Flexural and shear strength properties of unidirectional carbon fiber reinforced polymer composite interleaved with recycled carbon fiber and short virgin aramid fiber non-woven mats

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Abstract. Carbon fiber reinforced polymer composites (CFRPs) are one of the most widely used composite types and wastes associated with them (CFRPs) get generated through either their manufacturing or end-of-service-life. Predominately due to environmental concerns and governmental regulations, recycling these CFRPs is needed and to make use of the recycled carbon fibers (rCFs), a wet paper-making technique was used to convert the rCFs into a 60 g/m² non-woven mat. For comparison purposes, the same technique was used to convert short virgin aramid fibers (vAFs) into a 60 g/m² non-woven mat. Each mat was sandwiched with two resin films and then interleaved with 12-ply unidirectional (UD) prepreg tapes (carbon/epoxy). The assemblage was molded into composite laminates using a vacuum bagging assisted compression molding technique, and the samples for the tests were cut using a waterjet machine accordingly. Compared with the control, the results indicate an increment in the flexural modulus, and the specific flexural modulus for the CFRPs with non-woven mats: the flexural modulus increased by approximately 8.2% and 12.0% for the CFRP with rCF and vAF mats, respectively; the specific flexural modulus increased around 9.5% and 13.3%, respectively for the CFRP with rCF and vAF mats. On the other hand, the shear strength approximately decreased by 6.4% and 6.0% for the CFRP with rCF and vAF mats, respectively. The negative shear strength performances of the composite laminates with non-woven mats reflected on their flexural strength performances: the flexural strength increased about 1.1% and decreased by approximately 7.9% for the CFRP with vAF and rCF mats, respectively. To resolve the negative shear strength performances, it is recommended that the surfaces of the mats be treated with a coupling agent to improve their interfacial adhesions.

Keywords: Recycled Carbon Fibers; Non-woven Mats; Aramid Fibers; Reuse

1. Introduction

Carbon fiber reinforced polymer (CFRP) composites are widely used in many industries because of their high specific stiffness and high specific strength [1-3]. These properties are quantitatively adequately documented by Khanna et al. [4]. Predominately driven by environmental/sustainability purposes [5], there is the need to develop sustainable, eco-friendly materials that could compete with other materials. For light-weight structural applications, including aerospace applications where minimum structural weight is required, a high specific modulus is essential since this translates into more significant weight savings at high stiffness [6]. Flexural modulus is a mechanical property that indicates the capacity of a material to bend. Shear strength is an indicator of the interfacial adhesion of
the layers of the composite laminates: poor interfacial adhesion could be detrimental: -possible
delamination of the composite laminates.

In a recent comprehensive review article by Pakdel et al.[7], and May et al. [8], it has been noted that
highly energy-efficient and cost-effective recycling techniques which are industrially applicable have
been developed for carbon fiber reinforced composites. In the same review articles, very positive
environmental and waste management impacts were noted. Furthermore, Pakdel et al. indicated that
despite all the positives associated with recycling these fibers, very little research has been done into
the end usage by turning these recycled fibers into non-wovens for making composite laminates. In
addition, Oliveux et al. [9]also conducted a comprehensive literature review of the various recycling
technologies (mechanical recycling; thermal processes: pyrolysis, fluidized bed pyrolysis, micro-
waves assisted pyrolysis; and solvolysis) that are in existence to aid in the recycling of fiber-reinforced
polymer composites (fibers: glass, carbon, and aramid). The review also focused on reusing the
reclaimed fibers: particularly, the way they have been reused in newer materials or applications and
the major technical difficulties at hand. It was noted in the review article that recycled fibers can
substitute little amounts of virgin fibers in products for structural applications but not at a high fiber
loading amount. This means that only a certain percentage of recycled carbon fibers (rCFs) could be
used in a material destined for structural applications—whether it is light-weight structural applications
or not. And, also, the recovered carbon fibers from high-end technology applications cannot be reused
in the same applications from which they were reclaimed since they suffer a slight decrement in fiber
properties, so alternative applications must be found to reuse these fibers. Regarding the above
observation, it can be concluded that, for example, fibers recovered from the wing-box of an aircraft
cannot be used in any parts of the wing-box again but rather, such fibers could be used in other light-
weight structural applications in the cabin of the aircraft. Applications could range from cockpit panels
to cabin trays in the aircraft.

To develop sustainable, eco-friendly materials that could compete with other materials, it is essential
to adopt the hybridization technique since there is a slight decrement in the properties of the involving
rCFs. Hybridization is a method in which two or more fibers are combined to enhance the properties
of their composites. However, it should be noted that hybridization has its shortcomings: there is either
a negative or positive effect [10]. In hybridization, the final composite is expected to exhibit the
advantages of involving fibers (positive hybridization), but that is always not the case. A scenario
could arise where the 'wrong' fibers are combined to take the disadvantages of the involving fibers
rather than the advantages of the involving fibers in the final composite product (negative
hybridization).

Since there was a slight decrement in the properties of the reclaimed fibers of the carbon-reinforced
composites, combining high strength, standard modulus fiber, T700 with high modulus fiber, M46J
should take advantage of both properties fibers.

The use of aramid non-woven mats for structural applications has been in existence for a long time,
and as seen in Figure 1 [11], such materials have been used in the construction of the Boeing 757
aircraft: it has been used in making the fixed trailing edge panels; wing-to-body fairings; and flap
support fairings (the forward segment). Boeings 757 and 767 were simultaneously developed in the
1980s. According to the Boeing company, the 757 and 767 both had the same technological advances
in materials, avionics, aerodynamics, and propulsions [12]. According to the above statement,
although the 757 was phased out in early 2004, the 767 is still being constructed; hence materials
made with aramid non-woven mats are still being used for structural applications. Although aramid
non-woven mats in structural composites are known for toughness improvements such as compression
after impact (CAI) and resistance to delamination [13-16], with regards to the fixed trailing edge
panels, for example, during their operation, one of the loads they experience is bending loads which
are characterized as flexural moduli hence the use of aramid non-woven mats for benchmarking
purposes in the study of the flexural and shear strength properties of UD CFRP composite interleaved with recycled carbon fiber non-woven mat is not far-fetched.

Figure 1. The main structure of the Boeing 757-200 aircraft showing composite parts used in its manufacturing [11]. Source: Copyright 2021, McGraw Hill LLC.

The composite laminates with the non-woven mats can be recycled despite the introduction of the vAFs. From the works of Asmatulu et al. [17], Okajima et al. [18], Yuyan et al. [19], and Chuang et al. [20], vAFs are recyclable. Although no literature has been sighted at the time of writing wherein mixed fibers (hybridized carbon and aramid fibers) have been recycled, with the current progress of recycling technologies, such materials can inevitably be recycled sooner than later. Regarding the configuration with the rCF mat only, to the best of the authors’ knowledge by way of extensive literature, the methodology applied (the interleaving of UD fiber/epoxy prepreg with rCF non-woven mat) and the product developed (UD CFRP composite interleaved with recycled carbon fibers’ non-woven mat) is the first of its kind. The UD CFRP composite interleaved with aramid fibers’ non-woven mat is solely for comparison purposes.

Investigating the flexural and shear strength properties of unidirectional carbon/epoxy laminated composite interleaved with recycled carbon fibers non-woven mat for possible light-weight structural applications is the objective of this paper. Since the rCFs are fluffy and discontinuous; the best way to utilize them for potential light-weight structural applications is to convert them into non-woven mats using a paper-making technique and use the non-woven mats to interleave continuous fibers. With that, a closed-loop sustainable, environmentally friendly material can be developed for possible light-weight structural applications.

2. Materials and methods
2.1. Materials
The virgin aramid fibers, poly (p-phenylene terephthalamide) branded as Kevlar® 29 (K29) was supplied by DuPont in chopped strands with an average length of 3.0 mm. Kevlar® 29 (K29) is a para-amid with good mechanical and damping properties and is widely used for structural applications. The fiber has a diameter of 12 μm, a specific density of 1.44, ultimate tensile strengths of 2.92 GPa to 3.62 GPa, and a tensile modulus of 70.5 GPa. The picture of vAF is shown in Figure 3(a).
The recycled carbon fibers (rCFs) are standard modulus and high modulus Toray T700SC and M46J, respectively, recovered by the fluidized bed recycling process at the University of Nottingham, UK. Although the properties of the rCFs were not measured in this project, it is expected that the rCFs will suffer a slight decrement in their tensile strengths.

The vAFs and rCFs in the form of 60 g/m² non-woven mats (mats), as seen in Figure 3(b) and Figure 4(c), respectively, were used as the interleaving materials (they were used to interleave a commercially available unidirectional carbon (UD) fiber/epoxy prepreg, Toray T700SC carbon fiber (12K) prepregs). The prepreg has a ply thickness of 0.20 mm, areal density of 200 g/m², and resin content of 35 wt.%. The resin film has an areal density of 125 g/m², a ply thickness of 0.10 mm and resin content of 100 wt.%. Both the UD carbon fiber prepreg and the resin film have the same resin system, YPH-42T, and were supplied by CA composites limited, Shanghai, PR China.

2.2. Experimental methods

2.2.1. Manufacture of recycled carbon fiber and chopped virgin aramid fibers non-woven mats.

Figure 2 shows the front view of the in-house rig used to make the non-woven mat using a paper-making technique. The wet paper-making technique was used to convert both recycled carbons and chopped virgin aramid fibers into rectangularly shaped non-woven mats (340 × 240 mm²) with an areal density of 60 g/m² using the in-house rig. The technique consists of three stages: fiber dispersion, fiber filtration, and non-woven mat drying.

Firstly, an amount of water is determined using a set of parameters, and the resulting measurement is pumped into the dispersion tank. Next, the chemical compositions found in table 1 are measured and added to the water in the dispersion tank. The chemicals are carefully added and thoroughly mixed for 8 hours (the resultant solution is used for a particular areal density as many times as possible) using a high-shear radial impeller at about 1200 ± 50 rpm. The resulting solution is pumped into the storage tank, and the desired amount is pumped from the storage tank into the dispersion for the mixing of the fibers and subsequently making the mats.

In reference to the steel mesh size on which the resulting mat will settle, the masses of rCFs and vAFs are measured in grams and added to the resultant solution in the dispersion tank separately. The rCFs measured are T700SC and M46J in the ratio of 75:25—the percentage ratio was because of the processing limitations associated with the M46J rCFs: the two were added to the solution simultaneously. Next, the fiber suspension is mixed for 30 ± 5 minutes with a high-shear radial impeller at about 1200 ± 50 rpm. The speed and time reported here were the best-compromised values that resulted in the making of good mats. The fiber-slurry is then pumped into the sheet former and the fiber-slurry sieve through a stainless-steel mesh with the aid of a vacuum pump. The resulting mat is wet and fragile at this stage; hence it is carefully removed from the stainless-steel mesh and dried at 100°C for 10 minutes.

2.2.2. Composite laminates’ manufacturing.

The composites were fabricated using the vacuum bagging-assisted compression molding technique. Firstly, the Toray T700SC UD carbon fiber (12K) epoxy prepregs and mats were cut to 328 × 228 mm² using a ply cutter. The configurations with mats had a single mat sandwiched between two resin films (F₂) before being inserted into the middle of 12 layers of prepregs. With the arrangements with mats, six prepregs (P₆) were stacked in the UD fiber direction, followed by the sandwiched mat, then with another six prepregs. The layup sequence [P₁₂], [P₆F₁MᵣC₉F₁P₀], and [P₆F₁MᵥAF₉F₁P₀] are configurations with no mat (the control laminate), 100% rCF mat and 100% vAF, respectively.
Figure 2. Front view of the non-woven mat rig.

Figure 3. (a) vAFs and (b) manufactured vAF non-woven mat.

Figure 4. (a) 75% of T700 rCFs, (b) 25% of M46J rCFs and (c) manufactured 100% rCF non-woven mat.

Table 1. Chemical compositions.

| Chemicals                  | Supplier | Functionality                        | Composition |
|----------------------------|----------|--------------------------------------|-------------|
| Hydroxyethyl Cellulose     | Macklin  | Binding and Viscosity Modifier       | 5.5 g/l     |
| Brij 35 non-ionic surfactant| Aladdin  | Wetting and Dispersing Agent         | 1.0 g/l     |
| Tributyl phosphate         | Aladdin  | Anti-foaming agent                   | 0.5 g/l     |

The stainless steel mold and frame were cleaned with Acetone before being wiped with the mold releasing agent, Chemlease® PMR EZ, to remove the cured composite laminates. The preforms were then fitted into a 330 × 230 mm² (inner dimensions) stainless steel frame mold and two PTFE release films were used to cover the top and bottom of the steel plates. The assemblage was then placed on a 600 × 500 × 4 mm³ stainless steel plate, with breather laid on top and vacuum, bagged using a sealant tape. A vacuum pump was connected, and the preforms were degassed at about room temperature till the vacuum pressure reaches 0.1 MPa. Afterward, the assemblage was relocated to the hot press and dwelled at 80°C for about 30 min. At the about 30th minute, an additional 5 MPa pressure from the hot-
press was applied, and the curing temperature was increased to 130°C in about 25 minutes and it
dwelled at 130°C for 120 minutes. The cured composites were then taken out of the hot press at the
120th minute and allowed to cool down to about room temperature with the vacuum still applied
(vacuum is applied throughout). Figure 5 shows the curing cycle. After the fabrication of the
composite laminates, the samples for the various tests were cut using a water jet cutter. The samples
were cleaned and dried in an oven at 100°C for 24 hours before testing.

\[ E_f = \frac{L^3m}{4wt^3} \]  
where: \( E_f \) = flexural modulus (MPa), \( L \) = support span (mm), \( w \) = width (mm), \( t \) = thickness (mm),
and \( m \) = slope of the tangent to the initial straight-line section of the load-deflection curve, (N/mm of
deflection); \( m \) is estimated by drawing a straight-line to the steepest initial section of the load-
deflection curve.

With regards to the evaluation of the flexural strength, ordinarily, Equation (2) is used to compute the
flexural strength but according to the standard, when higher support span-to-thickness ratios are used
(ratios greater than 16) (32 was used in this project), significant-end forces get developed at the
support noses. This will affect the moment in a simply supported beam. As such, Equation (2) includes
an approximate correction parameter for the effect of these end forces hence Equation (3) being used
to estimate the flexural strength.
\[ \sigma_f = \frac{3F}{2wt^2} \]  

(2)

where: \( \sigma_f \) = flexural strength (MPa), \( F \) = maximum load encountered before failure (N), \( L \) = support span (mm), \( w \) = width (mm), and \( t \) = thickness (mm).

\[ \sigma_f = \frac{3F}{2wt^2} \left[ 1 + 6 \left( \frac{D}{L} \right)^2 - 4 \left( \frac{t}{L} \right) \left( \frac{D}{L} \right) \right] \]  

(3)

where: \( \sigma_f, F, L, w, \) and \( t \) = the same as for Equation (2), and \( D \) = deflection of the centerline of the specimen at the middle of the support span or maximum deflection encountered before failure (mm).

2.3.2. Short beam shear strength.

In addition, to assess the composite laminates' interfacial adhesion, the short beam shear test was done according to the standard, ASTM D2344-2016 [22], wherein the interlaminar shear strength is calculated to quantify the interfacial adhesion. The specimen had the dimension, 8 mm × 6 mm with a thickness of 2.31 mm, 2.51 mm, and 2.52 mm for the control, configurations with rCF mat and vAF mats, respectively. The span length-to-thickness ratio was approximately 4. All the shear tests were done at room temperature with a crosshead rate of 1.0 mm/min using the same universal testing machine utilized in the flexural tests. The short beam shear strength was computed using Equation (4) [22]:

\[ P_{sbs} = 0.75 \times \frac{F_m}{wxt} \]  

(4)

where: \( P_{sbs} \) = short-beam shear strength (MPa); \( F_m \) = maximum load observed during the test (N); \( w \) = width (mm), and \( t \) = thickness (mm).

2.3.3. Density determinations.

The density was determined according to the ASTM D3171-15 (Test method II) standard [23] by using a set of vernier calipers and a Mettler Toledo laboratory scale (0.0001 g). All thickness variations of the tested samples were measured, and the average value was determined. A 20 mm × 30 mm sample size was suitable per the standard recommendations and available materials. The density in g/cm³ was calculated using Equation (5) [23]:

\[ \rho_c = \frac{M_t}{A \times t \times 100} \]  

(5)

where: \( M_t \) = mass of the tested sample (g); \( A \) = area of the tested sample (m²); and \( t \) = thickness (mm).

2.3.4. Surface electron microscopy (SEM).

The surface structure of the recycled M46J fibers was analyzed using a Zeiss Sigma VP scanning electron microscope with an accelerating voltage (EHT) of 5.0 kV. The working distance (WD) was 11.3 mm. Secondary electron detectors (SE2), and in-lens detectors were employed in the SEM imaging. Before the SEM imaging, a thin layer of gold was applied to the surfaces of the fibers using Leica SCD 500 gold sputter.

3. Results and discussion

3.1. Flexural modulus of the composite laminates

The flexural modulus of the control sample and the configurations with non-woven mats are summarized in table 2. From the table, the incorporation of the non-woven mats improved the flexural modulus.

The average flexural modulus in GPa for the control, CFRP with rCF non-woven mat and vAF non-woven mat is 125.1, 135.3, and 140.1, respectively.
### Table 2. List of the average flexural modulus of the composite laminates.

| Sample       | Flexural modulus/GPa | Differences (%) compared with the control |
|--------------|----------------------|-------------------------------------------|
| $[P_{12}]$   | 125.1±3.1            | 0.0                                       |
| $[P_{6}F_{1}M_{rCF}F_{1}P_{6}]$ | 135.3±5.1            | 8.2                                       |
| $[P_{6}F_{1}M_{vAF}F_{1}P_{6}]$ | 140.1±3.3            | 12.0                                      |

Some of the factors affecting the flexural modulus are the properties of the fibers and fiber contents; hence the incorporation of the mats is expected to increase the flexural modulus because of the additional fibers from the non-woven mats. In addition, the 'somewhat' sandwich structure could explain further why the CFRP with non-woven mats had better flexural modulus compared with the control. The mats’ configurations can be considered a sandwich structure with six skins (upper and bottom each) separated by a core material (the non-woven mats). A sandwich structure is made up of two skins separated by a core material inserted centrally. The core material behaves like the web in an I-beam which increases the moment of inertia without much increment in weight thereby producing an efficient structure for sustaining bending and buckling loads [24]. This phenomenon is a possible explanation of why the configurations with mats had better flexural modulus values than the control sample. Regarding the vAF mat's performance, because it is slightly thicker than the rCF mat, this makes the moment of inertia significant, thereby increasing the flexural modulus; hence the CFRP with the vAF mat having better values compared with the configuration with the rCF mat. Notwithstanding the slight decrement in fiber properties of the reclaimed carbon fibers, to further suggest possible reasons for why the CFRP with rCF mathad slightly lower values compared with the CFRP with vAFmat, more studies must be done whiles considering other factors like the effect of rCF types and their micro-hybridization with vAF.

### 3.2. Flexural strength of the composite laminates

The flexural strength of the control sample and the configurations with non-woven mats are summarized in table 3. From table 3, the incorporation of the rCF non-woven mat had negative flexural strength performance, whiles the vAF non-woven mat had a marginal positive performance. The fundamental reason for the versions observed here is the correlation between the interfacial adhesion (see 3.4) and the flexural strength performances. Good interfacial adhesion generally leads to good flexural strength performances.

The average flexural strength in MPa for the control, CFRP with rCF non-woven mat and vAF non-woven mat is 1567.6, 1444.1, and 1585.3, respectively.

### Table 3. List of the average flexural strength of the composite laminates.

| Sample       | Flexural strength/MPa | Differences (%) compared with the control |
|--------------|-----------------------|-------------------------------------------|
| $[P_{12}]$   | 1567.6±74.3           | 0.0                                       |
| $[P_{6}F_{1}M_{rCF}F_{1}P_{6}]$ | 1444.1±187.6          | -7.9                                      |
| $[P_{6}F_{1}M_{vAF}F_{1}P_{6}]$ | 1585.3±89.3           | 1.1                                       |

### 3.3. Specific flexural modulus

The specific flexural modulus of the control sample and the configurations with non-woven mats are summarized in table 4. From the table, the incorporation of the non-woven mats had a positive impact on the specific flexural modulus. Compared to the control, the specific flexural modulus increased by 9.5% and 13.3%, respectively, for the CFRP with rCF and vAF mats.
Table 4. List of the specific flexural modulus of the composite laminates.

| Sample            | Specific flexural modulus (GPa) | Differences (%) compared with the control |
|-------------------|--------------------------------|------------------------------------------|
| $[P_{12}]$        | 79.2±2.0                       | 0.0                                      |
| $[P_6F_1M_{rCF}F_1P_6]$ | 86.7±3.3                      | 9.5                                      |
| $[P_6F_1M_{vAF}F_1P_6]$ | 89.7±2.1                      | 13.3                                     |

Compared with the control sample, the increments observed by the inclusion of the non-woven mats demonstrated that in applications where minimum structural weight is required at high flexural modulus, the configurations with the mats would be competitive materials.

3.4. Short beam shear strength

The short-beam strength of the control sample and the configurations with non-woven mats are summarized in Table 5. From the table, the incorporation of the non-woven mats harmed the short-beam strength.

Chemically, aramids have inert surfaces that usually result in poor interfacial adhesion, but in this work, the vAF mats had better performance when compared with the rCF mats. Resin compatibility issues might explain this discrepancy. For example, taking the M46J rCFs, they have some resin residues on their fibers, as shown in Figure 6. These resin residues can result in resin compatibility issues that could lead to poor interfacial adhesions, as observed here.

The solution here is to either find an appropriate sizing agent and ratios to treat the surfaces of the manufactured mats or to strive to use cleaner rCFs. Improving interfacial adhesion is critical because poor interfacial adhesion could lead to poor flexural strength values and delamination issues with the final product.

Table 5. List of the average short-beam strength of the composite laminates.

| Sample            | Short-beam strength (MPa) | Differences (%) compared with the control |
|-------------------|---------------------------|------------------------------------------|
| $[P_{12}]$        | 87.1±5.3                  | 0.0                                      |
| $[P_6F_1M_{rCF}F_1P_6]$ | 81.5±3.4                  | -6.4                                     |
| $[P_6F_1M_{vAF}F_1P_6]$ | 81.9±2.1                  | -6.0                                     |

Figure 6. SEM images of the M46J rCFs.
4. Conclusion
This study evaluated the flexural and shear strength properties of a 12-ply unidirectional (UD) carbon/epoxy (T700/YPH-42T) laminated composite to incorporate recycled carbon fibers (rCFs) into carbon fiber reinforced composites for possible light-weight structural applications. The flexural and shear strength properties were evaluated according to the ASTM D790-17 and ASTM D2344-16 standards, respectively.
When compared with the control sample, it was found that the flexural modulus increased by approximately 8.2% and 12.0% for the CFRP with rCF and vAF mats, respectively; the specific flexural modulus increased approximately by 9.5% and 13.2% respectively for the CFRP with rCF and vAF mats; the flexural strength decreased by approximately 7.9% and increased by approximately 1.1% for the CFRP with rCF and vAF mats, respectively; and the shear strength approximately decreased by 6.4% and 6.0% for the CFRP with rCF and vAF mats, respectively. The correlation between the shear and flexural strengths means that good interfacial adhesion is necessary to improve the flexural strengths. Therefore, to resolve the negative shear strength performances, it is recommended that either cleaner rCFs should be used or the surfaces of their mats should be treated with a coupling agent to improve their interfacial adhesions.

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