

# TENSILE STRENGTH EVALUATION OF CARBON FIBER BUNDLES USING CAPSTAN GRIPS AND DIGITAL IMAGE CORRELATION

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**Keywords:** Carbon fiber bundles, Tensile testing, Digital image correlation

## ABSTRACT

The tensile strength of carbon fibers (CFs) is typically assessed via monofilament tests, requiring meticulous specimen preparation and large sample sizes. The use of CF bundles eases their evaluation by dramatically lowering the number of tests required. However, standard tests still require elaborate specimen preparation and evaluation. To address these challenges, we propose a technique using dry CF bundles, capstan grips and digital image correlation (DIC). This work aims at easing the specimen preparation by winding the CF bundle around two capstans and measuring strain directly on the specimen by optical means. Additionally, reliance on extensometers or machine compliance is avoided.

Tensile tests were performed on CF bundles using capstan grips as fixation means. To validate the repeatability of the method prior to relying on DIC, tests were carried out at three crosshead speeds ( $v = \{10, 50, 200\} \text{ mm} \cdot \text{min}^{-1}$ ) and two gauge lengths ( $GL = \{200, 420\} \text{ mm}$ ), with a sample size of  $n = 50$  for each test condition. Maximum load values ( $P^*$ ) obtained at  $GL = 420 \text{ mm}$  were  $\sim 14 \%$  lower than those at  $200 \text{ mm}$ , corroborating the impact of  $GL$  on the CF bundles' mechanical properties. Crosshead speeds showed a certain influence over  $P^*$  at  $GL = 200 \text{ mm}$ , with minimal differences observed at  $420 \text{ mm}$ . Weibull parameters obtained therefrom suggest good repeatability. DIC was finally introduced to enable direct strain measurement. The technique was validated at  $v = 10 \text{ mm} \cdot \text{min}^{-1}$  and  $GL = 420 \text{ mm}$ , allowing to determine the maximum number of filaments loaded at the beginning of each test, the CF bundle mean strength as well as the Weibull modulus and average tensile strength of individual CFs. Overall, the technique shows a high potential for screening purposes or when standard evaluation means are not possible.

## 1 INTRODUCTION

The tensile strength of carbon fibers (CFs) is typically assessed by means of monofilament testing at short gauge lengths ( $GL$ ) [1]. However, this technique is time-consuming and needs careful specimen preparation and handling. Moreover, due to the brittle nature of CFs, the statistical analysis based on the Weibull distribution [2] requires a large number of tests, e.g.,  $n \geq 20$  [1] or  $n \geq 80$  [3], with Weibull parameters being highly dependent on the sample size [3].

Other techniques have been developed to facilitate the extraction of fiber tensile properties. For instance, bundle-based tests provide alternative means, requiring less meticulousness during specimen preparation and handling. A first approach relies on consolidated fiber bundles, i.e., resin-impregnated specimens. In this case, the load is redistributed *locally* owing to the polymeric matrix, analogous to unidirectional composites [4]. Only a small number of valid tests are required ( $n = 4$ ) [5], although the specimen preparation can prove to be time-consuming. The second approach considers unconsolidated fiber bundles, where the load is shared and redistributed *globally* [6]. A standardized procedure has been recently defined [7], requiring bonding the fiber bundle ends to metallic cylinders or to a window-type holder. A  $GL$  of  $200 \text{ mm}$  is to be considered, differing from the monofilament test  $GL$  [1] by one order of magnitude. A very small sample size ( $n = 3$ ) is needed if the fiber bundle elongation is precisely

monitored, for instance, using an extensometer next to the grips. Otherwise, the compliance of the tensile test machine must be determined considering at least two additional GLs (100 mm and 300 mm), increasing the number of tests. The method considers the slope of the linear part of the force-displacement curve as well as the maximum force and displacement values to determine the number of fibers loaded and the fibers' Weibull parameters.

Strain measurement and quantification of load-bearing fibers are critical to an accurate calculation of tensile properties. Optical strain measurement methods, e.g., Digital Image Correlation (DIC), are an attractive avenue to be explored since they might exert minimal to no influence on the fiber breakage behavior. For instance, Depuydt *et al.* [8] used DIC to obtain the tensile properties of steel, flax, and bamboo fiber bundles. They attached optical flags, i.e., speckle patterns, close to the grips using a correction pen's paint. Alternatively, Callaway and Zok [9] painted speckle patterns directly on SiC fiber bundles and measured strain by means of DIC. Additional information such as the number of loaded fibers or their failure throughout the test can also be determined via acoustic emission [10] or electrical resistance measurement [11].

In this work, we propose a modified bundle-based test method to determine the average tensile strength of CFs, aiming to ease the specimen preparation and fixation using capstan grips. Inspired by the works of Depuydt *et al.* [8] and Callaway & Zok [9], our method relies on direct measurements of the bundle strain by means of DIC using modified markers and strain averaging. For validation purposes, we evaluate fiber bundles of a commercial CF. We present and discuss the results, concluding with the advantages and shortfalls of our proposed methodology.

## 2 EXPERIMENTAL

### 2.1 Materials and specimen preparation

An intermediate modulus polyacrylonitrile (PAN)-based CF (IM7 12K, Hexcel Corp.) was selected for evaluation. The tensile strength, modulus and diameter of this CF type are 5688 MPa, 276 GPa and 5.2  $\mu\text{m}$ , respectively, as reported by the manufacturer [12]. CFs were tested in the form of bundle as extracted from the spool. Bundles were handled using non-textured nitrile gloves to avoid damaging fibers, ensuring that most of the fibers were intact by the time of testing. The first end of the bundle was taped using pressure-sensitive polypropylene tape (low-tack, 0.06 mm-thick). Fiber bundles were drawn up to a length that allowed to roll them once around each capstan drum and get attached at the capstan's grip area. Once rolled around the second capstan, the other end of the fiber bundle was taped in a similar fashion prior to attaching it.

### 2.2 Tensile testing

Monotonic tensile tests were carried out on a universal test machine (INSIGHT, MTS) using a 1 kN load cell (Model 569327-02, MTS) at three constant crosshead speeds ( $v = \{10, 50, 200\} \text{ mm} \cdot \text{min}^{-1}$ ) and two GLs ( $\{200, 420\} \text{ mm}$ ). A pair of capstan grips (50 kN model, Universal Grip Co.) with an effective diameter of 270 mm was selected to attach the specimens, i.e., CF bundles. For each test, grips were tightened manually. Force and crosshead displacement were recorded at 10 Hz using a commercial interface (TestSuite TW, MTS).

Figure 1 shows a conceptual representation of the experimental arrangement comprised by the two capstan grips, the fiber bundle and, when applicable, the cameras used for DIC (described in §2.2.2). In this case, the GL is the vertical distance between the drums' horizontal centerline, roughly corresponding to the tangential points of the fiber bundle and the drums' surfaces.

#### 2.2.1 Tests without DIC

A large test campaign considering a sample size of  $n = 50$  for each  $v$  - GL combination was initially carried out to determine the maximum load ( $P^*$ ) that the CF bundles could withstand. This allowed to determine the effects of  $v$  and GL on  $P^*$ , as well as to assess the repeatability of our technique applying Weibull analysis.

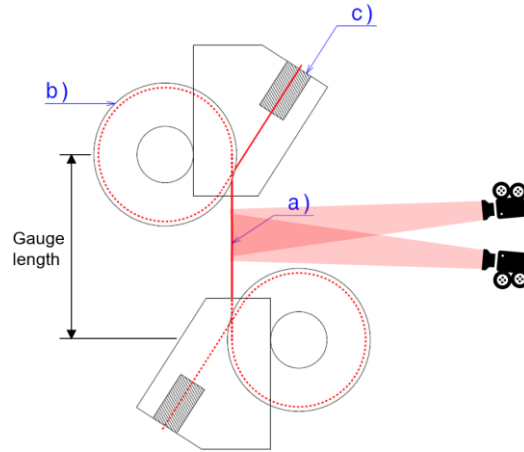


Figure 1. Set-up schematic showing the fiber bundle (a), capstan drum (b) and grip zone (c) of one of the two capstans as well as the two cameras used for DIC.

### 2.2.2 Tests with DIC

DIC provides the means to extract strain fields within the FOV. Tests with direct strain measurement helped to determine the average CF tensile strength considering three tests at  $v = 10 \text{ mm} \cdot \text{min}^{-1}$  and  $GL = 420 \text{ mm}$ . The details of the calculation method relying on DIC-based data are outlined in §3.

A pair of cameras (5 MP Point Grey, Correlated Solutions), each with a 1:1.8/75mm lens (HF75SA-1, Fujinon) were installed considering a vertical separation of 100-200 mm between them and 600-1000 mm from the fiber bundle. Images were acquired at a rate of 2-4 Hz and treated with a dedicated DIC software (VIC3D 9, Correlated Solutions), allowing coordination with the data acquisition system of the tensile test set-up described in §2.2. Three-dimensional calibration was performed using a 14x10-dots / 5 mm spacing target (7DF0D3016, Correlated Solutions). Speckle patterns were created on paper sheet using an ink roller (.007" dot size, Correlated Solutions). Synthetic rubber vacuum bag sealant (AT-200Y and 213-3, General Sealants) was used to affix the speckle pattern onto the fiber bundle within the GL with a 25-30 mm spacing between targets.

In the context of our technique, the evolution of the distance between any two points located within the markers can be determined by tracking any pair of points selected within the speckle patterns. Several virtual strain gauges (VSGs) with three different dimensions were placed aligned lengthwise with respect to the CF bundle. Figure 2 shows a typical distribution of fifteen VSGs used to measure strain between different regions of the marker. The VSGs are distributed along the CF bundle width in three sets of five, shown separately for clarity purposes. The VSGs on the first (left), second (middle) and third (right) sets are typically (41 to 45) mm, (32 to 36) mm, and (26 to 30) mm long, respectively. The first and third views focused on the outer and inner portion portions of the square markers, respectively, whereas the second set (middle) relied on a diagonal distribution.

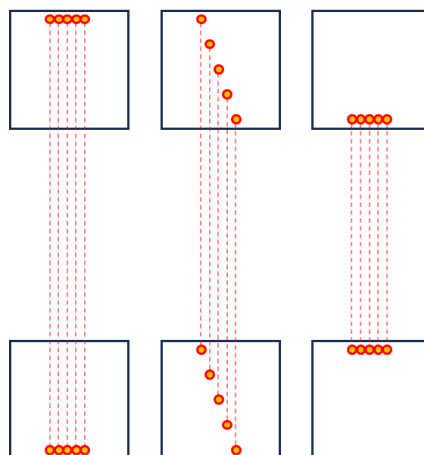


Figure 2. Set of VSGs (divided in three groups for clarity purposes).

### 3 CALCULATIONS

Weibull analyses of brittle materials including single CF filaments or CF bundles are traditionally carried out in terms of strength [9, 13]. As mentioned in §2.2, two types of tensile tests were carried out in this work. On the one hand, only  $P^*$  values were considered when strain was not directly measured via DIC. Although strength calculation was not possible, Weibull analyses were carried out. Weibull and cumulative density function (CDF) plots based on  $P^*$  values were deemed preliminary means to assess the effect of  $\nu$  and GL as well as the technique's repeatability.

When DIC was introduced to determine strain, it was possible to determine the several CF properties relying on the methodology proposed by R'Mili *et al.* [14]. This allowed to estimate the maximum number of fibers loaded during the beginning of each test ( $N_l$ ), the bundle mean strength ( $\sigma_b$ ) as well as the Weibull modulus ( $m$ ) (aka shape parameter) and the average strength ( $\langle\sigma\rangle$ ) of individual fibers. The analytical sequence proposed by R'Mili *et al.* is summarized in Equations (1) through (4) and is briefly described below in order to grasp the relevance of each value.

The number of fibers loaded during the initial part of the test is given by

$$N_1 = \frac{S_0}{A \cdot E_f} \quad (1)$$

where  $N_l$  is the number of fibers loaded at the beginning of the test,  $S_0$  is the initial slope of the Load-Strain curve,  $A$  is the cross-section area of a single filament, and  $E_f$  is the modulus of a single filament.

The Weibull shape parameter can then be obtained from the following relationship

$$\frac{1}{m} = \ln\left(\frac{S_0}{S^*}\right) \quad (2)$$

where  $S^*$  is the slope of the straight line created between the origin of the Load-Strain curve and  $P^*$ . Subsequently, the calculation of the bundle mean strength ( $\sigma_b$ ) is given by

$$\sigma_b = \frac{P^*}{N_1 \cdot A} \quad (3)$$

Finally, the average strength of the fibers ( $\langle\sigma\rangle$ ) can be determined based on  $m$  and  $\sigma_b$  values with the help of the gamma function ( $\Gamma$ ) following

$$\langle\sigma\rangle = \sigma_b \cdot (2.72 \cdot m)^{\frac{1}{m}} \cdot \Gamma\left(1 + \frac{1}{m}\right) \quad (4)$$

## 4 RESULTS AND DISCUSSION

### 4.1 Tests without DIC

Figure 3a shows the Weibull plot for all  $\nu$ -GL combinations from tests where DIC was not used. The  $x$ -axis values correspond to the natural logarithm of  $P^*$  values, whereas the  $y$ -axis corresponds to the Weibull probability, a.k.a. failure probability, given by  $\ln(-\ln(1 - F(P^*)))$ . The solid lines were determined by linear least-squares regression considering individual  $P^*$  values. From this plot, two-parameter Weibull distribution values were determined, i.e., shape ( $m$ ) and scale ( $P_0^*$ ), which correspond to the slope and the abscissa value at  $y = 0$  of the regression line, respectively. Figure 3b shows the CDF plot of the same ordered  $P^*$  values and their probability on the  $x$ - and  $y$ -axes, respectively. Here,  $P_0^*$  can also be understood as the value where the failure probability is 63.2%. Noteworthy, the Weibull plots obtained from tensile testing of brittle fibers or fiber bundles typically rely on strength values. However, in these tests where we assessed the repeatability of the technique, the number of loaded fibers is unknown at any moment and the true stress cannot be calculated nor inferred. Thus, Weibull parameters were obtained considering force rather than stress values.

Figures Figure 3a and Figure 3b are complementary of each other, clearly showing the effect of GL on  $P_0^*$ . In general, tests carried out at GL = 420 mm yielded ~14% lower values of  $P_0^*$  with respect to those at GL = 200 mm, regardless of the speed. Conversely, the effects of  $\nu$  are different for both GLs. On the one hand, small differences in  $P_0$  can be observed at GL=420 mm where the highest loads were generally reached at 50 mm·s<sup>-1</sup>, closely followed by tests carried out at 10 and 200 mm·s<sup>-1</sup>. These

differences are minimal yet discernible on both Figures, where the  $P_0^*$  value obtained at  $v = 200 \text{ mm}\cdot\text{s}^{-1}$  is only 3 % lower than its counterpart from  $v = 50 \text{ mm}\cdot\text{s}^{-1}$ . On the other hand, tests carried out at  $GL = 200 \text{ mm}$  yielded virtually the same results regardless of the speed, indicated by the generalized overlap between individual values. In this case, there are minimal differences between regression lines and CDF curves from Figures Figure 3a and Figure 3b, respectively.

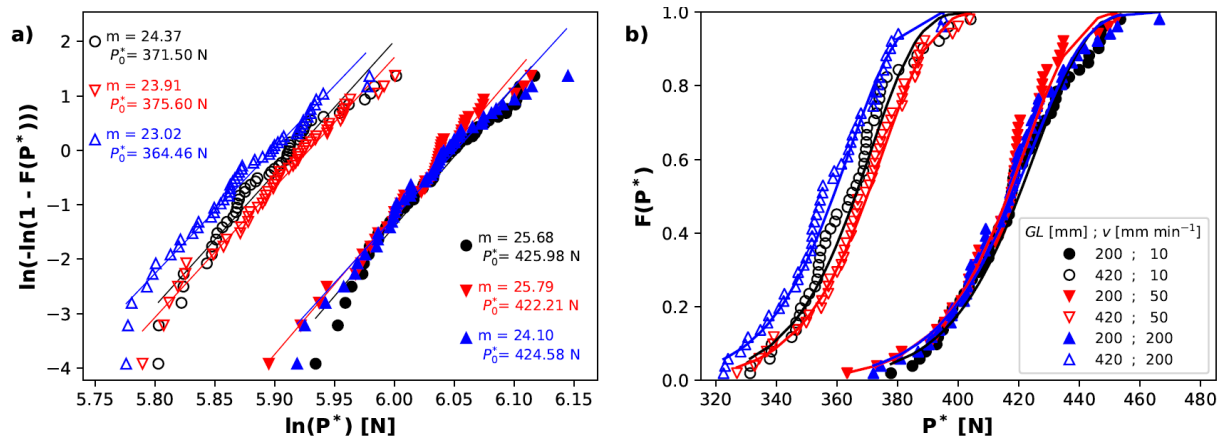


Figure 3. Weibull (a) and CDF (b) plots for IM7 CF bundles considering  $P^*$  values.

Further analysis of data contained in Figure 3a can help to determine whether our technique provides repeatable means to assess CF bundles. From a Weibull distribution standpoint, high  $m$  values are indicative of low data dispersion. In the context of this technique, this suggests good repeatability at both GLs. Furthermore, upon visual inspection, the apparent curvature created by the lowest values deserves attention. In terms of monofilament testing, this has been associated with small sample sizes, multiple defect populations or flaw types [3]. However, in these bundle-based tests, this may suggest different bundle failure mechanisms, e.g., friction-induced damage or stress concentrations at the grip areas. In consequence, further investigations are needed to determine the meaning and origin of these inflections.

## 4.2 Tests with DIC

Figure 4 summarizes our DIC-based technique, showing several aspects of a typical specimen tested at  $v = 10 \text{ mm}\cdot\text{min}^{-1}$  and  $GL = 420 \text{ mm}$ . In first place, Figure 4a shows the field of view (FOV) featuring a CF bundle, whose appearance is shown before (start position) and after (end position) tensile testing. Before and at the beginning of each test, the CF bundle had a relatively smooth surface as indicated by minimal to no changes on light reflection. Conversely, during the test, wavy filaments started shining at different positions and, due to the angled illumination, fiber breakage became noticeable. With the selected CF bundles, i.e., unsized IM7, no catastrophic failure was observed on the CF bundle. The CF bundle was held in “one piece”, avoiding catastrophic failure partially due to inter-fiber friction. This led to a gradual reduction of the force recorded by the load cell. This effect can be seen in Figure 4b, which shows a gradual reduction of force after reaching the maximum load instead of a one-step sharp decrease, which is typical of brittle materials including CFs or polymer matrix composites reinforced with long CFs.

### 4.2.1 Marker effects on strain measurement

Since we relied on a compliant material to affix the markers onto the CF bundle, i.e., vacuum bag sealant, a preliminary assessment of its effects on strain measurement was carried out. The data obtained from fifteen VSGs is shown in Figure 4b as gray dots (●), whereas the average strain values are indicated by black dots (●). It is possible to notice their similarity with to those obtained from the VSG roughly placed at the center of both markers. Only after reaching the maximum force, the apparent grouping of the strain points into three sets can be noticed. These three groups correspond to the same VSG sets shown in Figure 2, also in the same order (left, middle and right). VSGs belonging to the same set, i.e.,

with the same length, should have yielded strain values with minimal variation between VSGs. It must be highlighted that the strain values range from 0.46 to 0.85, directly depending on the VSG selected for data extraction. These differences between parallel VSGs with the same nominal length may be attributed to uneven bonding between the CF bundle and the sealant tape. An additional factor may be a slight misalignment with respect to the CF bundle longitudinal axis. Overall, these variations have serious implications since the maximum number of fibers loaded during the first part of the test and the Weibull modulus ( $N_l$  and  $m$ , respectively) rely on  $S_0$  and  $S^*$  as indicated in Equations (1) and (2). Thus, the means used to affix the speckle pattern onto the specimens as well as the VSG alignment will be considered as critical parameters in future test campaigns.

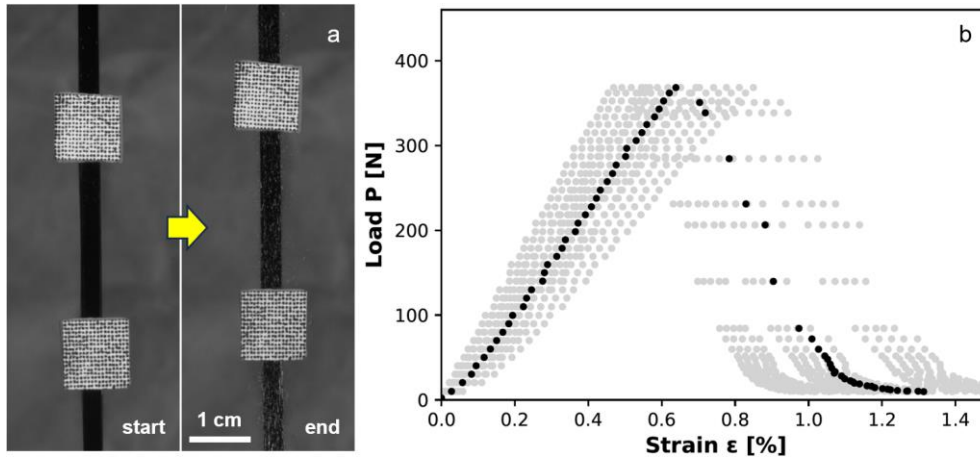


Figure 4. DIC details of a typical specimen tested at  $v = 10 \text{ mm min}^{-1}$  and  $GL = 420 \text{ mm}$ : start and end positions of a CF bundle and its targets within the FOV (a); Load-Strain graph with gray (●) and black (●) data points corresponding to individual values extracted from all VSGs and their average, respectively (b).

#### 4.2.2 Number of loaded filaments, Weibull shape parameter and average strength

Figure 5 shows the Load-Strain graphs from three tests (Runs 1 to 3) performed at  $v = 10 \text{ mm min}^{-1}$  and  $GL = 420 \text{ mm}$ . On the one hand, the individual markers correspond to data extracted from DIC, averaged from fifteen VSGs as indicated in Figure 4b. It must be noted that Run 3 corresponds to the same averaged data shown at Figure 4b. On the other hand, the lines correspond to data fitted by piecewise linear regression using least squares, for which a maximum of three segments were considered to consider gradual fiber breakage close the point of maximum load without affecting the data at the beginning of the test. Upon visual inspection, the three tests exhibit a similar behavior, suggesting good repeatability.

Figure 5 also has two Insets ('a' and 'b') that help to understand the evolution of the CF bundle at the early stage of the test. First, Inset 'a', shows a 'toe' feature at the beginning of the three tests, i.e., within  $P = [0, 50] \text{ N}$  and  $\varepsilon = [0, 0.1]$ . This non-linear portion might be explained by marker realignment or slippage of the CF bundle around both capstan drums' surfaces prior to getting self-locked. Inset 'b' shows the behavior of the CF bundle past the toe feature, i.e., within  $P = [50, 100] \text{ N}$  and  $\varepsilon = [0.1, 0.2]$ . In this range, the Load-Strain data already followed a linear trend.

It must be emphasized that the initial slope of each Run used in Equations (1) and (2), i.e.,  $S_0$ , corresponds to the value obtained from the linear fits shown in Inset 'b', within the range  $\varepsilon = [0.15, 0.20]$ . Subsequently, since  $S^*$  relies on the slope created by the origin of the Load-Strain data and the maximum load value, the "toe" feature was compensated by considering the strain value of the fit line with slope  $S_0$  at  $y = 0$ .

The maximum load as well as the maximum strain are similar in all cases, suggesting again a repeatable method. Better insight was gained upon calculation of CF bundle and individual fiber properties.

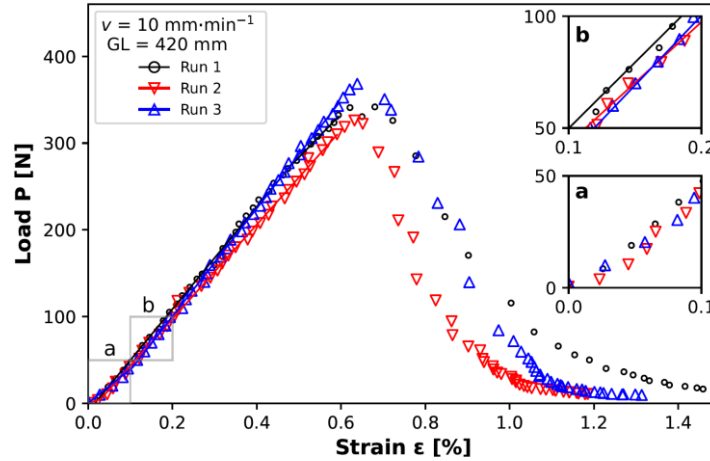


Figure 5. Load-Strain graph with individual points extracted via DIC. CF bundles were tested at 10 mm min<sup>-1</sup> and GL = 420 mm. Insets ‘a’ and ‘b’ show the toe and first linear portion of the tests, respectively. Run 3 corresponds to the same averaged data shown at Figure 4b.

Table 1 shows a summary of the CF bundle and individual filament properties obtained via the sequence introduced in §3. The maximum number of fibers loaded during the first linear portion of the test ( $N$ ) ranged from ~77 % to ~88 % of the total number of filaments contained in the CF bundle. This means that ~1400 to ~2800 filaments do not contribute to bearing the load at the initial stage of the test. This might be explained by fibers that were broken due to handling, specimen misalignment or fiber waviness also induced during the specimen preparation. The Weibull modulus ( $m$ ) of individual fibers ranged from 2.4 to 4.5, suggesting moderate variability of the results. Future test campaigns should consider more tests to determine whether the CF bundles selected for testing are representative of the whole spool. The bundle strength values ( $\sigma_b$ ) correspond to approximately one third of the hypothetical value that could be reached under even loading and at GLs one order of magnitude shorter, i.e., in the range of 25 mm. This is partially explained due to the ineffective initial loading and the sequential fiber failure as well as the flaw distribution at large GLs. Finally, the average fiber strength ( $\langle\sigma\rangle$ ) ranges from ~45 to 54 % of the reported value [12]. This is also explained by the large GL value considered in this step, in agreement with tests performed on single fibers [13]. These results are in line with the weakest link theory and covered by the Coleman factor [15] already considered in Equation (4).

Table 1. Summary of results obtained from DIC data. Tensile tests were carried out at  $v = 10 \text{ mm}\cdot\text{min}^{-1}$  and  $\text{GL} = 420 \text{ mm}$ .

$N$	$m$	$P^*$	$\varepsilon$ (at $P^*$ )	$\sigma_b$	$\langle\sigma\rangle$
[-]	[%] <sup>#</sup>	[N]	[%]	[MPa]	[MPa]
10063	83.8	341.7	0.68	1598.9	3079
9271	77.2	326.0	0.63	1655.8	2776
10621	88.5	368.3	0.64	1632.9	2606

<sup>#</sup> vs. 12K filaments

<sup>##</sup> vs. TDS, i.e.,  $\langle\sigma\rangle/\sigma_{\text{TDS}}$ ;  $\sigma_{\text{TDS}} = 5688 \text{ MPa}$  [12]

## 4 CONCLUSIONS

A bundle-based technique was developed to determine the tensile strength of CFs using alternative specimen holding fixtures and direct strain measurements. The use of capstan grips eased the specimen preparation and fixation, requiring no bundle consolidation nor bonding. Bundle strain was determined by means of DIC. This technique eliminated the need to calculate the machine compliance, opening a new avenue towards calculating the maximum number of loaded fibers, the CF bundle mean tensile

strength as well as the Weibull modulus and average tensile strength of individual fibers. Preliminary results provided good insight into the repeatability of the technique.

DIC-based data suggests that our technique works at low cross crosshead speeds. The largest portion of the initial slope ( $S_0$ ) as well as maximum load ( $P^*$ ) and strain at  $P^*$  ( $S^*$ ) are similar in all cases, suggesting again a repeatable method. However, the means of attachment of DIC markers (speckle patterns) must be improved to reduce variability on strain calculation. Higher image acquisition rates will also help to validate the technique in case that faster crosshead speeds are used in future test campaigns. Other aspects of specimen conditioning such as lubrication to avoid friction between fibers might need to be assessed.

The potential of this technique is at least twofold. First, fibers can be readily tested for screening purposes with only a handful of specimens needed. Second, fibers could be treated or exposed to harsh conditions that may prevent the preparation and use of consolidated/glued bundles, e.g., exposing CF bundles to fire [16], followed by DIC analysis. The next steps in the validation process of the technique also include considering different GLs, crosshead speeds and types of CFs, i.e., PAN-based with different mechanical properties or fibers obtained from other precursors.

### ACKNOWLEDGMENTS

The authors would like to thank the financial support provided by the Natural Sciences and Engineering Research Council of Canada (NSERC/CRSNG) grant no. RGPIN-06410-2016 (NSERC Discovery). PCG is also indebted to the National Council of Science and Technology of Mexico (CONACYT) for his doctoral scholarship, partially supporting the present work.

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