Short carbon fibre-reinforced epoxy composite foams with isotropic cellular structure and anisotropic mechanical response from liquid foam templates

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Abstract. In this work, we show that mechanically anisotropic short carbon fibre (sCF)-reinforced epoxy foams with an isotropic cellular structure can be fabricated from liquid foam templates. Short carbon fibres were mechanically frothed in an uncured liquid epoxy resin to produce an air-in-resin liquid foam template, followed by subsequent polymerisation to produce sCF-reinforced epoxy foam with an isotropic cellular structure. Fracture toughness test showed that the incorporation of chopped carbon fibres into epoxy foams led to a significant increase in critical stress intensity factor. It was also observed that neat epoxy foams failed catastrophically whilst sCF-reinforced epoxy foams failed in a progressive manner. Compression test further showed that the in-plane compressive moduli of mechanically frothed sCF-reinforced epoxy foams were significantly higher than their out-of-plane compressive moduli, signifying an anisotropic mechanical response. This anisotropic mechanical response stemmed from the radial flow generated by the high intensity mechanical frothing process, facilitating the preferential orientation of the added chopped carbon fibres in-plane whilst the entrained air bubbles during the mechanical frothing process were in equilibrium with the surrounding uncured liquid epoxy resin, resulting in an epoxy foam with an isotropic (spherical) cellular structure.

Keywords. A. Short-fibre composites; B. Fracture toughness; C. Anisotropy; D. Fractography E. Mechanical frothing
1.0 Introduction

Macroporous polymers or polymer foams are an important class of lightweight materials used in a variety of applications. Open-cell polymer foams, characterised by a highly interconnected pore structure, are often employed as scaffolds for tissue engineering [1] and support for catalyst [2]. Polymer foams with a closed-cell structure are often used as thermal insulation [3], electromagnetic interference shielding of electronic components [4], packaging [5], as well as in applications where weight-saving is critical, such as core material in sandwich construction [6]. In the context of sandwich construction, polymer foams with mechanical anisotropy are particularly suited for directionally dependent applications [7-9]. For instance, a bicycle helmet made of mechanically anisotropic polymer foam could absorb a significant amount of rotational energy whilst allowing tangential impacts to the helmet, thereby reducing the rotational acceleration or deceleration to the head of the cyclist compared to that made with an isotropic polymer foam [10].

The mechanical properties of a polymer foam are governed by its relative density, the mechanical properties of the cell wall material and the cellular structure [11, 12]. A polymer foam with higher relative density will possess higher mechanical properties if the cell wall material is the same. Whilst the effect of cell size on the mechanical response of a polymer foam is not well-understood, a polymer foam with an open-cell structure has been shown to possess lower mechanical properties compared to its closed-cell counterparts [13]. Anisotropy in the cellular structure of a polymer foam has also been shown to influence its mechanical behaviour significantly [14]. At the same relative density, the mechanical properties of a polymer foam with higher shape anisotropy ratio, defined as the ratio of the mean intercept length in the rise direction to that in the perpendicular plane, are shown to be higher in the rise direction compared to its perpendicular plane, as well as its isotropic counterparts. In addition to cellular structure, such mechanical anisotropy could also arise
from mechanical anisotropy of the cell wall material. Natural wood, for example, is a porous material with an anisotropic cellular structure and mechanically anisotropic cell wall material [15]. The cell wall of natural wood consists of cellulose microfibrils embedded in a lignin matrix, with hemicellulose as the compatibiliser. The cellulose microfibrils, which provide mechanical support to natural wood, are preferentially orientated along the longitudinal directions in the wood trunk, leading to remarkable load bearing capability in longitudinal compared to radial directions [16].

A common method used to produce polymer foams with high shape anisotropy ratio is through the unidirectional freezing of a polymer solution or a polymer suspension, followed by freeze-drying to remove the solvent. Such method has been used to produce graphene oxide-reinforced polyimide foam [17], nanocellulose foam [18] and cellulose-montmorillonite composite foam [19] with high shape anisotropy ratio and mechanical anisotropy. Polymer foams with different shape anisotropy ratio could also be fabricated through controlling the expansion direction of a polymer foam. Oliveira-Salmazo et al. [20] produced chemically blown natural rubber foams with different shape anisotropy ratios by controlling the expansion direction of the precursor material. Natural rubber foam with the highest shape anisotropy ratio was found to possess highest compressive modulus in the rise direction. However, the compressive modulus of natural rubber foam with high shape anisotropy ratio in the perpendicular plane was found to be lower than the compressive modulus of an isotropic natural rubber foam due to its anisotropic cellular structure.

We have previously demonstrated that high performance polymer foams could be produced using a surfactant- and blowing agent-free liquid foam templating technique [21], whereby an air-in-resin liquid foam template was first produced using high energy mechanical frothing, followed by curing of the liquid foam template. The spherical air bubbles introduced during the frothing process formed an isotropic cellular structure in the
resulting foams. Polymerised acrylated epoxidized soybean oil (polyAESO) foam [22], lignin derived carbon foam [23], epoxy foam [24-26] and phenolic foam [27] have been produced using this technique. In this work, we report the fabrication of mechanically anisotropic high-performance short carbon fibre-reinforced epoxy foams with an isotropic cellular structure using this foam templating technique. The radial flow generated by the high intensity mechanical frothing process facilitated the preferential orientation of the added short carbon fibres whilst the introduced air bubbles were dispersed in equilibrium with the surrounding liquid, leading to the production of mechanically anisotropic epoxy foams with an isotropic cellular structure. The morphology, compressive properties and fracture toughness of the short carbon fibre-reinforced epoxy foams were characterised and discussed.

2.0 Experimental

2.1 Materials

Liquid epoxy resin with 56 ± 2% biomass content (Greenpoxy 56, ρ = 1.20 ± 0.01 g cm⁻³, η = 2500 mPa s at 15 °C) and an amine-based hardener with 58 ± 3% biomass content (GP 505 V2, ρ = 0.99 ± 0.01 g cm⁻³, η = 1800 mPa s at 15 °C) were purchased from Matrix Composite Materials Company Ltd (Bristol, UK) and used as received. Short carbon fibres (sCF), in the form of chopped carbon fibre tows (Carbiso™, 6 mm in length, tow size = 50K, sizing content < 1.5%), were kindly provided by ELG Carbon Fibre Ltd (West Midlands, UK).

2.2 Preparation of mechanically frothed neat and sCF-reinforced epoxy foams

2.2.1 Preparation of neat epoxy foams with different mechanical frothing time

Polymer foams of neat epoxy (polymer foam 0) were prepared following previously described protocol [25] with two different frothing times used. Briefly, 70.0 g of liquid epoxy resin and 29.4 g of amine-based hardener were added into a 2 L Pyrex glass bowl and mechanically frothed using a handheld electric mixer equipped with two stainless steel eggbeater mixing paddles (HM 730B, Sainsbury’s Supermarket Ltd, London, UK) operating
at maximum power output of 200 W for 20 min. Liquid epoxy resin and hardener mixture frothed for 2.5 min was also prepared as previously described. The resulting air-in-resin liquid foam templates were then transferred into a polytetrafluoroethylene (PTFE)-coated aluminium mould with length, width and height of 100 mm (X) × 100 mm (Y) × 20 mm (Z) and cured at room temperature for 24 h. Polymer foams of neat epoxy produced from the liquid foam templates frothed for 20 min and 2.5 min are herein termed polymer foams 0-A and 0-B, respectively.

### 2.2.2 Preparation of sCF-reinforced epoxy foams

Polymer foams of sCF-reinforced epoxy consisting of 2.5 wt.-% and 5.0 wt.-% sCF were also prepared. Liquid epoxy resin (70.0 g) and amine-based hardener (29.4 g) were added into a Pyrex glass bowl and mechanically frothed first for 20 min. Carbon fibres were then added into the air-in-resin liquid foam template and frothed for an additional period of 20 s. A short mixing time of only 20 s was employed as significant carbon fibre entanglement around the two stainless steel eggbeater-type mixing paddles was observed when longer mixing time was used.

Increasing the mixing time of the carbon fibres within the air-in-resin liquid foam templates would produce a better dispersion of sCF in the epoxy foams. To achieve this, a modified mechanical frothing process was adopted. After the initial frothing of the neat epoxy resin, 2.5 wt.-% or 5.0 wt.-% sCF were then added into the air-in-resin liquid foam templates and mixed with only a single stainless-steel eggbeater-type mixing paddle covered with duct tape. This produced a radial flow of the resin-hardener-chopped carbon fibre mixture around the mixing paddle without severe carbon fibre entanglement. A mechanical frothing time of 2 min was employed.

All the prepared air-in-resin liquid foams containing sCF were then carefully transferred into a PTFE-coated aluminium mould with length, width and height of 100 mm
(X) × 100 mm (Y) × 20 mm (Z) to avoid the disruption of sCF orientation within the liquid foam. The samples were cured at room temperature for 24 h. Polymer foams of sCF-reinforced epoxy produced by mixing the added carbon fibres for a short period of only 20 s are herein termed polymer foam 1 and sCF-reinforced epoxy foams manufactured by frothing the added carbon fibres for a longer period of 2 min are herein termed polymer foam 2.

2.3 Characterisation of mechanically frothed neat and sCF-reinforced epoxy foams

2.3.1 Density and porosity of the (sCF-reinforced) epoxy foams

He pycnometry (Accupyc II 1340, Micromeritics Ltd, Hexton, UK) was used to determine the true density of the cell wall material ($\rho_f$). Prior to the measurement, the prepared (sCF-reinforced) epoxy foams were milled into powder using a jet mill (Picojet 40 AFG, Hosokawa Alpine, Augsburg, Germany) operating at a pressure of 5 bar. The porosity of the fabricated polymer foams ($P$) was calculated using:

$$P = \left(1 - \frac{\rho_e}{\rho_f}\right) \times 100\%.$$  

where $\rho_e$ denotes the envelope density of the (sCF-reinforced) epoxy foams, evaluated from the mass and envelope volume of the samples.

2.3.2 Structure and morphology of the (sCF-reinforced) epoxy foams

The internal structure of the fabricated samples was investigated using a large chamber scanning electron microscope (SEM) (Hitachi S-3700N, Tokyo, Japan) operating at an accelerating voltage of 5 kV. Prior to SEM, the polymer foams were cut into rectangular-shaped specimens of 20 mm × 20 mm × 10 mm and broken into two halves by hand to reveal the internal structures. The samples were then mounted onto aluminium stubs using carbon tabs and coated with Au (Agar Auto Sputter Coater, Essex, UK) at 40 mA for 1 min. The average pore diameter ($D_{pore}$) was determined from the SEM images with a population size of 300 pores.

2.3.3 Fracture toughness of the (sCF-reinforced) epoxy foams
The fracture toughness of the fabricated (sCF-reinforced) epoxy foams was determined in accordance with ASTM D5045 on single edge notched beams (SENB) loaded in 3-point bending mode. Prior to the test, the skin of the samples was removed using a belt grinder and cut into rectangular test specimens with dimensions of 100 mm \((l) \times 20\, \text{mm}\) \((w) \times 10\, \text{mm} \) \((b)\). The span length used in the 3-point bending test was 80 mm. An initial crack of \(~10\, \text{mm}\) was introduced such that the initial crack length \(a\) to width \(w\) ratio fulfil the requirement of \(0.45 \leq \frac{a}{w} \leq 0.55\). To produce the initial crack, the first 8 mm were cut using a bandsaw and the remaining 2 mm were cut with a sharp scalpel. Fractographic analysis was conducted after the SENB test to determine the precise crack length for subsequent data reduction. The test was conducted using an Instron universal tester (Model 5969, Norwood, USA) equipped with 1 kN load cell. The crosshead displacement speed used was 1 mm min\(^{-1}\). A total of five specimens were tested for each type of sample. The initial critical stress intensity factor \((K_{1c})\) was calculated using:

\[
K_{1c} = \frac{P_c}{bw^{0.5}} \times f\left(\frac{a}{w}\right)
\]  

where \(P_c\) is the load at crack initiation, \(b\) is the thickness of the specimen and \(f\left(\frac{a}{w}\right)\) is:

\[
f\left(\frac{a}{w}\right) = 6 \left(\frac{a}{w}\right)^{0.5} \frac{[1.99-a/w(1-a/w)(2.15-3.93a/w+2.7(a/w)^2)]}{(1+2a/w)(1-a/w)^{1.5}}.
\]  

\[2.3.4\] **Compressive properties of (sCF-reinforced) epoxy foams**

Compression tests were performed on the fabricated (sCF-reinforced) epoxy foams using an Instron universal tester (Model 5969, Norwood, USA) in accordance with ASTM D1621. Test specimens with dimensions of 20 mm \((l) \times 20\, \text{mm}\) \((w) \times 20\, \text{mm} \) \((b)\) were compressed between two flat and parallel polished plates. The strain was monitored using a non-contact video extensometer (Imetrum Video Gauge, Bristol, UK). The load cell and crosshead displacement speed used were 50 kN and 2 mm min\(^{-1}\) (equivalent to a strain rate of 0.1 min\(^{-1}\)), respectively. A total of five specimens were tested for each type of sample.
3.0 Results and discussion

3.1 Structure and morphology of the mechanically frothed (sCF-reinforced) epoxy foams

SEM images showing the internal structure and morphology of the fabricated polymer foams are presented in Fig. 1. It can be seen from these SEM images that all the epoxy foams possess an isotropic cellular structure, i.e. spherical pores with a closed-cell structure. This indicated that the trapped air bubbles were always in equilibrium with the air-in-resin liquid foam templates (with or without short carbon fibres), as spherically-shaped air bubbles minimised surface tension [28]. Carbon fibre bundles could be observed in the family of polymer foam 1 (Fig. 1c - 2.5 wt.-% sCF-reinforced and Fig. 1d - 5.0 wt.-% sCF-reinforced) whilst individualised carbon fibres could be seen in the family of polymer foam 2 (Fig. 1e - 2.5 wt.0% sCF-reinforced and Fig. 1f - 5.0 wt.-% sCF-reinforced). This can be attributed to the differences in the mixing time of carbon fibres in the air-in-resin liquid foam templates. The short mixing time of only 20 s led to a dispersion of the added carbon fibres at a tow level in the air-in-resin liquid foam templates whilst increasing the frothing time to 2 min uniformly dispersed single carbon fibres throughout the liquid foam templates. The effect of mixing time on the dispersion of carbon fibres in both polymer foams 1 and 2 could also be observed macroscopically (see Fig. 2). Bundles of carbon fibres could be seen in the cross-section of polymer foam 1 whilst such morphology was not present in polymer foam 2. Instead, a “marbling” morphology was observed. This “marbling” morphology is characteristic of an eggbeater flow [29, 30], whereby the dispersed phase is stretched and folded by the shear forces generated during mixing. In the case of polymer foam 2, the high intensity mechanical frothing process and longer mixing time of the added carbon fibres within the air-in-resin liquid foam templates led to the shearing of the carbon
fibre tows, producing striations of carbon fibres characteristic of stretch-and-fold eggbeater flow.

The density, porosity and average pore diameter of the fabricated (sCF-reinforced) epoxy foams are summarised in Table 1. Their pore size distributions are shown in Fig. 1. Polymer foam 0-A, neat epoxy mechanically frothed for 20 min, possessed an envelope density ($\rho_e$) of 362 kg m$^{-3}$ and a porosity ($P$) of 69%, with an average pore diameter ($D_{pore}$) of 143 µm. Reducing the mechanical frothing time of the neat epoxy to only 2.5 min (polymer foam 0-B) led to a significant decrease in porosity to only 60%. This was accompanied by an increase in average pore diameter to ~200 µm and the broadening of the pore size distribution (see Fig. 1a and 1d). The shorter mechanical frothing time employed to produce polymer foam 0-B led to insufficient entrainment of air into the air-in-resin liquid foam, as well as insufficient energy input to break up the entrained air into smaller bubbles.

When the added carbon fibres were frothed for only 20 s in the air-in-resin liquid foam templates, the resulting sCF-reinforced epoxy foams (polymer foam 1) were found to possess an envelope density of 366-369 kg m$^{-3}$ and porosity of 69%, similar to that of polymer foam 0-A. However, increasing the mixing time of the carbon fibres in the air-in-resin liquid foam templates to 2 min led to a significant increase in envelope density and reduction in porosity of polymer foam 2 to 459-475 kg m$^{-3}$ and 60-61%, respectively. This tendency has been observed by many researchers [31-33] and can be attributed to the disruption of the liquid foam structure as a result of the longer mixing time employed to disperse the carbon fibres in the air-in-resin liquid foam templates, which is thought to lead to phase separation and the escape of the entrained air bubbles from the air-in-resin liquid foam.

In addition to this, the viscosity of the liquid foam is anticipated to increase as frothing of the carbon fibres in the air-in-resin liquid foam templates progresses due to the viscosifying effect of carbon fibres in a liquid suspension [34, 35]. The increase in viscosity
of the liquid foam reduced the efficiency of air entrainment during the frothing of carbon fibres, which also contributed to the decrease in porosity of polymer foam 2. This is consistent with the observation that the introduction of carbon fibres also led to a decrease in the $D_{pore}$ of polymer foams 1 and 2, where $D_{pore}$ was also found to decrease from 143 µm to ~125 µm for polymer foam 1 and ~90 µm for polymer foam 2 (see Table 1). This can be attributed to the viscosifying effect of carbon fibres in the liquid foam, which reduced foam drainage and hence, suppressed the coalescence of air bubbles [36].

3.2 Fracture toughness of the mechanically frothed (sCF-reinforced) epoxy foams

Fig. 3a-d shows the representative load-displacement curves of the fracture toughness test specimens loaded in the in-plane and out-of-plane directions. The initial linear part of the load-displacement curves corresponded to an elastic response of the polymer foams, followed by a load decrease which indicated crack propagation. Neat epoxy foams (polymer foams 0-A and 0-B) failed catastrophically, characterised by a single decrease in load to nearly zero after peak load was reached. This is a direct result of the brittleness of the cell wall material in tension, stemming from the highly crosslinked nature of the epoxy network [37]. When sCF were added, polymer foams 1 and 2 failed in progressive manner, characterised by a sequence of load decrease and increase with displacement until failure.

Fractographic analysis was conducted on the SENB test specimens to investigate the origin of the observed progressive failure. Significant pull-out of carbon fibre bundles can be observed for polymer foam 1 (Fig. 3e). Single carbon fibre de-bonding and pull-out from the matrix material can be observed for polymer foam 2 (Fig. 3f). Both of these observations implied that when peak load was reached, crack propagated (e.g. a load decrease) until the advancing crack front encountered further resistance in the form of carbon fibre bundles (polymer foam 1) or regions with individualised carbon fibres (polymer foam 2). This led to a load increase as additional energy was required to divert the crack, de-bond and/or potentially
fracture the carbon fibres. Once the necessary load applied was sufficient to overcome the resistance, the crack continued to propagate and this was accompanied by another load drop. Similar fracture morphology has been observed in randomly oriented chopped carbon fibre-reinforced polymer composites, which has been identified as a toughening mechanism for composite materials [38, 39].

The critical stress intensity factor ($K_{IC}$) of the fabricated (sCF-reinforced) epoxy foams tested in the in-plane and out-of-plane directions are summarised in Table 2. With the addition of 2.5 wt.-% and 5.0 wt.% of sCF, the $K_{IC}$ of polymer foam 1 increased by ~30% and ~54%, respectively, compared to polymer foam 0-A (similar porosity of 69%). The increase in the $K_{IC}$ of polymer foam 2 was even more pronounced; ~54% and ~113% for 2.5 wt.-% and 5.0-% sCF-reinforced epoxy foams, respectively, compared to polymer foam 0-B (at similar porosity of 61%). Since $K_{IC}$ is influenced by both pore size and relative density of a polymer foam [40], a normalised critical stress intensity factor ($\tilde{K}_{IC}$) was calculated to ascertain the influence of carbon fibres on the measured fracture toughness of the sCF-reinforced epoxy foams (see Table 2). It can be seen from this table that the $\tilde{K}_{IC}$ values of sCF-reinforced epoxy foams are still consistently higher than the neat epoxy foams (polymer foams 0-A and 0-B). The introduction of carbon fibres increased the tensile strength of the cell wall material, leading to an improvement in the fracture toughness of the sCF-reinforced epoxy foams. The higher $\tilde{K}_{IC}$ values of polymer foam 2 compared to polymer foam 1 can be attributed to the more uniform dispersion of the individualised carbon fibres.

3.3 Compressive properties of the mechanically frothed (sCF-reinforced) epoxy foams

Fig. 4 shows the representative compressive stress-strain curves of the fabricated (sCF-reinforced) epoxy foams loaded in the out-of-plane (Fig. 4a-c) and in-plane (Fig. 4b-d) directions. The mechanical response of polymer foams 0-A and 0-B was very similar when loaded in-plane or out-of-plane. This indicated that fabricated neat epoxy foams are
mechanically isotropic. Once the maximum compressive stress was reached after the initial elastic response, the stress transitioned smoothly into a long plateau (plastic collapse) before densification.

For epoxy foams reinforced with short carbon fibres, the evolution of the compressive stress-strain curves was dependent on the loading directions. When sCF-reinforced epoxy foams were compressed in the in-plane direction, a decrease in stress can be observed after maximum stress was reached. This can be attributed to the brittleness of the cell wall material when reinforced by carbon fibres [41, 42], which fractured catastrophically. This was then followed by a plastic collapse of the cell wall material. The compressive response of sCF-reinforced epoxy foams in the out-of-plane direction exhibited a strain-hardening response, i.e. an increase in strain led to an increase in stress beyond the initial linear-elastic region. This suggested an in-plane orientation of the carbon fibres within the epoxy foams, which increased the resistance towards the in-plane deformation (e.g. polymer foam stretching) of the specimens under out-of-plane loading. Since sCF-reinforced epoxy foams have isotropic cellular structure (see Fig. 1 – polymer foams contained only spherical pores, with no preferred direction of elongation), the observed anisotropy in the mechanical response arises from the anisotropy of the cell wall material, i.e. carbon fibre orientation. The high intensity mechanical frothing process generated a radial flow of the liquid foam that distributed the reinforcing carbon fibres preferentially in the in-plane direction, leading to the better mechanical performance in the in-plane direction than the out-of-plane direction [43].

The observed mechanical anisotropy in the compressive stress-strain curves is also evident in the compressive properties of the (sCF-reinforced) epoxy foams (Table 2). The compressive modulus and strength of polymer foam 0-A ($\rho_e = 362 \pm 9 \text{ kg m}^{-3}$) were found to be $220 \pm 34 \text{ MPa}$ and $6.7 \pm 0.3 \text{ MPa}$, respectively, when loaded in the in-plane direction and $235 \pm 28 \text{ MPa}$ and $6.8 \pm 0.3 \text{ MPa}$, respectively, when loaded in the out-of-plane direction.
The compressive modulus and strength of polymer foam 0-B ($\rho_e = 461 \pm 8$ kg m$^{-3}$) were found to be $319 \pm 52$ MPa and $9.7 \pm 0.2$ MPa, respectively, when loaded in the in-plane direction and $343 \pm 42$ MPa and $9.5 \pm 0.5$ MPa, respectively, when loaded in the out-of-plane direction. The in-plane and out-of-plane properties of polymer foam 0 are similar within the error of the measurements. These results suggest the mechanical anisotropy of polymer foam 0. These results are also in good agreement with the fracture toughness of the neat epoxy foams, which was also found to be independent of the loading direction. This is due to the structural and cell wall material isotropy of the neat epoxy foams.

When the epoxy foams were reinforced with carbon fibres, the in-plane compressive modulus and strength of sCF-reinforced epoxy foams increased, reaching values of up to 347 MPa and 7.4 MPa, respectively, for polymer foam 1 reinforced with 5.0 wt.-% sCF ($\rho_e = 369 \pm 14$ kg m$^{-3}$), and 845 MPa and 15 MPa, respectively, for polymer foam 2 reinforced with 5.0 wt.-% sCF ($\rho_e = 475 \pm 16$ kg m$^{-3}$). The introduction of carbon fibres, on the other hand, did not improve the out-of-plane compressive modulus of the sCF-reinforced epoxy foams. The out-of-plane compressive modulus of the sCF-reinforced epoxy foams were measured to be $\sim 220$ MPa for polymer foam 1, similar to polymer foam 0-A, and $\sim 330$ MPa for polymer foam 2, similar to polymer foam 0-B. With the introduction of carbon fibres, the out-of-plane compressive strength of sCF-reinforced epoxy foams decreased by up to 20% (from 6.8 MPa to 5.5 MPa) for polymer foam 1 and 25% (from 9.5 MPa to 7.1 MPa) for polymer foam 2. This is thought to be due to the brittle nature of carbon fibre-reinforced cell wall material.

For comparison, we have also included the density and mechanical properties of various carbon fibre-reinforced epoxy foams reported in the literature (see Table 3). The density of most carbon fibre-reinforced epoxy foams reported in the literature is higher than the carbon fibre-reinforced epoxy foams reported in this study. This is due to the fact that most epoxy foams were fabricated by dispersing hollow glass microsphere into the epoxy.
resin (e.g. syntactic epoxy foams) [44-46]. Excessive concentration of hollow glass microspheres will lead to a dramatic increase in the viscosity of the liquid epoxy resin, which leads to processing issues and in turn limited the porosity of the resulting syntactic epoxy foams [47].

As the mechanical properties of a porous material depends on its density [11], the compressive properties of our carbon fibre-reinforced epoxy foams can be compared with the work of Smorygo et al. [48] (see table 3), who fabricated carbon fibre-reinforced epoxy foams with a foam density of 405 kg m$^{-3}$ using a sacrificial template method, whereby uncured epoxy and hardener mixture was first infiltrated into a mould packed with carbamide granule template, followed by curing the epoxy and washing out the carbamide template. Their neat epoxy foam possessed compressive modulus of 190 MPa and strength of 6.7 MPa, similar to ours (polymer foam 0-A). With the addition of up to 9 wt.-% carbon fibre, the compressive modulus and strength of their composite foams improved to 245 MPa and 7.6 MPa, respectively. Comparatively, our epoxy foam reinforced with only 2.5 wt.-% carbon fibre (polymer foam 1) exhibited much higher in-plane modulus and similar strength. This higher reinforcing efficiency is largely due to the preferred in-plane distribution of our added carbon fibres.

4.0 Conclusions

Mechanically anisotropic sCF-reinforced epoxy foams with an isotropic cellular structure have been successfully produced in this work. The high intensity mechanical frothing process generated a radial flow of the liquid foam which led to the preferred distribution of the added chopped carbon fibres in the in-plane direction, leading to the production of sCF-reinforced epoxy cell wall material with mechanical anisotropy. As the entrained air bubbles remained in equilibrium with the surrounding liquid epoxy, an isotropic (spherical) pore structure of the (sCF-reinforced) epoxy foams was obtained. The incorporation of sCF into epoxy foam led to
a significant improvement in the fracture toughness of the resulting sCF-reinforced epoxy foam of up to 54% and 113% for sCF-reinforced epoxy foams frothed for 20 s and 2 min, respectively. In addition to this, it was also observed that neat epoxy foams failed in a catastrophic manner whilst sCF-reinforced epoxy foams failed in a progressive manner. Compression tests carried out on the sCF-reinforced epoxy foams in both the in-plane and out-of-plane directions showed that the in-plane compressive modulus was significantly higher than the out-of-plane compressive modulus. This is attributed to the preferred orientation of the added sCF in the in-plane direction due to the radial flow generated by the high intensity mechanical frothing process. However, the introduction of sCF led to a significant decrease in the compressive strength of the sCF-reinforced epoxy foam (by up to 20% and 25% for sCF-reinforced epoxy foams frothed for 20 s and 2 min, respectively, compared to neat unreinforced epoxy foam). This is thought to be due to the brittle nature of carbon fibre-reinforced cell wall material.

Acknowledgements

The authors would like to thank Imperial College London and China Scholarship Council for funding WS. We also greatly acknowledge funding provided by the UK Engineering and Physical Science Research Council (EP/M012247/1).

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**Figures and tables**

Figure 1: Scanning electron micrographs and pore size distribution of the fabricated (sCF-reinforced) epoxy foams. (a) Polymer foam 0-A, (b) polymer foam 0-B, (c) polymer foam 1 (2.5 wt.-% sCF-reinforced), (d) polymer foam 1 (5.0 wt.-% sCF-reinforced), (e) polymer foam 2 (2.5 wt.-% sCF-reinforced) and (f) polymer foam 2 (5.0 wt.-% sCF-reinforced). The scale bar is 200 µm, and the bin size is 20 µm.

Figure 2: Representative cross-sections of (a) polymer foam 1 (2.5 wt.-% sCF-reinforced) and (b) polymer foam 2 (2.5 wt.-% sCF-reinforced).
Figure 3: Fracture toughness of the fabricated (sCF-reinforced) epoxy foams, showing the representative load-displacement curves of SENB specimens of (a) polymer foam 1 loaded in the out-of-plane direction and (b) polymer foam 1 loaded in the in-plane direction, relative to polymer foam 0-A, (c) polymer foam 2 loaded in the out-of-plane direction and (d) polymer foam 2 loaded in the in-plane direction, relative to polymer foam 0-B, respectively, as well as exemplary fracture surfaces of SENB specimens of (e) & (f) polymer foam 1 (2.5 wt.-% sCF-reinforced) and (g) & (h) polymer foam 2 (2.5 wt.-% sCF-reinforced).
Figure 4: Representative compressive stress-strain curves of (a) polymer foam 1 loaded in the out-of-plane direction and (b) polymer foam 1 loaded in the in-plane direction, relative to polymer foam 0-A, (c) polymer foam 2 loaded in the out-of-plane direction and (d) polymer foam 2 loaded in the in-plane direction, relative to polymer foam 0-B, respectively.

Table 1: The true density of the cell wall material ($\rho_s$), envelope density ($\rho_e$), porosity ($P$) and the average pore diameter ($D_{pore}$) of the fabricated polymer foams. The errors tabulated are standard deviations.

<table>
<thead>
<tr>
<th>Polymer foams</th>
<th>$\rho_s$ (kg m$^{-3}$)</th>
<th>$\rho_e$ (kg m$^{-3}$)</th>
<th>$P$ (%)</th>
<th>$D_{pore}$ (µm)</th>
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<tr>
<td>0-A</td>
<td>1183 ± 3</td>
<td>362 ± 9</td>
<td>69 ± 1</td>
<td>143 ± 101</td>
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<tr>
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<td>1182 ± 2</td>
<td>461 ± 8</td>
<td>61 ± 1</td>
<td>197 ± 111</td>
</tr>
<tr>
<td>2 (2.5 wt.-% sCF)</td>
<td>1191 ± 3</td>
<td>459 ± 14</td>
<td>61 ± 1</td>
<td>95 ± 42</td>
</tr>
<tr>
<td>2 (5.0 wt.-% sCF)</td>
<td>1203 ± 4</td>
<td>475 ± 16</td>
<td>60 ± 1</td>
<td>83 ± 44</td>
</tr>
</tbody>
</table>
Table 2: In-plane and out-of-plane critical stress intensity factor ($K_{1c}$), normalised critical stress intensity factor ($\tilde{K}_{1c}$), compressive modulus ($E$) and compressive strength ($\sigma$) of the fabricated polymer foams. The errors tabulated are standard deviations.

<table>
<thead>
<tr>
<th>Polymer foams</th>
<th>In-plane (X-Y)</th>
<th>Out-of-plane (X-Z)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$K_{1c}$ (MPa m$^{0.5}$)</td>
<td>$\tilde{K}_{1c}$ (MPa)</td>
</tr>
<tr>
<td>0-A</td>
<td>0.38 ± 0.02</td>
<td>181 ± 6</td>
</tr>
<tr>
<td>1 (2.5 wt.-% sCF)</td>
<td>0.46 ± 0.03</td>
<td>241 ± 14</td>
</tr>
<tr>
<td>1 (5.0 wt.-% sCF)</td>
<td>0.59 ± 0.06</td>
<td>302 ± 33</td>
</tr>
<tr>
<td>0-B</td>
<td>0.54 ± 0.04</td>
<td>161 ± 14</td>
</tr>
<tr>
<td>2 (2.5 wt.-% sCF)</td>
<td>0.83 ± 0.06</td>
<td>344 ± 29</td>
</tr>
<tr>
<td>2 (5.0 wt.-% sCF)</td>
<td>1.13 ± 0.10</td>
<td>502 ± 38</td>
</tr>
</tbody>
</table>

$\dagger$ Normalised critical stress intensity factor, $\tilde{K}_{1c} = \frac{K_{1c}}{\sqrt{\rho_p \rho_e}} \left( \frac{\rho_e}{\rho_p} \right)^{-1.5}$

$\dagger$ The compressive strength is defined as the stress at the yield point (the first point on the stress-strain diagram at which an increase in strain occurs without an increase in stress) if a yield point occurs before 10 % strain or, in the absence of such a yield point, the stress at 10 % strain deformation.
Table 3: Carbon fibre reinforced epoxy foams in literature. $c$ is the loading fraction of carbon fibre in weight fraction unless specified. $\rho$, $E$ and $\sigma$ are the density, modulus and strength of the epoxy foams.

<table>
<thead>
<tr>
<th>Ref.</th>
<th>Foam</th>
<th>Fibre length</th>
<th>c (%)</th>
<th>$\rho$ (kg m$^{-3}$)</th>
<th>Properties</th>
<th>$E$ (MPa)</th>
<th>$\sigma$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>[44]</td>
<td>Syntactic epoxy foam</td>
<td>0.1 mm</td>
<td>0</td>
<td>641 ± 8</td>
<td>Tension</td>
<td>3350 ± 80</td>
<td>21 ± 2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.5†</td>
<td>640†</td>
<td></td>
<td>3500†</td>
<td>15†</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.0†</td>
<td>650†</td>
<td></td>
<td>3600†</td>
<td>14†</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.6†</td>
<td>670†</td>
<td></td>
<td>3700†</td>
<td>17†</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2.2†</td>
<td>680†</td>
<td></td>
<td>3900†</td>
<td>23†</td>
</tr>
<tr>
<td></td>
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<td>3.7†</td>
<td>720†</td>
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<td>4500†</td>
<td>32†</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>6.9†</td>
<td>797 ± 7</td>
<td></td>
<td>5420 ± 450</td>
<td>32 ± 4</td>
</tr>
<tr>
<td>[45]</td>
<td>Syntactic epoxy foam</td>
<td>0.4 mm</td>
<td>0</td>
<td>914</td>
<td>Flexural</td>
<td>1200‡</td>
<td>33</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>916</td>
<td></td>
<td>1350‡</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2</td>
<td>924</td>
<td></td>
<td>1410‡</td>
<td>36</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3</td>
<td>939</td>
<td></td>
<td>1420‡</td>
<td>39</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>5</td>
<td>954</td>
<td></td>
<td>1600‡</td>
<td>41</td>
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<td></td>
<td></td>
<td>8</td>
<td>959</td>
<td></td>
<td>1500‡</td>
<td>37</td>
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<td>[46]</td>
<td>Syntactic epoxy foam</td>
<td>3 mm</td>
<td>0</td>
<td>843</td>
<td>Compression</td>
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<td>30 ± 1</td>
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<td></td>
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<td>0.5</td>
<td>865</td>
<td></td>
<td>--</td>
<td>31 ± 1</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>1.0</td>
<td>884</td>
<td></td>
<td>--</td>
<td>29 ± 1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.5</td>
<td>901</td>
<td></td>
<td>--</td>
<td>90 ± 2</td>
</tr>
<tr>
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<td></td>
<td></td>
<td>2.0</td>
<td>914</td>
<td></td>
<td>--</td>
<td>29 ± 1</td>
</tr>
<tr>
<td>[47]</td>
<td>Sacrificially-templated epoxy foam</td>
<td>0.25 mm</td>
<td>0</td>
<td>405‡</td>
<td>Compression</td>
<td>190‡</td>
<td>6.7‡</td>
</tr>
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<td>0.5</td>
<td>404‡</td>
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<td>211‡</td>
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<td>225‡</td>
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<td>2</td>
<td>405‡</td>
<td></td>
<td>234‡</td>
<td>7.3‡</td>
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<td>3</td>
<td>404‡</td>
<td></td>
<td>241‡</td>
<td>7.0‡</td>
</tr>
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<td>5</td>
<td>403‡</td>
<td></td>
<td>235‡</td>
<td>7.5‡</td>
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<td>245‡</td>
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</tbody>
</table>

† Values are volume fraction.
‡ Values estimated from figures.