

FUEL EFFECT ON THE TENSILE STRENGTH EVOLUTION OF CARBON FIBRES UNDER DIRECT FLAME ATTACK

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Keywords: carbon fibre, tensile strength, oxidation

ABSTRACT

PAN-based carbon fibres have been subjected to simultaneous tensile load and open flame attack using a hybrid tow-based approach. Their oxidation behaviour has been determined under controlled atmosphere conditions through Thermogravimetric Analysis (TGA) whereas Scanning Electron Microscopy (SEM) was used to evaluate the morphology of burnt fibres. The effect of a methane-based non-premixed turbulent flame on the tow tensile strength has been assessed by varying the applied load and time of fire exposure. The evolution of residual tensile strength with respect to time is mainly attributed to a heterogeneous fibre diameter reduction within the carbon fibre tow along with localized pitting and channeling due to catalytic reactions.

1. INTRODUCTION

The high specific strength of Polymer Matrix Composites (PMCs) has led to their extensive use in civil aviation. Initially found in small shrouds and fairings, nowadays they can comprise half of the structural weight of modern commercial aircraft, including demanding high temperature and high humidity applications [1]. Nonetheless, their potential flammability is a well-known problem that is often alleviated using heavy protection, thus hindering the fuel savings that such materials can bring. PMCs may be subjected to extreme heat conditions, e.g. as part of aircraft firewalls, hence regulations and certification guidelines prescribe that fire testing must be performed as close as possible to real fire conditions. When exposed to heat or a fire source, the first concern related to PMCs is the matrix degradation and further decomposition (pyrolysis), preventing loads from being transferred, both at the micro- (interfibre) and mesoscopic (intertow and interply) level. Hence, shear and compressive failure may be rapidly observed once the matrix exceeds its glass transition temperature (T_g) and starts to decompose. However, tensile loads can still be borne by the fibres aligned with the exerted forces.

In this regard, carbon fibres are extensively used in high strength PMCs. They are heat resistant under inert conditions, exhibiting an elastic behavior at relatively low temperatures, developing into viscoelastic and viscoplastic behavior at medium and high temperatures, respectively [2]. Nonetheless, they are very reactive in the presence of air and they start oxidizing at approximately 400 °C [3, 4]. Several research groups have investigated the evolution of tensile properties of carbon fibres in oxidative conditions. However, most have relied on experiments under controlled atmosphere, with electric heating of carbon fibres exposed to a hot oxidizer flow or exposed to radiant heat sources. For instance, Sauder *et al.* [2] analyzed the mechanical behavior of PAN-, pitch- and rayon-based fibres at very high temperatures (at least 2000 °C for all of them) under inert conditions. Feih and Mouritz [4] characterized the tensile strength and modulus of PAN-based fibres after being heat treated in an oven up to 700 °C under oxidative and inert conditions. Tong *et al.* [5] followed a similar approach but using a pipe oven. Bertran *et al.* took fibres subjected to Thermogravimetric Analysis (TGA) under oxidative conditions using dry and wet air between 400 and 600 °C [6].

Contrarily, when real fire conditions are taken into account, research groups have focused on PMCs which mainly consider matrix-driven thermal properties (e.g. post-fire flexural strength) using small scale laminates [7] or, if simultaneous fire and mechanical loading is considered, they also address matrix-driven compressive failure [8]. Others have only focused on the morphological analysis of the fibrous residues. Nonetheless, the influence of flame chemistry and the interaction with mechanical parameters remain uninvestigated.

The goal of the present work is to provide insights into the influence of flame chemistry on carbon fibre oxidation in the context of fire resistance, using a gaseous fuel and analyzing the tensile strength evolution with simultaneous fire and tensile load followed by post-fire tensile tests until failure.

2. EXPERIMENTAL

2.1. Materials

A standard modulus Polyacrylonitrile (PAN)-based carbon fibre (Tenax®-E HTS45 P12 12K 800 tex, from Teijin Carbon) was selected for the present investigation. According to the manufacturer, it has a Polyetherimide (PEI)-based sizing, which is compatible with thermoplastic (TP) resin systems. It has a reported tensile strength and modulus of 4500 MPa (650 ksi) and 235 GPa (35 Msi), respectively [9]. Tows were extracted directly from the spool and were used as is.

2.2. Microstructure and dimensional analysis

The morphology and size of virgin and burnt fibres were assessed using Scanning Electron Microscopy (SEM, JEOL JSM7600F), with a 5kV acceleration voltage. In both cases, no coating was required to inspect the samples due to carbon's intrinsic conductivity. Initially, a fibre tow was extracted from the spool to determine the fibre average size that would be used for strength calculations. A 7.07 μm (SD = 0.42) average diameter was determined after 41 different measurements taken from the unburnt 12,000-filament tow. Post-fire fibre analyses were performed upon completion of the burning experiments (see 2.4.2) using the same measurement conditions.

2.3. Tensile strength of carbon fibres

The nature of carbon fibre failure leads to brittle fracture, which follows a 2-parameter Weibull distribution:

$$P_F(\sigma) = 1 - \exp \left[- \left(\frac{\sigma}{\sigma_o} \right)^m \right] \quad (1)$$

where $P_F(\sigma)$ is the probability of failure of the material subjected to uniaxial tensile stress σ ; m and σ_o are the shape (scatter) and scale (mean fracture stress) parameters, respectively. ASTM C1239 [10] is widely adopted to calculate and report these values, assuming a single-flaw failure or stable flaw population and no slow crack development. Such values are mainly obtained from single fibre tests at very short gauge lengths, which in turn lead to minimized variability and flaw probability.

2.3.1. Combined Tensile-Fire test rig

A 250 kN load cell (MTS 661.23A-01) was coupled on one side to the load frame of a universal testing machine using an alignment fixture (MTS 609) and, on the other side, to a 25kN force transducer (MTS 661.20E-01).

Figure 1 shows the general arrangement of the tensile test rig, consisting of a fibrous specimen (a), a burner (b), the drum fixtures (c) and an enclosure (d). Grips (e) are integrated with the drum fixture. For the present study, a gas-fueled non-premixed burner was used (see 2.4.2).

The enclosure protects the components of the tensile machine near the flame from excessive heat and soot deposition during the tests. As a result, the minimum gage length is limited by the enclosure height. This made it impossible to conduct single-fibre tests, so a tow-based tensile test methodology was chosen. Tow-based testing requires either consolidated or impregnated carbon tows as per ASTM D4018 [11]. As these would interfere with the objective of the present work, which is to investigate the behavior

of carbon fibre exposed to an open flame, a hybrid testing method is proposed to prevent fibre damage as much as possible. This approach eases fibre handling and is still statistically representative as it involves weak fibres that would break under single-filament-test manipulation [12, 13]. 50 kN rope grips (Universal Grip Co., Salem, MA) were used. Although traditionally employed in rope tensile tests (ISO2307 or ASTM A931), these fixtures were deemed appropriate for holding loose fibre tows whilst significantly reducing fibre damage. The fibre tows are turned around the drum in a single loop and gripped by the integrated clamps (Figure 1, detail e). The gauge length (GL) between vertical tangents was 640 mm for all experiments.

Careful handling of fibre tows was imperative. Challenges associated to tow-based tests are noteworthy: from workmanship issues (possible misalignment of fibres and overgeneralization of fibre diameter), up to biased strength calculation due to non-linear elastic effects [12]. Moreover, the tensile strength is expected to decrease with increasing GL. This reduction could be validated if values at lower GLs were known, given the linear behavior in the log-log scale [14]. However, this information is not available and requires an additional testing campaign which falls outside of the scope of the present work.

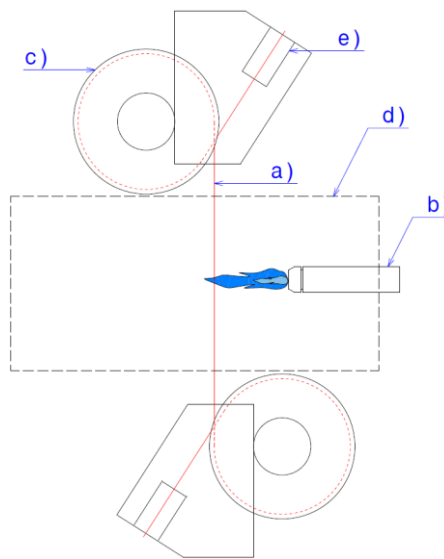


Figure 1: Tensile load + fire test arrangement showing tow specimen (a), burner (b), drum fixture(c), fire enclosure (d) and clamp (e).

2.3.2. Sample preparation

A 1820 mm-long (minimum) tow was drawn from the spool for each tensile test. Given the 640 mm GL, approximately 590 mm are to be spared for each end which equals one half-turn around the drum and hem for the gripped section. To ease tow preparation and installation, the following procedure was employed:

1. *Fray protection* - The open tow end on the spool is held together with a self-wrapped (i.e. half-fold), 2" pressure sensitive tape over the whole grip length (Figure 1, detail e) with a surplus (~30 mm) to allow for correct installation. The tow width is to be kept approximately equal the tow's original packing condition (for the present experimental setup, 9.5 ± 1.6 mm).
2. *Cut length* - The tow is drawn from the spool and rolled once on the upper drum fixture, then gently pulled down and rolled once again on the lower drum fixture up to the lower grip to attain full length.
3. The tow is then gently unwound and all the fibres are manually aligned. Step 2 is restarted whilst tow twisting is avoided. The step is repeated until the tow shows a homogeneous width.
4. *Fibre slack reduction* - The tow is visually assessed to detect loose fibres. The lower tow end is finally wrapped as in step 1 and fixed with the grip. Both grips are to be tightened once the tow is aligned and slack is kept to the minimum.

2.3.3. Pre- & Post-fire tensile testing protocol

To measure tensile strength, monotonic tensile tests with constant crosshead speed were carried out. According to ASTM D4018, a crosshead speed of 250 mm/min is the maximum acceptable for fibre tow tests. For the single-fibre tests, however, ASTM C1557 [15] requires a single-fibre-failure to happen within 30 s, suggesting an initial speed of 8×10^{-3} mm/min. A set of tests were carried out to determine an optimal crosshead speed (Table 1). The 640mm GL tests yielded the highest strength at a speed of 100 mm/min, with little variation from the rest of the results. Therefrom, tensile tests used this setting. Weibull analysis of virgin and burnt fibre tows was carried out using a minimum of 50 samples to ensure statistical relevance [12]. Weibull parameters were graphically obtained through linear regression.

Crosshead Speed (mm/min)	Failure force (N)	Std. Dev.	Ultimate Tensile Strength (MPa)
10	603.2	28.1	1280.4
25	592.4	22.4	1257.6
50	597.4	13.2	1268.2
100	614.2	21.5	1303.9
175	604.5	24.3	1283.3
250	600.1	21.7	1273.9

Table 1: Preliminary test results for a 640 mm gauge length ($n = 10$, per speed value).

Table 2 shows the parameters that were selected for simultaneous fire and tensile load tests. Two load levels, corresponding to approximately 10% and 20% of the unburnt tow's Ultimate Tensile Strength (UTS), were selected to compare the effect of fire exposure time. Five tests for each condition were carried out. The load was kept constant throughout the test which means that, in case of progressive fibre failure, stress would increase likewise. Nonetheless, the present work analyzes the UTS evolution based on the post-fire residual strength. Minimum and maximum load values were monitored and recorded to ensure constant loading, which was kept at $\pm 10\%$.

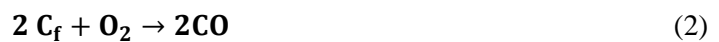
Fire exposure time (s)	Constant Load - virgin tow UTS ratio (%) [*]
60	10, 20
120	10, 15, 20, 35, 50
180	10, 20
240	10, 20

^{*}10 (9.76); 15 (14.65); 20 (19.53) ; 35 (34.19) ; 50 (48.84)

Table 2: Simultaneous tensile load & fire exposure conditions (real values shown in parentheses).

2.4. Carbon fibre oxidation

Carbon fibre (C_f) oxidation and gasification is driven by two major competing reactions yielding either carbon monoxide or carbon dioxide [16]:



and



Carbon fibres oxidize at different rates depending on the type of fibre (PAN-, pitch-, rayon-based), degree of graphitization (usually translated into strength and modulus) [17], fabrication process (precursor-dependant chemical processes) and, when found in a composite, the nature and additives of the matrix itself. All these variables will translate into uniform (around the periphery of the fibre) or localized (on preferential sites on the fibre surface) removal of fibre material. Intense localized reaction rates are believed to be caused by the presence of impurities (i.e. alkali metals) in the fibre, resulting in enhanced kinetics for the above reactions through catalytic effects and in the observation of fibre pitting and channeling [18, 19].

2.4.1. Thermal Analysis

The oxidation kinetics of the carbon fibres was analyzed with a thermogravimetric analyzer (TGA/DSC 1, Mettler Toledo), allowing for simultaneous Thermogravimetric Analyses (TGA) and Differential Scanning Calorimetry (DSC) measurements. Fibres were extracted from a tow and tested using a 70 μ l alumina crucible, to avoid catalytic reactions associated with the use of platinum [20]. Tests started at room temperature going up to 1000 °C following three different Heat-up Rates (HURs): 10, 25, and 50 °C/min in an oxidative atmosphere (60 ml/min air flow) as well as a single reference test in inert atmosphere (60 ml/min N₂ flow) at 10 °C/min.

2.4.2. Fire tests

The fire tests are based on a surface mix blowtorch (Nortel RedMax) supplied with a carbon dioxide (CO₂)-diluted fuel/oxidizer mix based on 45% methane (CH₄) and 45% oxygen (O₂). The experimental configuration is described in details elsewhere [21]. The distance from the burner exit to the surface spanned by the fibre tow is 140 mm. The burner is calibrated for the temperature and heat flux achieved at this position which are, in average, 1100 °C and 116 kW/m², respectively. The former is measured using K-type thermocouple while the latter is obtained from a water-flow calorimeter, similar to the one prescribed in FAA's AC-20-135 [22]. As described in Table 2, a constant load is imposed to minimize fibre slack and to promote a uniform fibre oxidation throughout the flame-impinged area. Additional plain fire tests without load applied were performed in a separate test rig. Contrary to the vertical alignment in the tensile setup, the fibre tows were here installed horizontally on two 12.7 mm-diameter posts (~460 mm separation) to ease burn tests as well as installation on the sample holder. The fibre tows were shielded from the flame until the burner reached the desired steady conditions and then subjected to fire attack at different intervals. The oxidation was stopped by quenching with a CO₂ jet stream after 60, 120, and 300 s. The post-fire fibre morphology was investigated using SEM (§ 2.2).

3. RESULTS

3.1. TGA/DSC

Figure 2 shows the normalized weight evolution and weight derivative with respect to temperature (Figure 2a and Figure 2b, respectively) along with the heat flow (Figure 2c) for the selected HURs and atmospheres. Samples in oxidative atmosphere show a one-step weight loss behavior (Figure 2a) with an onset temperature at approximately 500 °C. Such behavior can be described by an Arrhenius-type equation [23]. Figure 2c shows a positive heat flow, indicating that exothermic reactions take place. These results are consistent with the aforementioned chemical reactions. Since all experiments were performed well above the 500 °C threshold, severe fibre oxidation must be anticipated. However, detailed chemical kinetics analysis is beyond scope of the present work, but would be necessary for a thorough modelling of the fibre degradation rate.

3.2. SEM analysis

The burnt fibre tows investigated are obtained from plain fire tests without load (see § 2.4.2) with a horizontal alignment of the fibres. These tests suggest a degradation through two stages. Immediately after starting exposure to the flame (Figure 3a), a few carbon fibres may fail immediately due to the

combination of flame jet velocity and the aggressively oxidative conditions. Fibre diameter reduction and flaw creation due to the oxidizing flow then leads to progressive failure (Figure 3b). The packed fibre arrangement prevents homogenous diffusion of oxidative species within the tow and promotes heterogenous oxidation, starting at the periphery of the tow. This setup is thus more representative of woven fibre mats than single fibres that would fail earlier under the same conditions.

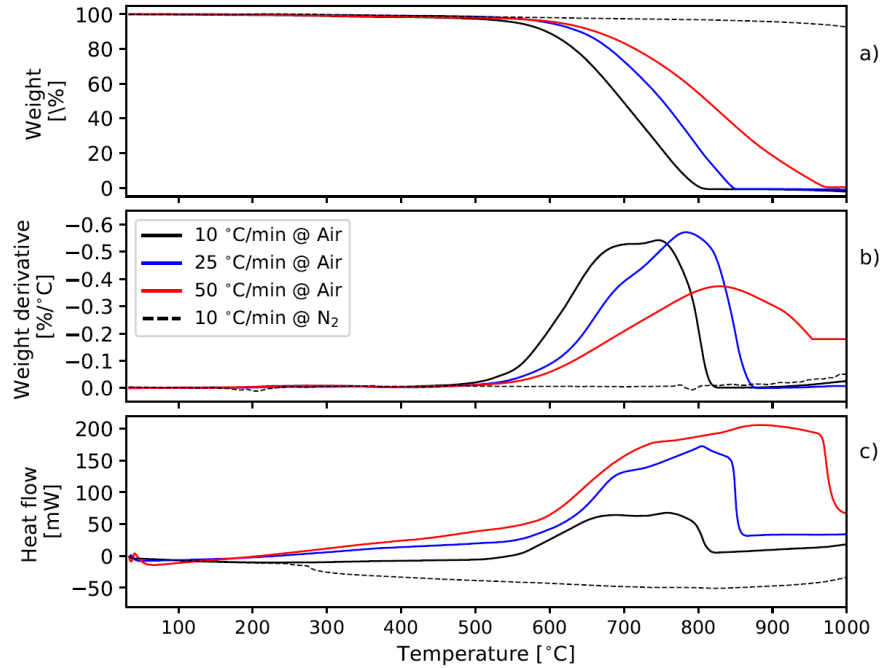


Figure 2: Simultaneous Thermal Analysis (TGA+DSC) results of HTS45 fibres extracted from a tow.

The partially-oxidized samples (Figure 4b through Figure 4f) show severe pitting and channeling, compared to the virgin carbon fibre (Figure 4a). These localized phenomena are assumed to be a result of catalyzed gasification and oxidation due to metallic impurities found in the fibre, which may be traced back to precursor preparation [16, 18, 19]. Moreover, in the case of PMCs, non-uniform oxidation could be accentuated by other catalytic reactions caused by the matrix's pyrolysates [24].

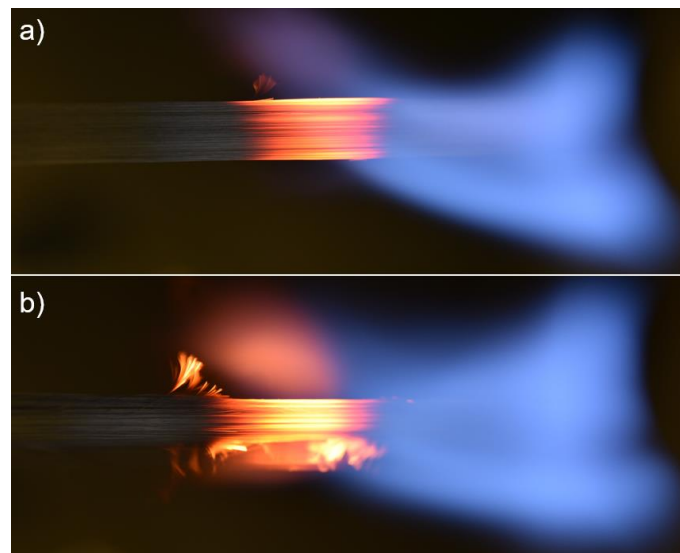


Figure 3: Incandescent full tow (a) and heavily oxidized / partially broken tow (b) whilst exposed to a CH₄-based non-premixed turbulent flame.

A very heterogeneous diameter distribution was observed in burnt fibres, thus making it virtually impossible to determine an average value that could lead to a direct calculation of strength reduction. This behavior is consistent with results obtained from conditions representative of fire hazards [18]. The progressive tow failure may be traced back to a partial fibre overlap which interferes with the diffusion of oxidizing species and heat within the tow. The direct assessment of the Ultimate Tensile Strength (UTS), obtained from quenching tests with increasing flame exposure durations, provides an appropriate means to study the degradation process and potentially the effect of the flame chemistry.

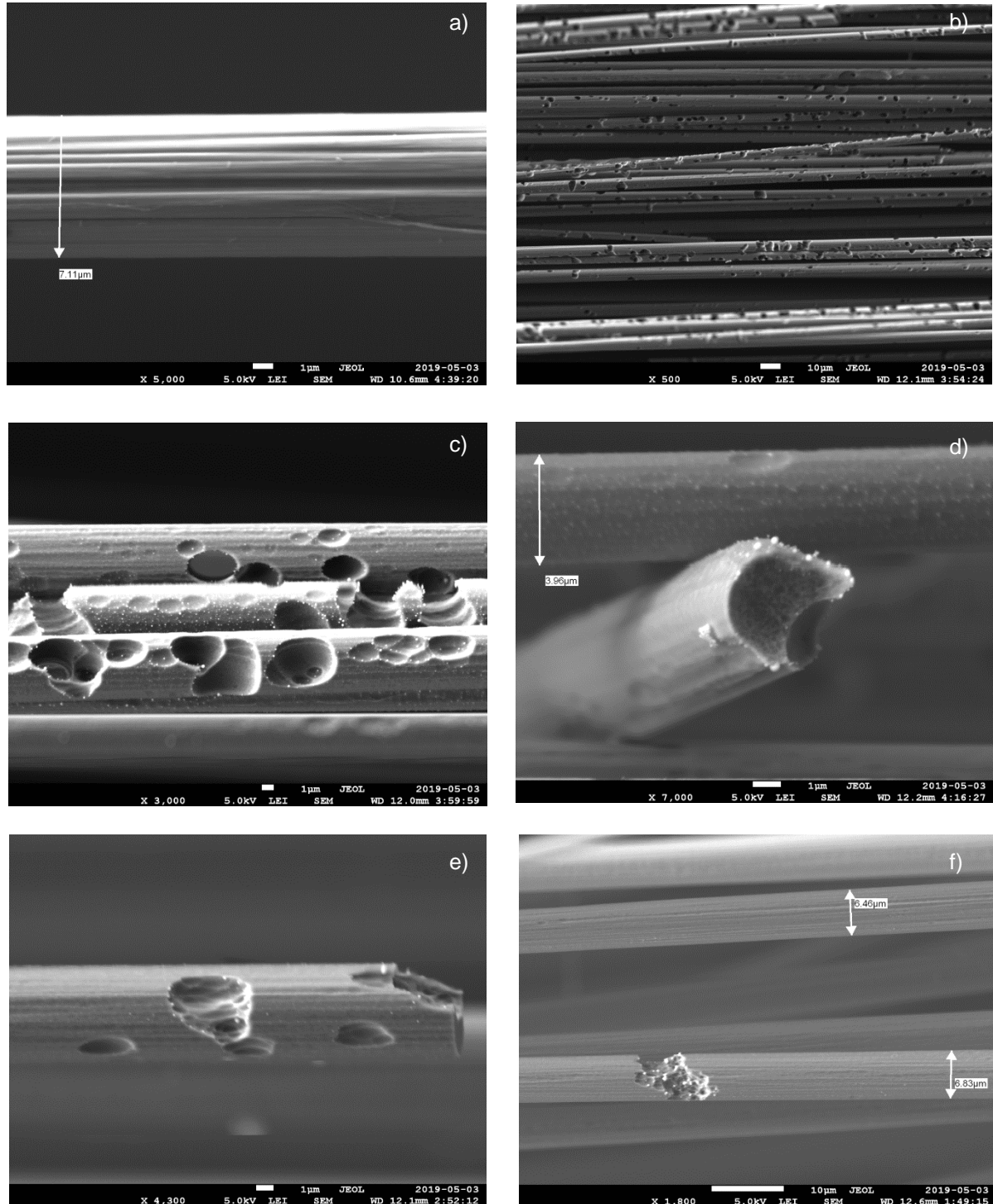


Figure 4: SEM of HTS45 carbon fibres: virgin (a) and subjected to a CH₄-based flame for 120 s (b & c), 240 s (d & e) and 300 s (f).

3.3. Tensile tests

Figure 5 shows the Weibull plot of virgin fibre tows based on their UTS. The average strength of the HTS45 tow-based tensile tests corresponds to 29.4 % of the value reported by the manufacturer. This reduction is expected for such a large GL, as length increases the flaw probability for each fibre [12, 14, 25]. A high Weibull shape parameter (m) indicates low scatter of the tensile strength results [26, 27], suggesting test repeatability. The m value obtained in this work could not be directly compared to other values due to data unavailability in the literature, which is chiefly based on small GLs and single-fibre approaches. Therefore, this aspect deserves attention in future works. All fibre tows failed within the vertical mid-section of the strand and to the naked eye, no single test exhibited failure around the grip and drum section.

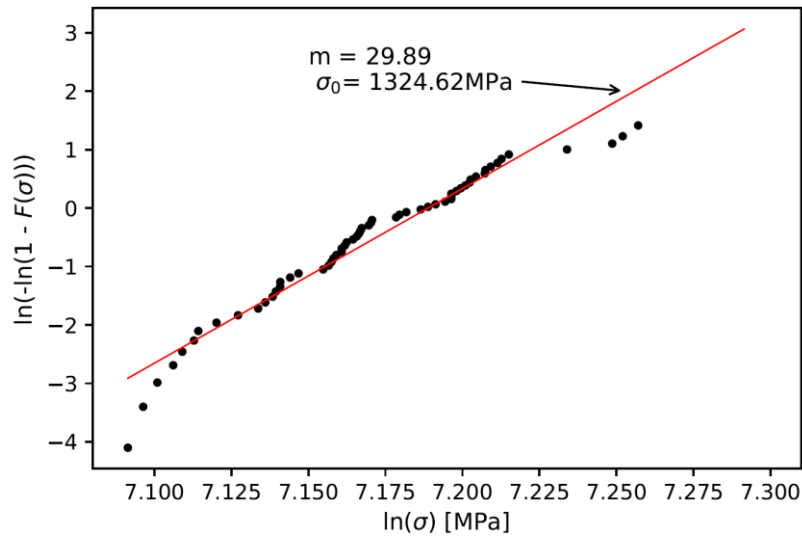


Figure 5: Weibull plot of virgin HTS45 fibre.

Figure 6 shows the evolution of a fibre tow subjected to a constant load (20 % of the virgin fibre tow's UTS) kept for 240 s. As seen in Figure 6a, the fixture's drum proved useful in keeping the fibre tow well distributed (widthwise) during the test, as indicated in § 2.3.2. Immediately after the test started, fibre oxidation was visible on the tow edges as these are the least protected areas (Figure 6b), leading to progressive failure towards the tow core (Figure 6c). Finally, Figure 6d shows the fibre tow still under load after flame removal. For each test, the remaining fibres were secondarily loaded until rupture using the same crosshead speed described in §2.3.3.

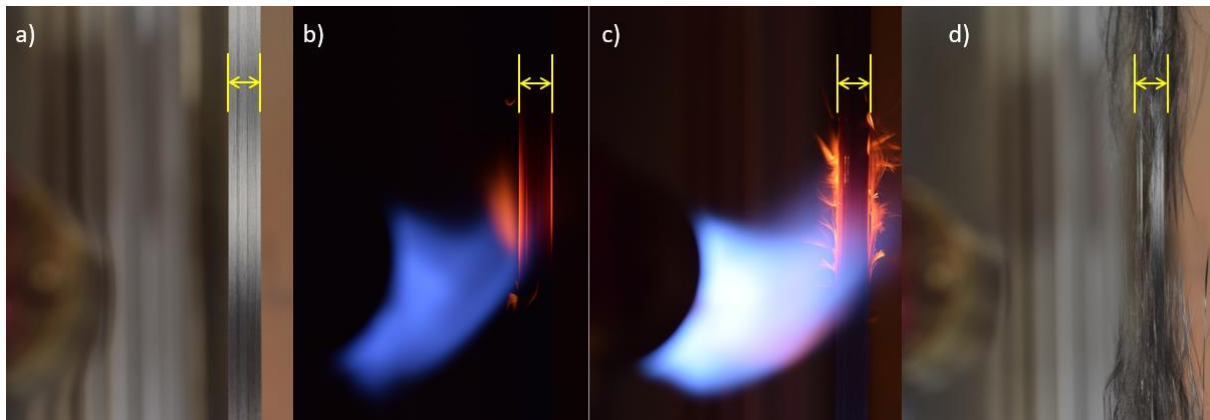


Figure 6: Fire & load test sequence: fibre tow in tension prior fire attack (a), beginning of test (b), partial fibre rupture (c) and fibre tow still under tensile load after fire attack before total failure (d). Original tow width is indicated by vertical lines for clarity.

A first strength comparison is given in Figure 7, which shows the normalized residual strength of fibre tows loaded at 10 % and 20% of the unburnt tow's UTS. In both cases, a constant force was exerted regardless of the amount of fibres that failed during the test. For a 10% load, an approximately linear decrease can be observed on the post-fire UTS with respect to time, while the 20% load yielded similar average strength values but with higher scatter.

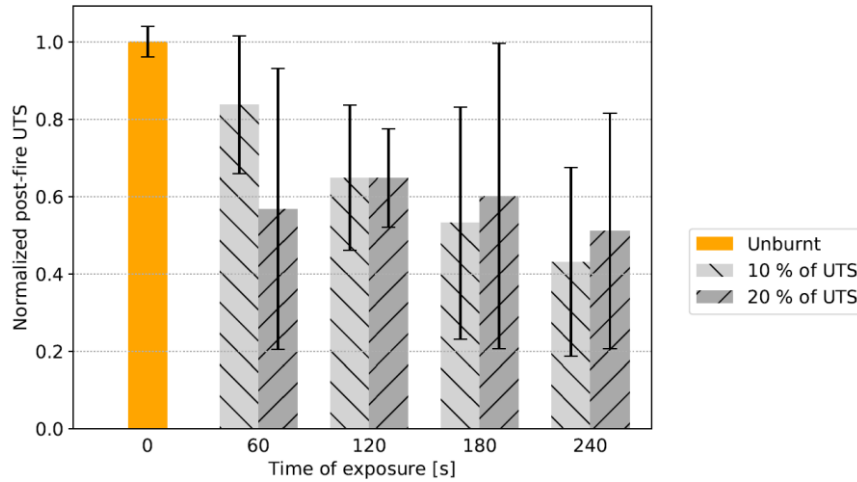


Figure 7: Residual tensile strength of fibre tows burnt at different intervals: 60, 120, 180 and 240 s. Values have been normalized with respect to unburnt tow's UTS.

The second case is presented in Figure 8, which shows the effect of different load levels considering the same time of exposure. Normalized results with respect to virgin tow strength suggest that residual strength was not strongly affected by the load for the same time-of-exposure.

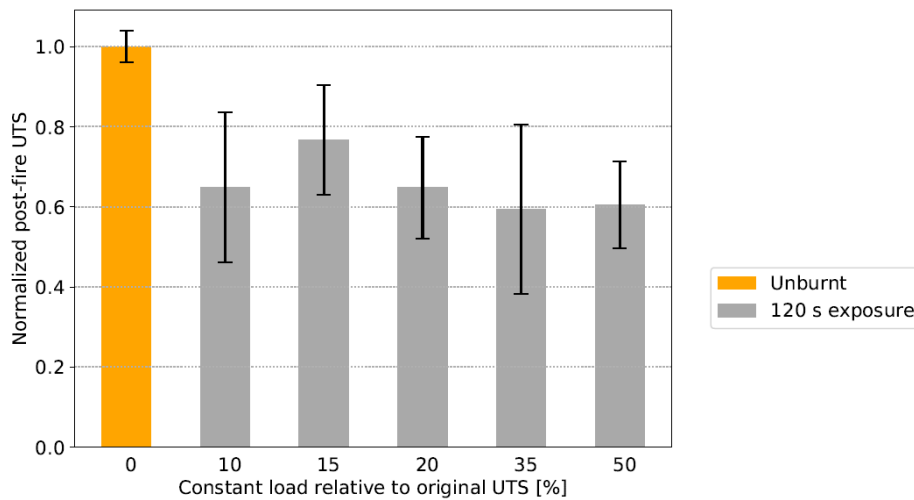


Figure 8: Residual tensile strength of fibre tows exposed to fire for 120 s at different load levels.

4. CONCLUSIONS

Carbon fibres have been subjected to severely oxidative conditions under both controlled atmosphere (TGA) and open flame conditions. Thermal analysis under controlled conditions provided an overview of the oxidation onset temperature, as well as the associated rate and level of exothermicity. Surface defects along with fibre diameter reduction have been correlated with a decrease in residual strength.

The hybrid test method combined requirements for single fibre and fibre tow tests, providing results in good agreement with the expected UTS evolution. On the one hand, the virgin fibre tow's tensile strength show low scatter owing to a large number of tests. Large scatter is observed on the burnt fibre

tows' post-fire UTS, which is explained by the plethora of parameters that can affect the outcome, namely the intra-tow fibre arrangement which translates into protection amongst fibres from oxidative species and a higher probability of fibre failure due to large GLs. Undoubtedly, further detailed statistical analysis is required to validate this test approach.

Oil-based burners are nowadays the only means approved for the evaluation of the fire resistance of aircraft structures, including composite-based constructions [22]. Therefore, a comparison between gas- and kerosene-based flames is needed for future research.

ACKNOWLEDGMENTS

The authors want to acknowledge the financial support from the Natural Sciences and Engineering Research Council of Canada (NSERC/CRSNG) grant no. CRDPJ 478687-15, and the partners from CRIAQ ENV-708 project. Pablo Chávez Gómez thanks the National Science and Technology Council of Mexico (CONACYT) for his doctoral Scholarship.

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