for compression for the whole range of temperatures. The absolute mean percentage errors (AMPE) presented in table 2 confirm the earlier analysis, since the highest values of this error were obtained for the Keller and Bai’s model. On the other hand, the smallest values of AMPE obtained for the modeling for the different types of load were obtained from Wang et al.’s [14] for tensile strength and from the model proposed in this study for shear strength and compressive strength. Thus, these are the more reliable models and its parameters are represented in table 3. It’s important to mention that the modeling curve of tensile strength obtained with Wang et al’s model, resulted from considering the case (i), for which the parameter estimation is done for a single temperature range (20 ≤ T < 220).
Table 2: Absolute mean percentage error (AMPE) obtained for each studied model.

<table>
<thead>
<tr>
<th>Models</th>
<th>AMPE [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tensile</td>
</tr>
<tr>
<td>Keller and Bai, Eq (1)</td>
<td>6.52</td>
</tr>
<tr>
<td>Gibson et al., Eq (2)</td>
<td>4.10</td>
</tr>
<tr>
<td>Mahieux et al., Eq (3)</td>
<td>4.81</td>
</tr>
<tr>
<td>Wang et al., Eq (4)</td>
<td>3.79</td>
</tr>
<tr>
<td>Present study, Eq (5)</td>
<td>4.32</td>
</tr>
</tbody>
</table>

Table 3: Simulation of the GFRP material strength degradation: parameters estimated for the best models.

<table>
<thead>
<tr>
<th>Model</th>
<th>Parameter</th>
<th>Tensile</th>
<th>Shear</th>
<th>Compressive</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wang et al. (Eq.4)</td>
<td>Case (i):</td>
<td>A=1, B=20, C=478.7, n=1.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Present study (Eq.5)</td>
<td>B [-]</td>
<td>-3.7</td>
<td>-42</td>
<td>-3.4</td>
</tr>
<tr>
<td></td>
<td>C [-]</td>
<td>-0.02</td>
<td>-0.05</td>
<td>-0.03</td>
</tr>
</tbody>
</table>

4.2.2 Elasticity modulus and shear modulus

Figures 23 and 24 plot the variation with temperature of the normalized elasticity modulus obtained from tensile and compressive tests and the respective modeling curves. The results for the modeling of the shear modulus are plotted in figure 25.

Through the obtained results it can be seen that in the case of tensile elasticity modulus, all obtained modeling curves are very similar and follow the continuous variation trend observed for this property. But, unlike the others, the curves obtained with Gibson et al.’s [12] model and with the model proposed in this study can adjust the elastic modulus values obtained for the highest temperature that was tested (220°C). Thus, these models are associated with smaller errors obtained (AMPE=3.8%, for both), being the most reliable models to adjust the tensile elasticity modulus.

Concerning the compressive elastic modulus, the results obtained show that all modeling curves are similar. But the modeling curve obtained from Keller and Bai’s [10,11] model, represents the worst fit obtained, which justifies its highest AMPE error (24.76%).

Finally, about the modeling performed over the shear modulus (Figure 25), it can be seen that the model proposed by Keller and Bai [10,11] overestimates these values, thereby representing the worst curve obtained (AMPE=50.2%), being the remainder very similar. The best fit was obtained with the model proposed by Wang et al. [14], for which the lowest AMPE value (3.1%) was obtained. It is worth to mention that the modeling of the shear modulus with Wang et al.’s model was performed for the case (ii) where two adjustment temperature ranges (20°C to 60°C and 60°C to 120°C) were considered.
The adjustment parameters that were obtained for the more reliable models for the elastic modulus modeling are listed in table 5.

<table>
<thead>
<tr>
<th>Model</th>
<th>Parameter</th>
<th>Tensile modulus</th>
<th>Compressive modulus</th>
<th>Shear modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gibson et al., Eq.(2)</td>
<td>(k') [-]</td>
<td>0.3</td>
<td>0.01</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>(T_{g.mech} ,{^\circ}C)</td>
<td>207.4</td>
<td>112.6</td>
<td>50.6</td>
</tr>
<tr>
<td>Mahieux et al., Eq.(3)</td>
<td>(m)</td>
<td>5</td>
<td>9</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>(T_o ,{^\circ}C)</td>
<td>1000</td>
<td>403.8</td>
<td>330.0</td>
</tr>
<tr>
<td>Wang et al., Eq.(4)</td>
<td>Case (ii)</td>
<td>20 (\leq T &lt; 60)</td>
<td>A=1, B=20, C=100, n=1.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>60 (\leq T &lt; 120)</td>
<td>A=0.6, B=60, C=427.9, n=1.0</td>
<td></td>
</tr>
<tr>
<td>Present study, Eq.(5)</td>
<td>B [-]</td>
<td>(-1 \times 10^{-5})</td>
<td>4</td>
<td>-4.3</td>
</tr>
<tr>
<td></td>
<td>C [-]</td>
<td>-0.25</td>
<td>0.02</td>
<td>5.5</td>
</tr>
</tbody>
</table>

5. Conclusions

This study aimed at (i) characterizing the variation of mechanical properties of GFRP pultruded material from ambient temperature up to 250\(^\circ\)C, and (ii) assessing the accuracy of different empirical and phenomenological models published in the literature, as well as an empirical model.
proposed in this study, to reproduce the variation pattern as temperature function, that was verified for the mechanical properties that were studied. The following conclusions are drawn:

- Experimental data reported herein confirmed that in the range of temperatures analysed, which includes the glass transition process of the polymeric matrix, the GFRP material is much more vulnerable under shear and compression than under tension. Therefore, for the highest temperature tested, the 250°C, was measured tensile strengths on the order of 50% of the resistance value measured at room temperature, while in the case of shear and compression resistance were measured in the order of 10% and 5% of this value, respectively.

- The stiffness reduction is lower than the strength reduction, namely for tensile loads, where the loss of stiffness was only relevant for the highest temperature tested (220°C); even though, for this temperature, the stiffness retention was 89%. Also here the stiffness in shear or compression is more affected by temperature than in tension - for 250°C it represents 56% and 22% compared to the value measured at room temperature, but still less affected than the resistance.

- The results obtained in this study are also in line with previous data reported in the literature. This agreement of results attests the precision of the experimental procedures used and validates the results obtained.

- All descriptive models analysed were able to simulate the pattern of variation of tensile, shear and compressive strengths as a function of temperature, providing reasonably accurate estimates of experimental strength data. The less accurate estimates were obtained from the phenomenological model proposed by Keller and Bai [10,11]; for some temperature ranges and types of loading this model was non conservative. The same conclusion can be drawn from the modeling performed for the elastic modulus and shear modulus.

- The model suggested in the present study, based on Gompertz [6] function, proved to be a good alternative to the others and seems to be particularly well suited to describe the degradation of shear and compressive strengths and the tensile elasticity modulus.

References


