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Co-curing bonding of carbon fibre/epoxy composite joints with excellent structure integrity using carbon fibre/PEEK tapes

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ABSTRACT

A novel co-curing process was proposed for the bonding of carbon fibre/epoxy composites by replacing traditional epoxy adhesives with carbon fibre/PEEK (CF/PEEK) tapes, with an attempt to improve the structure integrity. The lap-shear strengths, fatigue resistance and mode-I and mode-II fracture behaviour of the co-cured joints at 22 °C and 130 °C were investigated, and the failure mechanisms were also studied. The experimental results demonstrated that, by replacing an aerospace structural adhesive with surface-treated CF/PEEK tapes for the co-curing bonding of composite joints, the lap-shear strength of the joints had been increased by 47% and 68% at 22 °C and 130 °C, respectively; the fatigue life had been extended by 3.39 times; the mode-I fracture energy had been increased by 70% and 182% at 22 °C and 130 °C, respectively; and the mode-II fracture energy had been increased by 59% and 54% at 22 °C and 130 °C, respectively. An analysis on the failure surfaces of the tested specimens proved significant plastic deformation and breakage of the PEEK resin and extensive carbon fibre delamination being the main failure mechanisms of the CF/PEEK bonded joints. Overall, this study demonstrated a huge potential of replacing traditional film adhesives with CF/PEEK tapes for the co-curing bonding of aerospace composite joints with significantly enhanced structure integrity and thermal stability.

1. Introduction

Carbon fibre reinforced plastics are widely used in aerospace industry, owing to their low weight, excellent structural performance and high design flexibility. For example, the new generation civil aircrafts, including Airbus A350 and Boeing 787 consist of more than 50% composite materials by weight [1,2]. Consequently, it becomes essential to develop suitable joining techniques for composite materials to cope with their rapid growth in aerospace applications. Adhesive bonding is generally accepted to be an ideal process for composite joining due to the many advantages [3], such as the ability of achieving low weight assembling, the possibility of sealing the entire bonding area and hence obtaining good mechanical and fatigue properties and the potential of bonding any pair of dissimilar materials.

Depending on the state of the composite adherend, adhesive bonding can be classified as three categories, including secondary bonding [4,5], co-bonding [6,7] and co-curing [8,9] (see Fig. 1).

Among them, co-curing possesses the highest manufacturing efficiency that was achieved by curing the composite adherend and the

adhesive layer concurrently [10]. Accordingly, co-curing is usually preferred over secondary bonding and co-bonding for the joining of thermoset-to-thermoset composite joints [3]. Moretti et al. [11] investigated the process-induced strains during autoclave co-curing, co-bonding and secondary bonding of composite laminates, and reported a minimum warpage of the laminate adherend for the co-curing process. Similarly, Hasan [12] manufactured full-scale wing demos that were joined by either co-curing or secondary bonding process, and observed much less laminate warpage within the co-curing bonded joints than the secondary bonded ones. Additionally, it was demonstrated that the configuration of the co-curing bonded demos satisfied the set engineering tolerances, without defects or anomalies being inspected. Apart from the good manufacturing quality and accuracy, a number of studies have also reported adequate or excellent mechanical properties of the co-curing bonded joints [13–15]. For instance, Dhilipkumar and Rajesh [13] observed that the lap shear strengths of co-curing bonded composite joints were 67% and 52% higher than that of the co-bonding joints and secondary bonding joints, respectively. Kim et al. [14] had also reported that the co-curing hat-stiffened panels

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possessed a much higher pull-off strengths than the ones that were manufactured by co-bonding and secondary bonding techniques. Based on these studies, one can conclude that co-curing is a promising technique for composite joining, that can obtain good structure integrity with the minimum curing cycles.

Co-curing bonding generally refers to as the carbon fibre/epoxy composites are bonded by thermosetting adhesives, typically based on epoxies [7]. However, the technical characteristic of co-curing process offers a possibility of replacing the adhesive layers with alternative bonding materials, once a good bonding strength between the bonding materials and the composite adherend can be achieved. Nevertheless, this significant potential has not been explored yet. For example, advanced thermoplastic materials, including PEEK and PPS, are attractive candidates for composite co-curing bonding, as they possess exceptional mechanical properties, outstanding fracture toughness and excellent thermal stability [16,17]. Moreover, thermoplastic materials have many other advantages over epoxy adhesives if they are used for composite co-curing bonding, including an infinite shelf life, no requirement of low temperate for storage and transportation, no overflow during the co-curing process and hence a precise control on the bonding thickness. Another benefit can be foreseen is that the co-curing bonding using adhesives requires a perfect match between the curing conditions of the adhesive and the composite adherend, which is not necessary for the advanced thermoplastic materials. Considering the many significant benefits that were mentioned above, it is appealing to develop composite joints that are co-curing bonded by advanced thermoplastic materials. In this study, carbon fibre/PEEK tapes were used as alternatives of epoxy adhesives for the bonding of aerospace carbon fibre/epoxy composites using a co-curing process. The surface of the PEEK films were treated by a UV-irradiation technique, which had proved to significantly enhance their adhesion with epoxies [18]. The lap-shear strength, fatigue resistance, fracture toughness and thermal stability of the composite joints bonded by CF/PEEK tapes were investigated and compared with that of the adhesively bonded composite joints. The failure mechanisms of the co-cured joints under different loading conditions were also studied.

2. Experimental

2.1. Materials and sample preparation

The adherend was carbon fibre/epoxy prepreg, HexPly 8552-IM7-35%-134 from Hexcel. The carbon fibre/PEEK (CF/PEEK) tape was Cetex TC1200 from Toray, that consisted of unidirectional AS4 carbon fibres and 34% of PEEK matrix. The reference adhesive was FM300 from Solvay. It was an aerospace film adhesive containing thermoplastic random mat as support. The areal density of both of the carbon fibre/PEEK tape and the film adhesive was 146 g/m².

The two surfaces of a layer of CF/PEEK tape were treated by a high-power UV-irradiation technique for 10 s to improve the surface activities. The intensities of the UV spectral ranges applied to the CF/PEEK tape were measured to be 1979 mW/cm², 1546 mW/cm², 343 mW/cm² and 51 mW/cm² for the UVV (395–445 nm), UVA (320–390 nm), UVB (280–320 nm) and UVC (250–260 nm), respectively. Noteworthy, the

same treatment process had been utilised to treat the surfaces of a CF/PEEK composite in our previous study [16], and it proved to significantly increase the oxygen content of the composite surface from 14.93% to 22.97%, and decreased its water contact angle from 80.22° to 67.49°. This was because of the high-power UV-irradiation provided sufficient energy to break the C–C/C–H species, which were associated with the development of C–O, C=O and O–C=O species to the PEEK molecular chain [16,19,20].

To prepare the co-curing bonded composite joints, two layups with a [0₁₆] stacking sequence were prepared using a hand layup process. A debulking step lasting for 15 min was applied to the layups in between every fourth layer. The two [0₁₆] carbon fibre/epoxy layups were then assembled together with the UV-treated CF/PEEK tape being inserted at the joining area. The assemble was then sealed in a vacuum bag and placed in an autoclave for curing. The curing schedule was a dwell stage for 120 min with a 4 bar gauge pressure and 180 °C curing temperature in the autoclave and a 200 mbar vacuum pressure in the vacuum bag. After the curing, the composite joints were machined into desired dimensions for the following tests. Composite joints bonded by the FM300 adhesive were also prepared using the same process as references. Fig. 2 shows typical microscopy images of the cross-sections of the co-curing bonded joints, that were imaged using a laser microscope (VK-X1000 from KEYENCE Corporation).

The fibres within the adhesive layer were the thermoplastic mats for supporting the film adhesive. The red dashed lines in Fig. 2(b) indicate the boundary of the CF/PEEK layer, which was identified based on the slight difference in the colour of the PEEK matrix and the epoxy matrix. It was observed that the CF/PEEK tape was well integrated with the carbon fibre/epoxy laminates, without any visible air bubbles within the co-cured joints. The average thickness of the adhesive and the CF/PEEK within the cured joints was measured to be 110 μm and 140 μm, respectively.

2.2. Testing and characterisation

Single-lap joint specimens with dimensions that are schematically shown in Fig. 3(a) were used to evaluate the mechanical properties of the composite joints.

The single-lap joints were tested under three different conditions, including static loading conditions at 22 °C and 130 °C and a fatigue loading condition at 22 °C. The static tests were performed at a constant displacement rate of 3 mm/min within a temperature chamber on a 20 kN Zwick-Roell testing machine. The tension-tension fatigue tests were carried out on a 60 kN hydraulic fatigue machine at room temperature (about 22 °C). The maximum load, stress ratio and frequency of the fatigue tests were 6 kN, 0.1 and 10 Hz, respectively. It should be noted that the maximum load of the fatigue tests, i.e. 6 kN was approximately 50% of the static lap shear strengths (LSS) of the reference adhesive joints under static loading conditions. Three specimens were tested for the static tests, while the fatigue tests were repeated for four times.

A double cantilever beam (DCB) test [21] and an end notched flexural (ENF) test [22] were used to study the mode-I (pure opening mode) and mode-II (pure in-plane shear mode) fracture behaviour of the co-curing bonded composite joints, respectively. Illustrations of the DCB

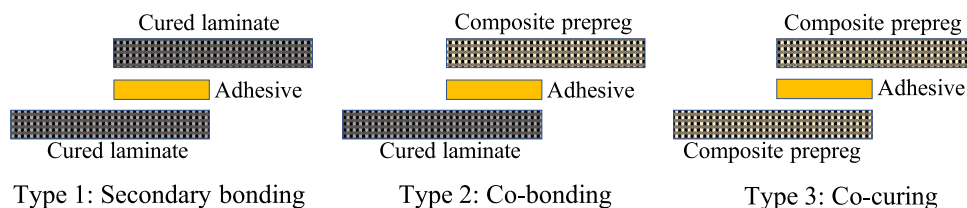


Fig. 1. Illustration of the three types of adhesive bonding processes.

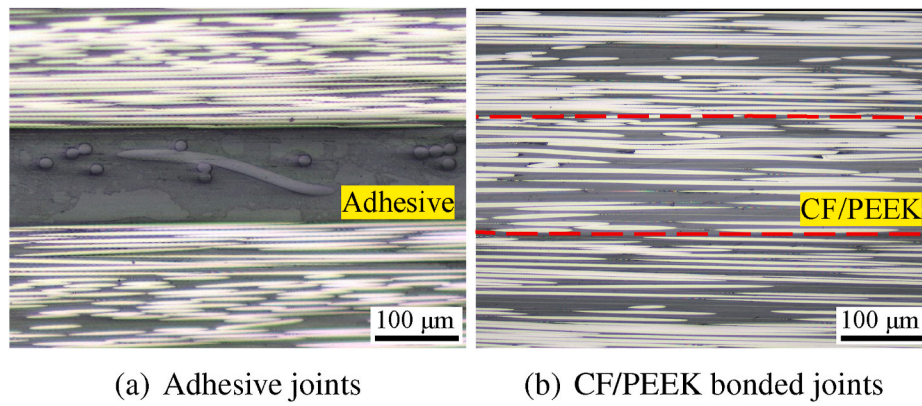


Fig. 2. Microscopy images of the cross-section of the co-curing bonded joints.

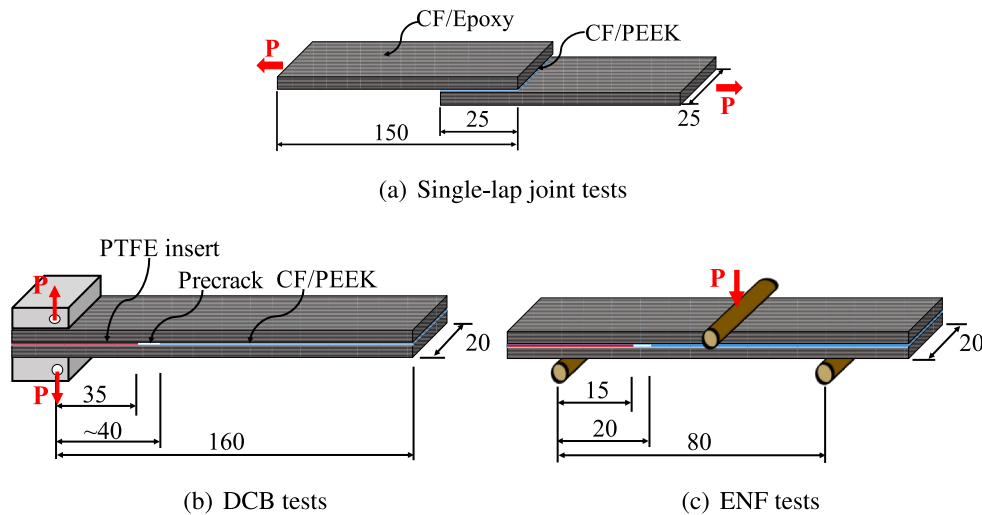


Fig. 3. Sample dimensions and schematics of the (a) single-lap shear tests, (b) DCB tests and (c) ENF tests.

and the ENF tests are shown in Fig. 3(b) and (c). The polytetrafluoroethylene (PTFE) film with a thickness of $13 \mu\text{m}$ was inserted into the joining plane during the layup process for generating a crack starter within the composite joint. The precrack with a length around 5 mm was created by loading the DCB and ENF specimens in an opening mode. During the testing, a tensile stress normal to the plane of the crack was applied to the crack tip of the DCB specimen, while a pure in-plane shear stress was applied to the crack tip of the ENF specimen. The fracture tests were carried out at 22°C and 130°C using the 20 kN Zwick-Roell testing machine. The loading speed of the DCB and the ENF tests was 2 mm/min and 0.5 mm/min, respectively. During the fracture tests, a high resolution digital camera was used to monitor the crack lengths of the specimens. Three replicate tests were performed for each set of test. The mode-I fracture energy, G_{IC} was calculated using a modified beam theory method [21]:

$$G_{IC} = \frac{3P\delta}{2b(a + |\Delta|)} \quad (1)$$

where P is the load, δ is the load point displacement, b is specimen width and a is the precrack length. A compliance calibration (CC) method was used to determine the mode-II fracture energy, G_{IIC} of the ENF specimens [22]:

$$G_{IIC} = \frac{3mP^2a^2}{2b} \quad (2)$$

where m is the CC coefficient, that was obtained by carrying the CC tests with a precrack length of 20 mm, 30 mm and 40 mm [22].

To analyse the failure mechanisms of the composite joints, the failure surfaces of the single-lap joints, DCB and ENF specimens were imaged using the laser microscope and a scanning electron microscope (JEOL JSM-7500F Field Emission Scanning Electron Microscope, SEM). The samples for the SEM analysis were sputter coated with a layer of gold with a thickness of about 5 nm.

2.3. Results and discussion

2.3.1. The single-lap joint tests

Fig. 4(a) shows the LSSs of the composite joints that were bonded by the FM300 adhesive and the CF/PEEK tape.

It was obvious that replacing the FM300 adhesives with the CF/PEEK tapes significantly increased the LSSs of the co-curing bonded joints at both of 22°C and 130°C . In specific, a LSS of 18 MPa and 15 MPa was measured for the FM300 adhesive bonded joints at 22°C and 130°C , respectively. The LSS of the CF/PEEK bonded joints was 26.4 MPa and 25.2 MPa at 22°C and 130°C , respectively. This corresponded to an increase of 47% and 68%, respectively. Moreover, the CF/PEEK bonded joints exhibited a much better thermal stability than the adhesive joints. As the temperature increased from 22°C to 130°C , the LSS of the adhesive joints obviously decreased by 17%, while only a 5% decrease was observed for the LSS of the CF/PEEK bonded joints. More encouragingly, the fatigue resistance of the CF/PEEK bonded joints was far higher than

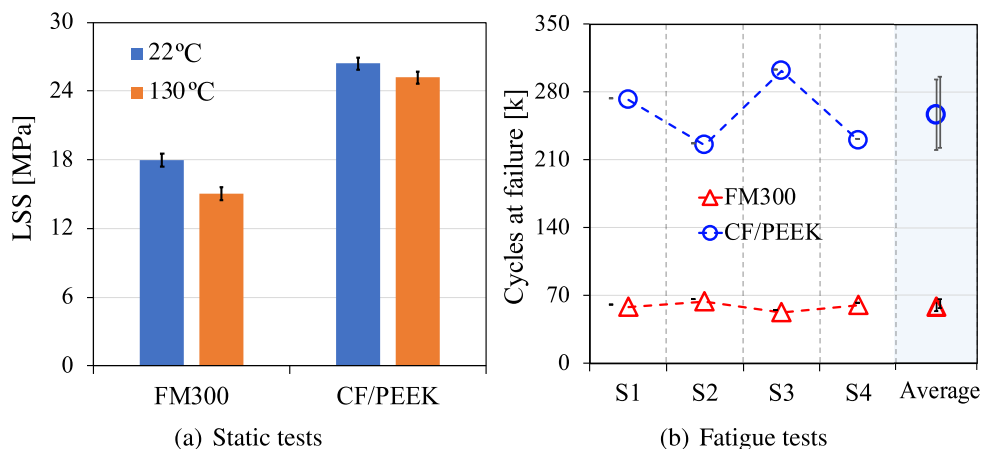


Fig. 4. The experimental results of the single-lap shear tests: (a) the LSSs of the co-curing bonded joints at 22 °C and 130 °C, and (b) the fatigue life of the co-curing bonded joints.

that of the adhesive joints, as shown in Fig. 4(b). The number of cycles at failure under fatigue was determined to be 58.4 k and 257.0 k for the adhesive bonded joints and the CF/PEEK bonded joints, respectively. This means that the fatigue life of the CF/PEEK bonded joints was 3.4 times longer than that of the adhesive joints under the same loading condition.

Fig. 5 shows representative images of the failure surfaces and cross-section of the single-lap joint specimens, that were tested under the static conditions at 22 °C and 130 °C and the fatigue condition.

From Fig. 5(a)–(d), it was observed that the failure initiated at the two joining interfaces of the overlapping area, and then merged in the middle of the overlap in all the cases. This type of failure mode

consequently caused extensive compressive failure of the carbon fibres (i.e. kinking, crushing and cracking of carbon fibres) at the failure merging area, as shown by Fig. 5(e)–(g). Typical SEM images of the failure surfaces of the single-lap joint specimens are shown in Fig. 6.

Evidence of carbon fibre debonding and breakage was observed on the failure surfaces in all the cases, see Fig. 6(a)–(c). Additionally, Fig. 6 (a) and (b) and their insert images show extensive plastic deformation and failure of the PEEK matrix around the carbon fibre prints for the static specimens, which were more extensive for the static tests at 130 °C due to the thermal softening. In summary, the extensive plastic deformation and failure of the PEEK matrix and the compressive failure of the carbon fibres contributed to the high LSSs of the CF/PEEK bonded joints

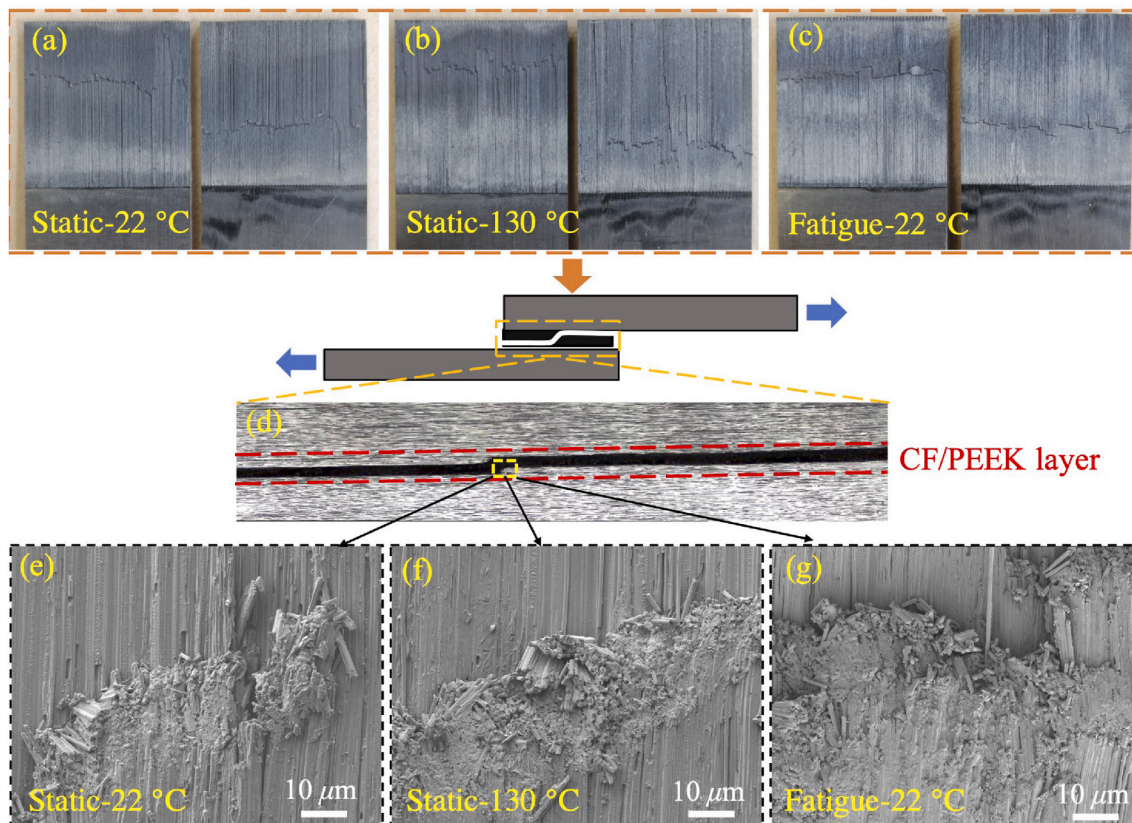


Fig. 5. Photographs and microscopy images of the failure surfaces of the single-lap joint specimens: (a)–(c) photographs of the failure surfaces, (d) a typical side-view image of the tested specimens, and (e)–(g) typical SEM images of the failure surfaces.

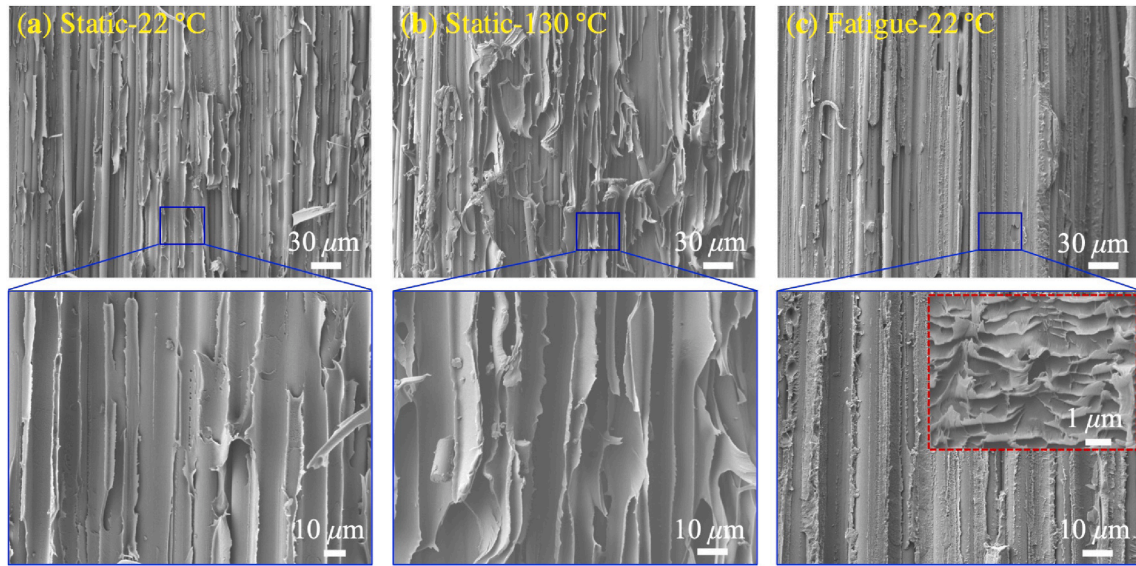
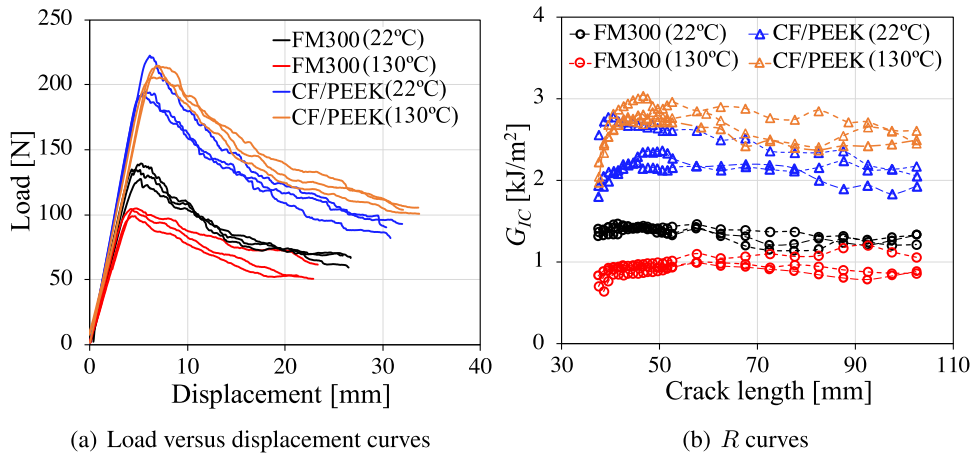


Fig. 6. Typical SEM images of the failure surfaces of the single-lap joint specimens for the CF/PEEK bonded composites, that were tested at (a) 22 °C under static, (b) 130 °C under static and (c) 22 °C under fatigue.

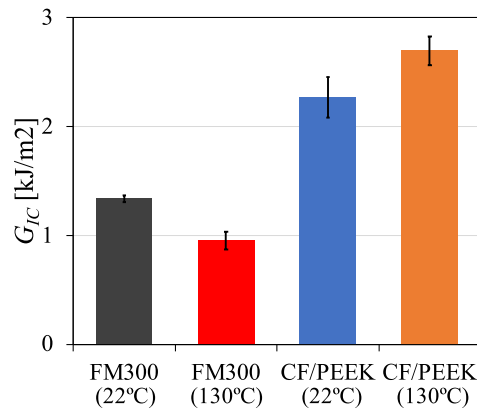
that was observed in Fig. 4(a). In contrast, no obvious plastic deformation and elongation of the PEEK matrix was observed in Fig. 6(c). However, the insert image of Fig. 6(c) show obvious down-scale plastic deformation, peeling and rolling of the PEEK matrix due to the applied

cycling loads. This means that the PEEK matrix underwent extensive damage and breakage at a relatively small scale during the fatigue test. Owing to the exceptional mechanical properties and fracture toughness of the PEEK matrix, remarkable fatigue resistance was observed for the



(a) Load versus displacement curves

(b) R curves



(c) Mode-I fracture energies

Fig. 7. The experimental results of the DCB tests: (a) the load versus displacement curves, (b) the R-curves and (c) the mode-I fracture energies of the co-curing bonded joints.

CF/PEEK bonded joints in Fig. 4(b).

2.3.2. The mode-I fracture tests

Fig. 7 shows the load versus displacement curves, R -curves and corresponding mode-I fracture energies (G_{IC}) from the DCB tests of the co-cured composite joints.

From Fig. 7(a), it was obvious that the crack propagation loads of the CF/PEEK bonded joints were much higher than that of the adhesive bonded joints at both of 22 °C and 130 °C. Consequently, the CF/PEEK joints exhibited much higher R -curves than the adhesive bonded joints, see Fig. 7(b). Additionally, as the testing temperature increased from 22 °C to 130 °C, opposite changes in the crack propagation loads were observed, i.e. the failure loads of the DCB specimens decreased for the adhesive bonded joints but increased for the CF/PEEK bonded joints, as shown in Fig. 7(a). This resulted in higher R -curves of the CF/PEEK bonded joints at 130 °C than at 22 °C, but relatively lower R -curves of the adhesive joints at 130 °C than at 22 °C, see Fig. 7(b). Fig. 7(c) summarises G_{IC} of the composite joints that were calculated from the R -curves of the DCB specimens. The value of G_{IC} for the adhesive bonded joints was measured to be 1.34 kJ/m² at 22 °C, and this value significantly decreased to 0.96 kJ/m² (by 28.4%) as the testing temperature increased to 130 °C. The replacement of the adhesives with CF/PEEK tapes increased G_{IC} to 2.27 kJ/m² at 22 °C, that was 70% higher than that of the adhesive joints. More encouragingly, a larger G_{IC} of 2.70 kJ/m² was measured for the CF/PEEK bonded joints at 130 °C, corresponding to an increase of 182% when compared with their adhesively bonded counterparts. Moreover, Shi et al. [18] reported that G_{IC} of the same carbon fibre/epoxy composite and its joints bonded by UV-irradiated PEEK films was 0.38 kJ/m² and 0.82 kJ/m², respectively. These values were much smaller than G_{IC} of the CF/PEEK bonded joints, i.e. 2.27 kJ/m² at 22 °C that were developed in this study. Overall, the composite joints bonded by CF/PEEK tapes exhibited excellent mode-I fracture resistance and thermal stability.

Fig. 8(a) and (b) show the side-view images of the CF/PEEK bonded joints during the mode-I fracture tests, and Fig. 8(c) and (d) present the photographs of the mode-I fracture surfaces.

As can be seen from Fig. 8(a) and (b), extensive carbon fibre debonding and bridging took place during the mode-I fracture process of the CF/PEEK bonded joints at both of 22 °C and 130 °C. Moreover, by taking a closer look at the side-view images, it was observed that the fibre bridging region behind the crack tip was longer at 130 °C than at 22 °C, that resulted in the larger G_{IC} at 130 °C, see Fig. 7(c). This was mainly caused by the softening of the PEEK matrix at a high temperature, which allowed more carbon fibres to debond and bridge without breaking during the mode-I fracture tests. Consequently, a larger amount of delaminated carbon fibres were observed on the fracture surfaces at 130 °C than at 22 °C, see Fig. 8(c) and (d). Fig. 9 presents typical SEM

images of the mode-I fracture surfaces of the CF/PEEK bonded composite joints that were tested at 22 °C and 130 °C.

These images further confirmed significant carbon fibre delamination, bridging and breakage mechanisms during the mode-I fracture tests in both of the cases, that is evidenced by the presence of a large amount of debonded and broken carbon fibres. Additionally, the insert images of Fig. 9 show obvious elongation, plastic deformation and failure of the PEEK matrix, with the higher temperature case exhibiting more intensive PEEK damage due to thermal softening. These observations clearly indicated that the main fracture mechanisms of the CF/PEEK bonded joints were delamination and bridging of the carbon fibres and extensive damage of the PEEK matrix. These mechanisms contributed to the high mode-I fracture energies of the CF/PEEK bonded joints in Fig. 7(c).

2.3.3. The mode-II fracture tests

The load versus displacement curves and mode-II fracture energies (G_{IIC}) of the co-curing bonded joints from the ENF tests are shown in Fig. 10.

It was observed that the crack propagation loads of the CF/PEEK bonded joints were much higher than that of the adhesive joints at both of 22 °C and 130 °C. However, no significant changes in the crack propagation loads were observed as the testing temperature increased from 22 °C to 130 °C in both of the cases. Similarly as G_{IC} , the CF/PEEK bonded joints possessed much higher values of G_{IIC} than the adhesive bonded joints, as shown by Fig. 10(b). In particular, a value of 1.78 kJ/m² and 1.64 kJ/m² was measured for G_{IIC} of the reference adhesive joints at 22 °C and 130 °C, respectively. These values significantly increased to 2.83 kJ/m² and 2.53 kJ/m², respectively by replacing the adhesives with CF/PEEK tape for the co-curing bonding of the composite joints. That corresponded to an increase of 59.0% and 54.3%, respectively. In general, one can conclude that the CF/PEEK bonded composite joints exhibited much better mode-II fracture properties than the adhesive bonded joints.

Fig. 11 presents the photographs and typical SEM images of the mode-II fracture surfaces of the CF/PEEK bonded joints.

Many delaminated carbon fibre bundles could be observed on the photographs of the mode-II fracture surfaces by naked eyes in both of the cases. Additionally, the amount of delaminated carbon fibre bundles was obviously more on the fracture surfaces at 130 °C than at 22 °C. These observations indicated significant carbon fibre debonding and bridging during the mode-II fracture process, which were more extensive at 130 °C. While the insert SEM images of Fig. 11 confirmed the debonding of the carbon fibre bundles, significant plastic deformation and failure of the PEEK matrix was also identified. Accordingly, the main mode-II fracture mechanisms of the CF/PEEK bonded joints were the same as the mode-I fracture mechanisms, i.e. significant debonding and bridging of the carbon fibres and extensive damage of the PEEK matrix.

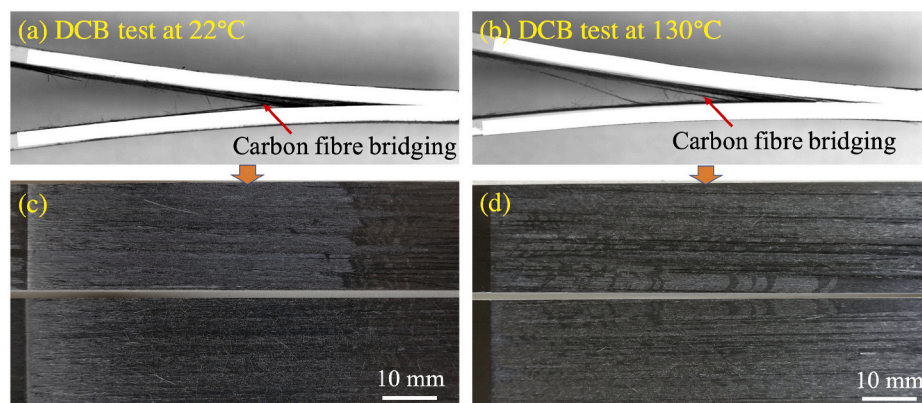


Fig. 8. (a) and (b): side-view images of the DCB specimens of the CF/PEEK bonded joints during the test, and (c) and (d): photographs of the corresponding mode-I fracture surfaces.

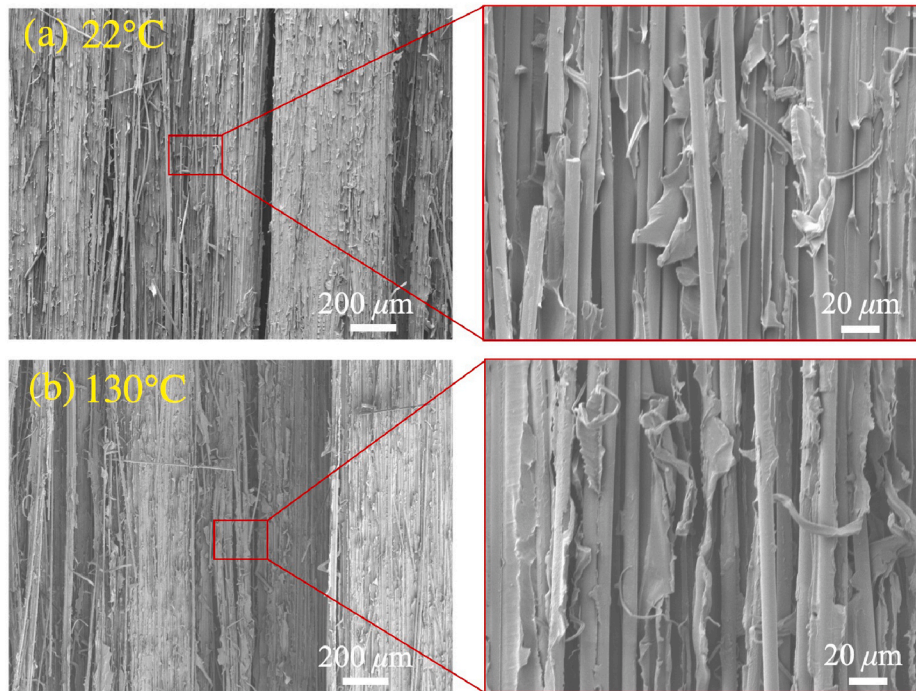


Fig. 9. Typical SEM images of the mode-I fracture surfaces of the CF/PEEK bonded joints that were tested at (a) 22 °C, and (b) 130 °C.

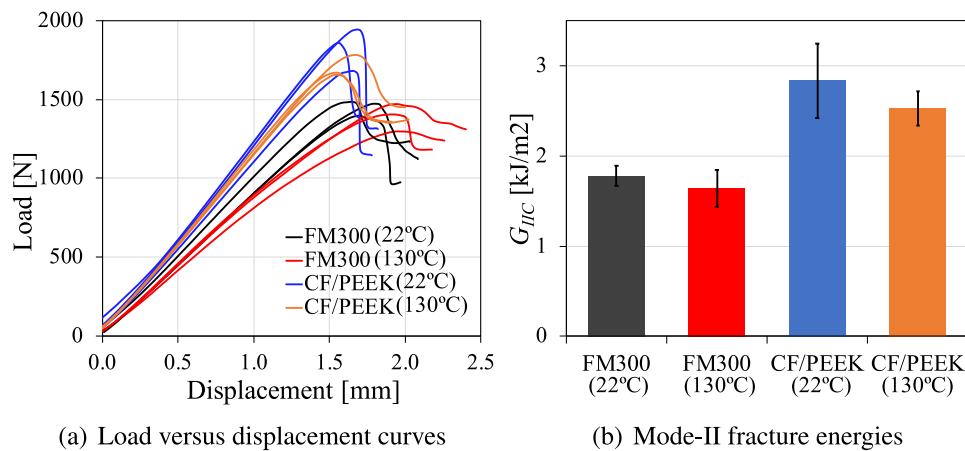


Fig. 10. The experimental results of the ENF tests: (a) the load versus displacement curves, and (b) the mode-I fracture energies of the co-curing bonded joints.

2.4. Discussion

Fig. 12 summarises the critical structure properties of the CF/PEEK bonded composite joints that were tested in this work, with the values for the adhesive joints being as the reference.

It was observed that remarkable improvements were obtained by replacing the film adhesive with the UV-treated CF/PEEK joints in all the cases. Considering the FM300 adhesive is a well commercialised and certificated aerospace structural adhesive, one can conclude that the structure integrity of the CF/PEEK bonded joints is more than sufficient for aerospace applications. Additionally, as already proposed in Section 1, there are many other advantages of the CF/PEEK tapes over the epoxy adhesives for composite co-curing bonding, including an infinite shelf life, no special requirement for the storage and transportation environment, no overflow during the curing process and hence more precise control on the bonding thickness, and no limitation to the curing schedule of the composite adherend. All these advantages, together with the excellent structural properties of the CF/PEEK bonded

joints, proved CF/PEEK tapes excellent alternatives to epoxy adhesives for the co-curing bonding of composite materials for aerospace applications.

3. Conclusions

A novel co-curing bonding process was proposed by replacing traditional epoxy film adhesives with CF/PEEK tapes. Prior to the co-curing, the surfaces of the CF/PEEK tapes were irradiated by high-power UV lights for 10 s to enhance the surface activities. That had significantly improved the adhesion between the CF/PEEK tapes and the carbon fibre/epoxy adherend and ensured excellent structural properties of the CF/PEEK bonded joints. In particular, when compared to the composite joints that were bonded by the reference aerospace FM300 adhesive, the following improvements were obtained for the CF/PEEK bonded joints: (1) the lap shear strengths had been increased by 47% and 68% at 22 °C and 130 °C, respectively; (2) The fatigue life of the single-lap joints had been extended to 4.39 times; (3) the mode-I fracture

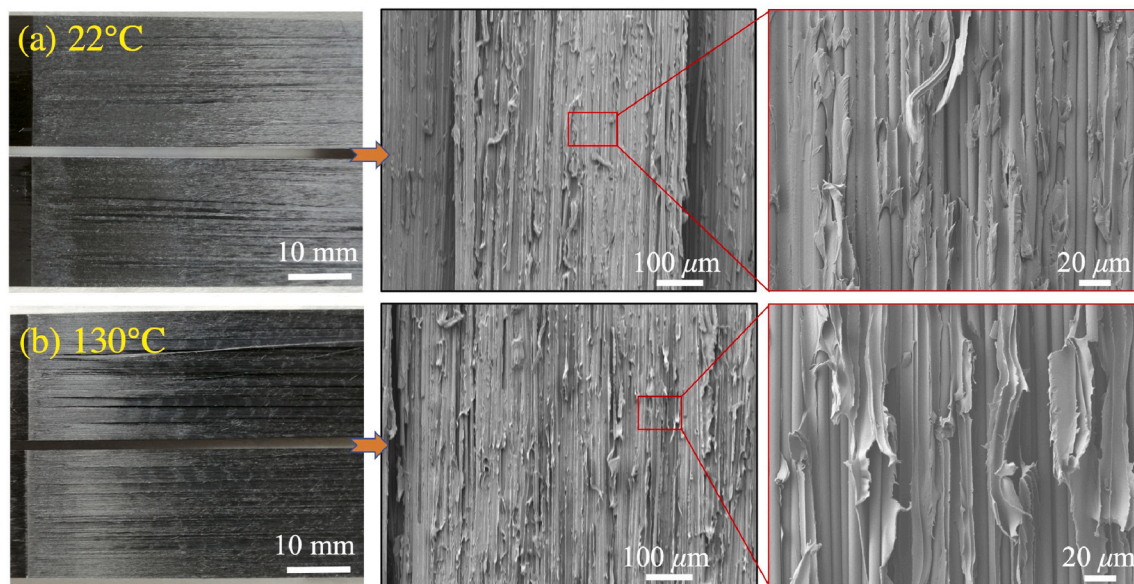


Fig. 11. Typical photographs and SEM images of the mode-II fracture surfaces of the CF/PEEK bonded joints, that were tested at (a) 22 °C and (b) 130 °C.

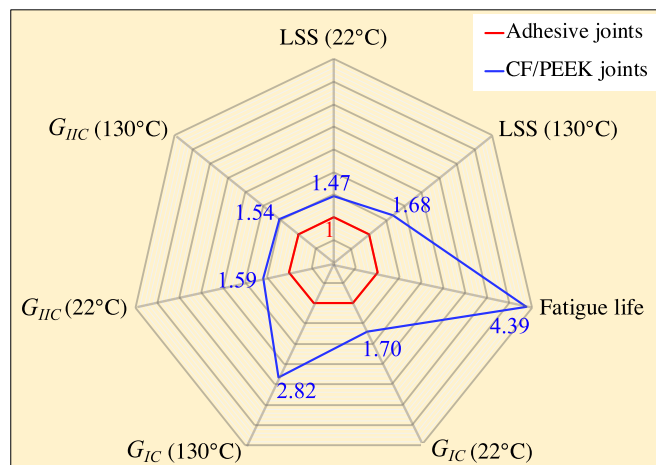


Fig. 12. The critical structure properties of the CF/PEEK bonded joints, with the values for the adhesive joints being as the reference.

energies had been increased by 70% and 182% at 22 °C and 130 °C, respectively; and (4) the mode-II fracture energies had been increased by 59% and 54% at 22 °C and 130 °C, respectively. These improvements were mainly attributed to the significant plastic deformation and failure mechanisms of the PEEK matrix, that were associated with extensive carbon fibre delamination and breakage during the failure process. In summary, this study shed lights on the development of composite joints with excellent structure integrity and thermal stability by co-curing bonding the carbon fibre/epoxy composites with CF/PEEK tapes.

Author statement

Dong Quan: Conceptualization, Investigation, Funding acquisition, Writing-Original Draft.

Guoqun Zhao: Project administration, Funding acquisition, Writing-Review & Editing.

Gennaro Scarselli: Resource, Writing-Review & Editing.

René Alderliesten: Methodology, Resource, Writing-Review & Editing.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as it forms an ongoing project.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.compscitech.2022.109567>.

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