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Short Communication

Interfacial performance of chemically recycled carbon fibres assessed by microdroplet debonding tests

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ABSTRACT

The reuse of recycled carbon fibres in structural composites requires understanding how recycling processes affect fibre/matrix interfacial properties. In this study, the interfacial behaviour of carbon fibres recovered from a recyclable epoxy/PHU composite through a mild chemical recycling process was investigated using microdroplet debonding tests. The interfacial shear strength (IFSS) of recycled fibres was compared with that of virgin fibres. Despite modifications of the fibre surface induced by recycling, including partial removal of the initial sizing and the presence of localized residual species, recycled fibres exhibited an IFSS comparable to that of virgin fibres. Post-debonding SEM observations revealed distinct local failure mechanisms, highlighting the heterogeneous nature of the recycled fibre interface. These results provide new insights into the interfacial behaviour of recycled carbon fibres and demonstrate that chemical recycling can enable the reuse of carbon fibres without compromising fibre/matrix adhesion or requiring additional sizing.

1. Introduction

Carbon fibre reinforced polymers (CFRPs) have become essential materials for lightweight structures due to their outstanding specific mechanical properties. However, the increasing use of CFRPs raises significant end-of-life challenges, as conventional disposal routes do not allow recovery of the high added value associated with carbon fibres. In this context, the development of efficient recycling strategies is essential to support the transition towards a more circular composite industry [1,2]. Several recycling approaches have been investigated, including mechanical, thermal, and chemical routes, each presenting different advantages and limitations in terms of fibre recovery, environmental/economical burden and preservation of their properties [3].

Among these approaches, chemical recycling has received increasing attention as it enables the recovery of carbon fibres under relatively mild conditions while limiting fibre degradation [4]. Seychal et al. [5] recently developed a recyclable carbon fibre-reinforced polymer based on a hybrid epoxy/polyhydroxyurethane (PHU) dynamic network. The incorporation of hydroxyurethane functionalities into conventional epoxy formulations enabled the design of a high-performance matrix exhibiting enhanced dynamic behaviour and chemical degradability. In particular, the resulting CFRP could be efficiently recycled through an oxidative acetic acid/hydrogen peroxide treatment generating in situ peracetic acid, allowing mild degradation of the matrix and recovery of carbon fibres. This approach preserved the mechanical integrity of the recovered fibres [4] while inducing limited surface oxidation, demonstrating its potential for high-value fibre recovery, and potential environmental benefits [6]. The recovered carbon fibres were subsequently reused to manufacture a new composite, demonstrating the feasibility of the recycling/reuse approach [5]. However, successful reuse of recycled carbon fibres also requires adequate adhesion with the new matrix, which remains insufficiently investigated.

The fibre/matrix interface plays indeed a critical role in load transfer mechanisms and therefore directly governs composite performance. Among the experimental methods available to characterize interfacial properties, the microdroplet debonding test is widely used to quantify the interfacial shear strength (IFSS) of single fibres by measuring the force required to debond a resin microdroplet from the fibre surface [7]. As a well-established micromechanical approach for assessing fibre/matrix adhesion, IFSS provides a relevant first-level indicator of the interfacial performance of recycled fibres. As such, it characterizes interfacial behaviour at the microscale and does not directly account for the transfer of these properties to composite-level mechanical performance. Despite the increasing development of recycling processes for

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CFRPs and the demonstrated feasibility of reusing recovered fibres, the impact of chemical recycling on the interfacial properties of recovered carbon fibres remains insufficiently understood.

The objective of this study is therefore to investigate the effect of the chemical recycling process proposed by Seychal et al. [5] on the interfacial behaviour of recovered carbon fibres. The IFSS of virgin and recycled carbon fibres is compared using microdroplet debonding tests, while post-debonding SEM observations provide further insight into the local interfacial failure mechanisms. This study thus complements the previous demonstration of fibre recovery and reuse by specifically addressing the interfacial behaviour of the recovered fibres with a new epoxy matrix.

2. Materials and methods

2.1. Carbon fibres and recycling process

The virgin carbon fibres were extracted from a commercial carbon fibre tape (CF-UD100 by Easy composites, UK; Pyrofil TR50S 15k, Mitsubishi Chemical Carbon Fiber and Composites, USA). The fibres are PAN-based high-strength carbon fibres with a nominal diameter of 7 µm, a density of 1.82 g m^{-3}, a tensile modulus of 240 GPa, a tensile strength of 4900 MPa, and an elongation at break of 2.0%. The fibres are coated with a proprietary sizing intended for epoxy–resin applications, the chemical composition of which was not disclosed by the manufacturer. Part of this tape was incorporated into a hybrid epoxy/polyhydroxyurethane matrix to form a composite that was subsequently recycled using the chemical recycling procedure described in [5], allowing the recovery of the carbon fibres. Briefly, the composite was treated in an 80:20 acetic acid/hydrogen peroxide solution at 60 °C for 4 h, generating in situ peracetic acid that cleaved the epoxy–amine linkage and enabled recovery of the carbon fibres by filtration. The recovered fibres were dried and used as the recycled fibre condition for comparison with the virgin one.

2.2. Characterization of virgin and recycled fibres

Scanning electron microscopy (SEM) observations were performed using a field emission gun (FEG) scanning electron microscope (TESCAN MIRA 3, Czech Republic) equipped with secondary electron detector. For both fibre conditions, a 3 nm gold metallic coating was previously deposited on samples and SEM images were acquired at an accelerating voltage of 5 kV.

2.3. Microbond tests

Specimen preparation and debonding tests were performed for both fibre conditions following the protocol described in [8]. Briefly, each single fibre was carefully extracted from the bundle and mounted on a frame. Microdroplets of epoxy matrix (EPOLAM 2020 resin with amine hardener 04728, Axson Technologies, France - resin/hardener mass ratio 100:34) were manually deposited on each fibre using a needle before curing for 24 h at ambient temperature followed by 16 h at 60 °C. The embedded surface within each droplet (embedded length L_e and fibre diameter d_f) was measured using a numerical microscope (VHX-5000, Keyence, Japan). The embedded lengths ranged from 64 to 147 µm for the virgin fibres and from 72 to 148 µm for the recycled fibres. The mean fibre diameter was 8.0 ± 0.4 µm for the virgin fibres and 8.1 ± 0.4 µm for the recycled fibres.

Microbond tests were achieved with an in-house setup, assuring accurate positioning of the blades, mounted on an ElectroForce 3230 machine (Bose, USA) with a 22 N force sensor. A constant displacement rate of 8 µm s^{-1} was applied to the fibre. A total of 71 droplets from 5 virgin carbon fibres and 45 droplets from 5 recycled fibres were debonded.

The IFSS was calculated, for each individual droplet, from the maximum debonding force F_{max} using the classical microdroplet model, assuming uniform stresses along the embedded fibre length (Eq. (1)):

$$\mathrm{IFSS} = \frac{F_{max}}{\pi \cdot d_f \cdot L_e} \tag{1}$$

Mean IFSS values and associated standard deviation were determined for both fibre conditions. Since both fibre conditions were tested using the same experimental procedure, with microdroplets deposited simultaneously from the same resin batch, the comparison between virgin and recycled fibres can be considered reliable. Statistical significance was assessed using Welch's t-test for comparison of mean IFSS values and the two-sample Kolmogorov–Smirnov (KS) test for comparison of the overall distributions. For both tests, ($p < 0.05$) was considered statistically significant, while ($p > 0.05$) indicated that no statistically significant difference was detected.

Post-debonding SEM observations were finally carried out on the recycled fibres under the same conditions as those described in Section 2.2 to investigate the debonding mechanisms after recycling.

3. Results and discussion

3.1. Changes in carbon fibre properties after recycling

First, the surface morphology of the virgin and recycled carbon fibres was examined using SEM. Representative micrographs are shown in Fig. 1. The virgin fibres exhibited heterogeneous surface morphologies, ranging from well-defined manufacturing striations (Fig. 1a) to locally thick sizing deposits (Fig. 1b) that partially or completely masked the underlying fibre topography. In some regions, the apparent fibre diameter was increased by up to approximately 30% due to the sizing layer. Following recycling, most of the epoxy/polyhydroxyurethane matrix was removed [5], leading to generally cleaner fibre surfaces (Fig. 1c). However, localized residues remained attached to the recycled fibres (Fig. 1d). These residues may correspond to remaining sizing, matrix-derived material, or a combination of both, and their exact chemical nature and surface coverage cannot be determined from SEM observations alone. Thus, both virgin and recycled fibres exhibit significant local surface variability, although the origins differ (sizing deposition for the virgin fibres and residual sizing and matrix-derived material after recycling). Such heterogeneity also highlights the importance of determining the local embedded geometry for each microdroplet when calculating the IFSS.

3.2. Effect of recycling on interfacial shear strength

The results from the microdroplet debonding tests are presented in Fig. 2.

The recycled carbon fibres exhibited an average IFSS of 31.6 ± 4.5 MPa, comparable to that of the virgin ones (31.4 ± 6.7 MPa). A Welch's t-test revealed no statistically significant difference between the mean IFSS values ($t = -0.229$, $p = 0.819$). Similarly, a two-sample Kolmogorov–Smirnov test did not detect a statistically significant difference between the two IFSS distributions ($D = 0.231$, $p = 0.091$). These results indicate that no statistically significant reduction in interfacial adhesion was detected after recycling. Nevertheless, the two distributions exhibit different shapes (Fig. 2b), which may reflect differences in interfacial heterogeneity following recycling, although this difference was not statistically significant according to the KS test. The measured IFSS reflects the adhesion of the recovered fibre surface to EPOLAM 2020 and may therefore be influenced by the chemical compatibility of the residual surface species with this specific matrix; these results should not be directly generalized to other matrix systems.

The preservation of IFSS after recycling is particularly noteworthy considering that the original sizing was likely partially removed during the chemical treatment. Previous studies have shown that sizing

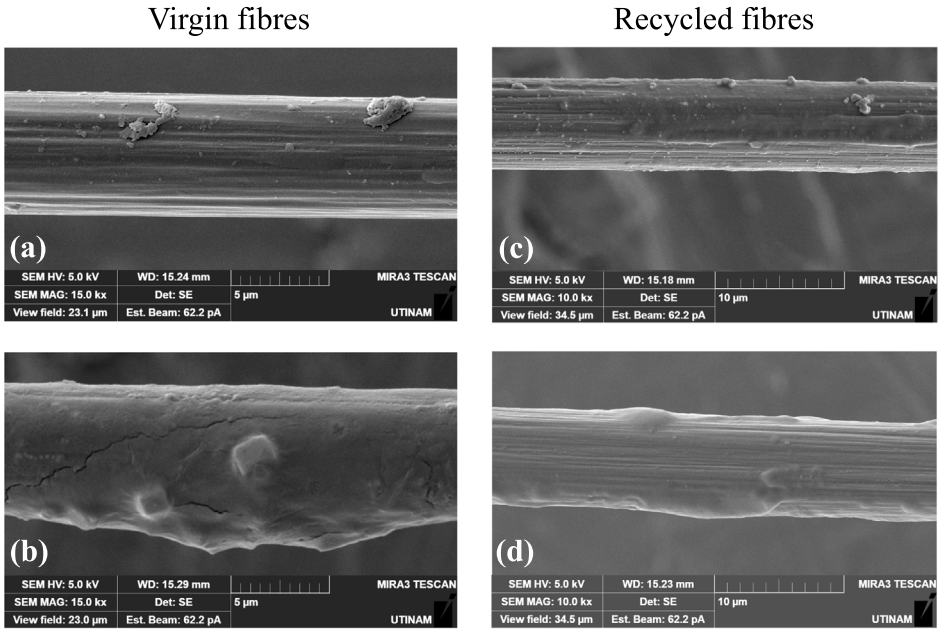


Fig. 1. SEM observations of two single virgin (a and b) and two single recycled (c and d) carbon fibres.

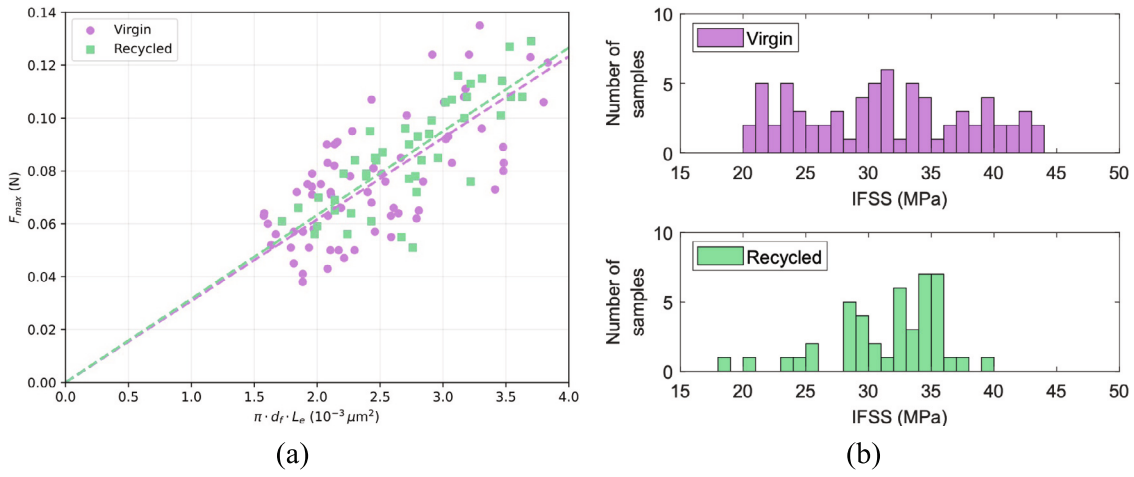


Fig. 2. (a) Debonding force as a function of the embedded fibre surface area for virgin and recycled carbon fibres. (b) IFSS distributions for both fibre conditions.

removal does not necessarily reduce interfacial adhesion, as the effect strongly depends on the sizing chemistry and the resulting fibre surface state [9]. Furthermore, several mechanisms may explain the maintained interfacial performance of the recycled fibres. Residual epoxy/PHU species on the fibre surface may promote chemical compatibility with the epoxy matrix, while the surface modifications induced during recycling [5], including increased roughness and oxidation, may enhance mechanical interlocking and interfacial bonding.

3.3. Debonding mechanisms of recycled fibres

Since the recycling process did not significantly modify the average IFSS value, post-debonding SEM observations were performed to investigate the local interfacial failure mechanisms occurring in recycled carbon fibre microdroplet specimens. Two debonded EPOLAM 2020 microdroplets located 0.8 mm apart along the same recycled carbon fibre were analysed (Fig. 3).

Two distinct failure scenarios were identified.

In the first case (Fig. 3a), an EPOLAM 2020 polymer film was visible on the left side of the residual meniscus, while significant residues attributed to either the original hybrid epoxy/PHU matrix or remaining sizing were observed on the opposite side. This observation is consistent with a crack path involving the interface between the EPOLAM 2020 microdroplet and the residual surface layer rather than the carbon fibre surface. However, based solely on the SEM observations, the exact location of the fracture plane cannot be unambiguously determined.

In the second case (Fig. 3b), the characteristic manufacturing striations of the carbon fibre were visible after debonding, with only sparse residual species highlighted in white. This observation is consistent with a failure mechanism occurring closer to the carbon fibre surface. However, due to the absence of surface characterization prior to microdroplet deposition, it remains unclear whether debonding occurred between the carbon fibre and the residual layer or directly at the fibre/EPOLAM 2020 interface.

In both cases, the loaded side of the debonded microdroplets retained a continuous meniscus curvature connecting the droplet to the

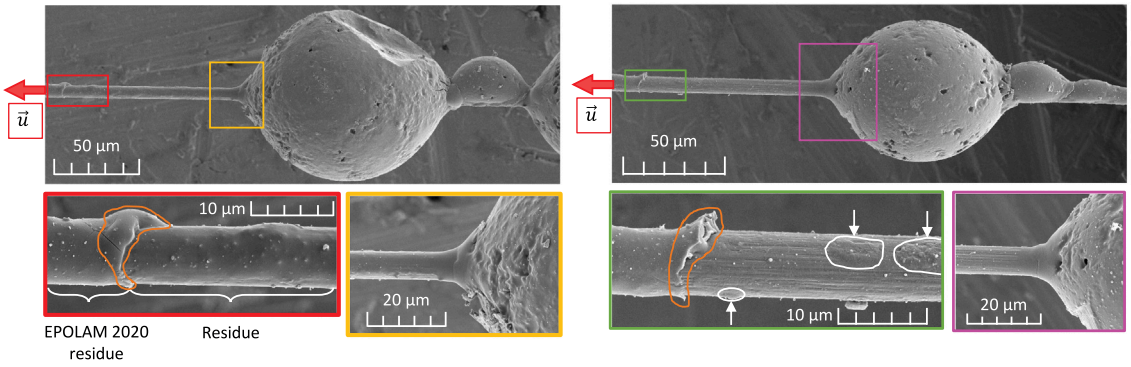


Fig. 3. SEM images of two debonded EPOLAM 2020 microdroplets located 0.8 mm apart along the same recycled carbon fibre: (a) first microdroplet (IFSS = 25 MPa) and (b) second microdroplet (IFSS = 35 MPa). The residual meniscus corresponding to the initial droplet position is highlighted by an orange dashed outline. The residue may originate from the original sizing, the PHU matrix, or a combination of both.

fibre, despite the presence of residual material at the initial droplet position. This observation suggests local variations in interfacial properties along individual fibres. The corresponding IFSS values of the two analysed droplets were 25 and 35 MPa, respectively, highlighting the influence of local surface conditions and failure mechanisms on the measured interfacial strength. Although the present measurements characterize the recycled fibres in their recovered surface state, the respective contributions of the carbon fibre surface, residual sizing, and matrix-derived residues to the measured adhesion cannot be independently distinguished. While the SEM observations provide indications of different local failure scenarios, the exact fracture paths and the respective contributions of the residual layer and fibre surface cannot be confirmed from SEM observations alone. Further investigation using controlled surface-cleaning and desizing conditions, combined with complementary surface characterization such as XPS analysis of the debonded surfaces, is therefore needed to elucidate the local interfacial mechanisms governing adhesion after chemical recycling.

4. Conclusion

This study investigated the effect of chemical recycling on the interfacial performance of recovered carbon fibres using microdroplet debonding tests. SEM observations revealed that recycling significantly modified the fibre surface condition by removing the original sizing and introducing localized residual species. Despite these surface modifications, recycled carbon fibres exhibited no statistically significant reduction in IFSS compared with virgin fibres, indicating that their interfacial adhesion with the new EPOLAM 2020 matrix is maintained after recycling. Post-debonding observations further highlighted distinct local interfacial failure mechanisms, reflecting the heterogeneous nature of recycled fibre surfaces. Combined with the previous demonstration of successful reuse of these recovered fibres in a new composite [5], the present results provide further support for the potential reuse of chemically recycled carbon fibres in composite applications. Further investigations are needed to elucidate the relationships between local surface characteristics, residual material and interfacial performance.

CRediT authorship contribution statement

Florian Boutenel: Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Adam Leveziel:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **Teddy Mebarki:** Writing – review & editing, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **Guillem Seychal:** Writing – review & editing, Writing – original draft, Validation, Resources, Methodology, Conceptualization. **Sébastien Thibaud:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Vincent Placet:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Xavier Gabrion:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of generative AI use

During the preparation of this work, the authors used ChatGPT in order to improve the clarity and readability of the manuscript. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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