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Editorial

Each volume gathers contributions on specific topics:

- Vol 1. Industrial applications**
- Vol 2. Material science**
- Vol 3. Material and Structural Behavior – Simulation & Testing**
- Vol 4. Experimental techniques**
- Vol 5. Manufacturing**
- Vol 6. Multifunctional and smart composites**
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This collection contains the proceedings of the 21st European Conference on Composite Materials (ECCM21), held in Nantes, France, July 2-5, 2024. ECCM21 is the 21st in a series of conferences organized every two years by the members of the European Society of Composite Materials (ESCM). As some of the papers in this collection show, this conference reaches far beyond the borders of Europe.

The ECCM21 conference was organized by the Nantes Université and the Ecole Centrale de Nantes, with the support of the Research Institute in Civil and Mechanical Engineering (GeM).

Nantes, the birthplace of the novelist Jules Verne, is at the heart of this edition, as are the imagination and vision that accompany the development of composite materials. They are embodied in the work of numerous participants from the academic world, but also of the many industrialists who are making a major contribution to the development of composite materials. Industry is well represented, reflecting the strong presence of composites in many application areas.

With a total of 1,064 oral and poster presentations and over 1,300 participants, the 4-day event enabled fruitful exchanges on all aspects of composites. The topics that traditionally attracted the most contributions were fracture and damage, multiscale modeling, durability, aging, process modeling and simulation and additive manufacturing.

However, the issues of energy and environmental transition, and more generally the sustainability of composite solutions, logically appear in this issue as important contextual elements guiding the work being carried out. This includes bio-sourced composites, material recycling and reuse of parts, the environmental impact of solutions, etc.

We appreciated the high level of research presented at the conference and the quality of the submissions, some of which are included in this collection. We hope that all those interested in the progress of European composites research in 2024 will find in this publication sources of inspiration and answers to their questions.

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FIBRE REINFORCED COMPOSITES WITH BICONTINUOUS SILICA-EPOXY MATRICES

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Keywords: Silica aerogel, nanocomposite, bicontinuous, nanoreinforcement

Abstract

Bicontinuous silica-epoxy nanocomposites are promising matrices for fibre composites due to the long-range connectivity of their reinforcing silica phase. This silica aerogel-derived reinforcement has been shown to give improvements in matrix compressive and shear moduli, key factors governing the overall compression performance of fibre composite materials. The synthesis of such hierarchical fibre-reinforced materials proceeds *via* an intermediate silica aerogel-fibre composite. Whilst silica aerogel and fibre reinforcement have previously been combined for thermal insulation applications little is reported concerning the fabrication of long-range mesoporous silica networks in the presence of continuous ordered high-performance fibre reinforcements. Here, a capillary-rise type method is proposed for applying such continuous silica aerogel to woven glass and carbon fibre reinforcements. Fibre sizing is varied to investigate the influence of interfacial chemistry on the wetting of fibres by aerogel precursor sol and the resulting aerogel morphology. Polyurethane-sized carbon fibre is found to be wetted well by the sol precursor leading to a thin, continuous mostly crack-free coating of silica aerogel which bridges adjacent fibres. This observation was accompanied by a reasonable fibre content of 49 vol.%. In contrast, epoxy-sized and de-sized carbon fibre leads to poor wetting and large, cracked excess of silica aerogel.

1. Introduction

Silica aerogel has been introduced previously as a 3D continuous reinforcement for epoxy resin [1-3] Fully consolidated specimens with silica reinforcement loadings of up to 22 silica vol.% were formed. Reductions in glass transition temperature were modest with no observed change in cure conditions. The continuous silica reinforcement improves the hardness and reduced modulus of the baseline resin by 23% and 17% respectively. These properties increase with aerogel content *via* a power law relationship which demonstrates the direct role of the connected silica phase as a reinforcing network. Such

bicontinuous nanocomposites are formed by backfilling the silica aerogel porous volume with epoxy resin to give interlocking networks of silica and epoxy resin. The long-range connectivity of the silica network facilitates effective load transfer, whilst the monolithic nature of the reinforcing structure gives excellent sample homogeneity. This load transfer may be beneficial when utilised as a matrix for fibre reinforcement in a hierarchical composite. Under compressive loads composite failure initiates through micro-buckling originating in the imperfect alignment of fibres [4]. Improved lateral support of fibres by a matrix with improved shear performance may therefore delay failure leading to greater composite compressive strength. Pertinently, measured bicontinuous nanocomposite (NC17) properties show improvements of 16% and 20% respectively for shear modulus (G) and compressive modulus (E) relative to epoxy resin baseline (ER) at 17 vol.% silica content (Table 1). Hierarchical composites (bicontinuous silica-epoxy matrix, continuous fibre reinforcement) were previously formed with woven glass fibre reinforcement yielding 27% improvements in both composite compressive strength and interlaminar shear strength [5]. However, the direct contribution of the silica reinforcement in restricting the onset of kink-banding was not investigated in detail. The present work is a step towards the development of carbon fibre reinforced silica-epoxy bicontinuous nanocomposites (Fig. 1 (a)).

Table 1. Summarised properties for epoxy resin baseline (ER) and bicontinuous nanocomposite (NC17). ρ = envelope density, E = compression elastic modulus, G = shear elastic modulus.

Sample	ρ [g.cm ⁻³]	E [GPa]	G [GPa]
ER	1.14 ± 0.00	3.53 ± 0.07	1.16 ± 0.02
NC17	1.18 ± 0.01	4.20 ± 0.14	1.34 ± 0.03

Fibre-reinforced bicontinuous matrix composite specimens are generated *via* fibre-aerogel composite intermediates which are then backfilled with epoxy resin to yield the bicontinuous structure. Silica aerogel-fibre composites are currently employed as thermal insulation materials. Such materials use non-woven fibre blankets as reinforcement and apply aerogel either as powder or by continuous spraying of a liquid precursor which is then processed to aerogel *in-situ* [6,7]. These methods do not generate long-range silica networks and hence are not suitable precursors for the fully connected bicontinuous materials. Herein a liquid sol precursor is applied to continuous, woven high-performance carbon fibre tape *via* capillary rise wetting. This method should ensure that only the minimum quantity of aerogel required to coat and connect adjacent fibres is applied, maximising fibre volume fraction whilst forming a continuous, mesoporous silica matrix. To achieve an even distribution of silica and appropriate fibre volume fraction (> 40%) this sol must have good fibre wetting properties. To investigate the effect of fibre surface chemistry on sol-fibre wetting (and subsequent aerogel micro- and nanoscale morphology) a liquid sol was applied to single-ply tape sections of three different reinforcements: epoxy-sized carbon fibre (HTA40), polyurethane sized carbon fibre (HTS40) and E-Glass fibre with an unknown pluricompatible size. Continuous silica aerogel is introduced by capillary rise of the aerogel precursor liquid sol to yield (after processing and drying) a 3D aerogel structure formed around the fibre reinforcement. Aerogel is dried via critical point drying (CPD) as this method was previously used when investigating bulk compressive and shear properties of silica-epoxy bicontinuous networks [7]. The relationship between matrix and composite properties is of interest hence it is desirable to use the same matrix.

2. Experimental Details

2.1. Materials

2.1.1 Chemical Reagents

For the synthesis of silica aerogel tetramethylorthosilicate (98%, TMOS) and chlorotrimethylsilane ($\geq 98\%$, TMCS) were purchased from Sigma Aldrich (GB), and acetone (technical grade), ethanol (absolute), water (HPLC grade), and *n*-hexane (dehydrated) were purchased from VWR (UK). All chemicals were used as received.

2.1.2 Woven Fibre Tapes

25 mm wide E-Glass fibre tape (TVU 400) and 25 mm wide polyurethane-sized carbon fibre tape (TCU 330) were purchased from Cristex Ltd. (UK). 25 mm wide epoxy-sized carbon fibre tape (named here as CF 250) was purchased from R&G Faserverbundwerkstoffe GmbH (DE). TCU 330 contains HTS40 carbon fibre and CF 250 contains HTS40 carbon fibre. Both HTS40 and HTA40 are Tenax® carbon fibres produced by Toho Tenax Europe GmbH (DE). Details of all given in Table 2.

Table 2. Product details for fibre tapes used in this work as provided by suppliers.

Tape	Warp Fibre (wt.%)	Weft Fibre (wt.%)	Filament Number	Areal weight [$\text{g}\cdot\text{m}^{-2}$]	Sizing	Warp Size Level [wt.%]
CF 250	HTA40 (90 wt.%)	E-Glass (10 wt.%)	6K	250	Epoxy	1.3
TCU 330	HTS40 (91 wt.%)	E-Glass (9 wt.%)	12K	335	Polyurethane	1
TVU 400	E-Glass (90 wt.%)	E-Glass (10 wt.%)	N/A	400	Pluricompatible	Unknown

2.1.3 Fibre Clamping and Alignment Rigs

Fibre tape clamp components were manufactured from 3 mm thick poly(tetrafluoroethylene) (PTFE) sheet (Direct Plastics Ltd (UK)) and 2 mm thick PTFE sheet (RS Components) on an XYZ CNC router (CA). Components were assembled with polyether ether ketone (PEEK) fastenings. M2 x 10 mm Phillips pan head machine screws were purchased from Accu Ltd. (UK) and M2 PEEK hexagon nuts were purchase from High Performance Polymer (UK). The tape alignment rig was machined from 12 mm thick aluminium plate (6082 cast aluminium tooling plate) using a Bridgeport router with ACU-RITE Millpwr G2 CNC controller.

2.1.4 Capillary Rise Infusion Apparatus

Glass petri dishes (54.5 mm x 20 mm) were purchased from VWR (UK). 300 mL polypropylene storage boxes (90 mm x 65 mm x 55 mm) were purchased from Viking (UK). 50 μm Nylon film (Capran 518) was purchased from Cytec Engineering Materials Ltd. (UK). LSM5127 High Temperature Sealant Tape was purchased from Composite Integration Ltd. (UK).

2.2. Removal of Fibre Sizing

The components (base, clamping arms and fastenings) of each fibre tape clamp assembly were weighed. Partially assembled fibre tape clamps were inserted into the alignment rig and a single ply of tape was inserted into the rig on top of the clamp bases to which the clamping arms were then attached (Fig. 1 (c)). The tape was then cut between the clamps using a scalpel blade to form three individual clamped fibre tape sections each of approximately 60 mm length. The individual clamped tape sections (specimens) were then removed from the alignment rig (Fig. 1 (b)) and weighed to determine the fibre mass. This procedure was repeated for each fibre tape type. For each tape type one specimen (3 specimens total) was then placed in a separate 300 mL glass jar along with 200 mL of acetone. Each specimen was removed, rinsed with acetone, and placed in a fresh acetone bath after 6 h, 24 h, 30 h, 36 h and 48 h. Specimens were then dried in a vacuum oven at 1 mbar and 50 °C for 30 min.

2.4. Preparation of Silica Aerogel Precursor Sol

Silica aerogel liquid sol was produced by combining water, TMOS and ethanol in a molar ratio of 24:1:1.8. This mixture was stirred at 1200 rpm for 3 h at 25 °C to give a single transparent sol phase.

2.5. Application of Continuous Silica Aerogel to Fibrous Reinforcement

Clamped fibre tape specimens were assembled and weighed as described in 2.2. Each specimen was then placed in a glass petri dish (diameter = 54.5 mm, height = 20 mm) at an inclination of 30 °. These petri dishes were then inserted into 300 mL storage boxes. 2 x 10 mL glass vials were installed in the storage box, held in place with sealant tape, and filled with ethanol. The box was then sealed closed with sealant tape and Nylon film to form an ethanol-saturated atmosphere within the box (Fig. 1 (e)). A 6 mL syringe was then used to pierce the film and add 4 mL of sol to the bottom of the petri dish while taking care to not apply any sol directly to the fibre tape. The hole created in the film by the syringe needle was then sealed with sealant tape to retain the ethanol-saturated atmosphere. The boxes were then left sealed for 4 days at 25 °C to age the wet silica gel. After 4 days the box was opened by cutting the Nylon film with a scalpel and the clamped specimen was removed from the petri dish and transferred to a 300 mL glass jar containing 100 mL of ethanol using a pair of tongs. Care was taken to only contact the PTFE frame and not the fibre tape section. After 24 h the specimen was transferred to a fresh ethanol bath. Hydrophobisation of the silica surface was performed by immersion of the clamped specimen in a TMCS:*n*-hexane:ethanol mixture (1:1:8 ratio by volume) for 4 days at 25 °C. Samples were then exchanged back to ethanol by replacing the volume of liquid with ethanol four times before a final exchange to acetone by replacing the volume with acetone 6 times over 3 days. Finally, samples were dried one at a time from supercritical CO₂ using a Leica EM CPD300 critical point dryer. Specimens were dried with a total of 30 CO₂ exchange cycles. The final dried clamped tape sections were then weighed to determine the mass of silica aerogel. The fibre volume fraction of each specimen was calculated from the measured fibre and aerogel masses, the monolithic aerogel envelope density [2], and the mean fibre density in each tape (1.84 g.cm⁻³ for TCU 330 and CF 250 and 2.56 g.cm⁻³ for TVU 400).

2.4. Characterisation Techniques

2.4.1 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was performed on as received and de-sized fibres using a STARE TGA DSC system (Mettler Toledo (US)). Analysis specimens were prepared by cutting short sections (2 mm – 3 mm) from the warp tows of each tape. These specimens (3 mg – 5 mg) were each placed in a pre-weighed aluminosilicate crucible. Mass was recorded during heating from room temperature to 100 °C at a rate of 10 °C.min⁻¹, isothermal heating at 100 °C for 20 min, and subsequent heating to 800 °C at a rate of 10 °C.min⁻¹. Data recorded for an empty crucible was subtracted from this data to give the mass change of the specimen. Data was processed using Origin 2022b ((64-bit) SR1 9.9.5.171, OriginLab Corporation, USA).

2.4.1 Scanning Electron Microscopy

Scanning electron microscopy was performed using a LEO Gemini 1525 FEG SEM with an accelerating voltage of 5 kV with In-lens detector. Fibre tape specimens (60 mm x 25 mm) were mounted on PTFE sheet (2 mm x 25 mm x 60 mm) and attached to an aluminium stub with conductive carbon tape. A 10 nm chromium coating was then applied (by sputtering) to create a conductive surface.

3. Results and Discussion

3.1. Removal of Fibre Sizing

Sizing was removed by soaking clamped tape sections (Fig. 1 (b)) (60 mm x 25 mm) in acetone at 25 °C for 48 h. Specimens were rinsed (with acetone) and transferred to fresh acetone five times during this 48 h period to remove sizing residues. The effectiveness of this de-sizing process was verified by TGA (Fig. 1 (d)). As received HTA40 exhibited a single mass loss event between 300 °C and 450 °C as previously reported for epoxy-based sizing agents [8]. The mass loss in this range (1.1%) is close to the manufacturer's reported value of 1.3%. HTS40 underwent mass loss in two stages, the first between 200 °C and 350 °C and the second between 350 °C and 430 °C. The total organic mass loss of 0.9% is comparable to the manufacturer's value of 1.0%. E-Glass fibres experienced mass loss in the range of 250 °C to 450 °C. The total mass loss was 0.3%. This value is significantly lower than that for the carbon fibre specimens due to their significantly lower specific surface area resulting from larger density (2.56 g.cm⁻³ vs. 1.77 g.cm⁻³) and diameter (12 µm vs. 7 µm). The acetone-treated fibres show no mass loss during heating indicating that all organic sizing was removed. Consequently, these specimens are deemed to have been successfully de-sized.

3.2. Application of Continuous Silica Aerogel to Fibrous Reinforcement

To maximise the uniformity of the composite silica distribution, the aerogel precursor sol was applied to the fibre tape sections *via* a capillary rise infusion method. This method decreases the likelihood of excess sol being applied as the balance between gravity and surface tension limits the quantity of sol at any given position along the length of the tape. In contrast, a dropwise application would require precise measurement of small quantities of sol to achieve the desired minimum uniform coating. Each clamped tape section (Fig. 1 (b)) was positioned in a sol bath at an inclination angle of 30° so that one end of the tape was immersed to a depth of approximately 1 mm when 4 mL of sol was added to the petri dish (Fig. 1 (e)). This relatively shallow angle was selected to ensure that the entire length of the tape section was coated prior to gelation by decreasing the height gain required. Future experimental determination of the most appropriate angle may be required to moderate the quantity of applied silica. Capillary rise infusion was performed in an ethanol-saturated atmosphere within a sealed box to prevent premature drying of the gelled silica network. Aerogel processing was then performed as described previously for monolithic specimens [2,3].

The relative composition of the resulting silica aerogel – fibre composites were determined gravimetrically (Table 3). As received polyurethane sized carbon fibre (HTS40) and as received E-Glass both show acceptable fibre content of > 40 vol.%. The remaining fibre types have significantly lower fibre content owing to relatively poor wetting of the sol during infusion resulting accumulation of excess silica. This excess is most prominent at the end of the tape which was inserted in the sol bath. SEM images of the composite surface illustrate the effect of poor wetting on the silica aerogel connectivity (Fig. 2). In regions with a large excess silica of cracking is observed parallel to the principle fibre direction (Fig. 2 (b,c,f)). This cracking is likely due to the non-uniform stress state which exists during aerogel drying in the presence of the fibre reinforcement. Where the aerogel coating is thin and uniform, for example in the as-received HTS40 (Fig. 2 (e)) and E-Glass (Fig. 2 (h)) specimens the fibres are better able to support the aerogel network during this shrinkage and cracking is therefore far less prevalent. In these specimens the silica network has successfully bridged adjacent fibres and hence this stiff inorganic network should act to directly transfer load between these fibres. Notably, the composite formed with

de-sized E-Glass fibres also exhibited good continuity despite containing a large relative excess of silica aerogel. This outcome may be due to the additional stability provided by covalent bonding at the silica-silica interface in this composite.

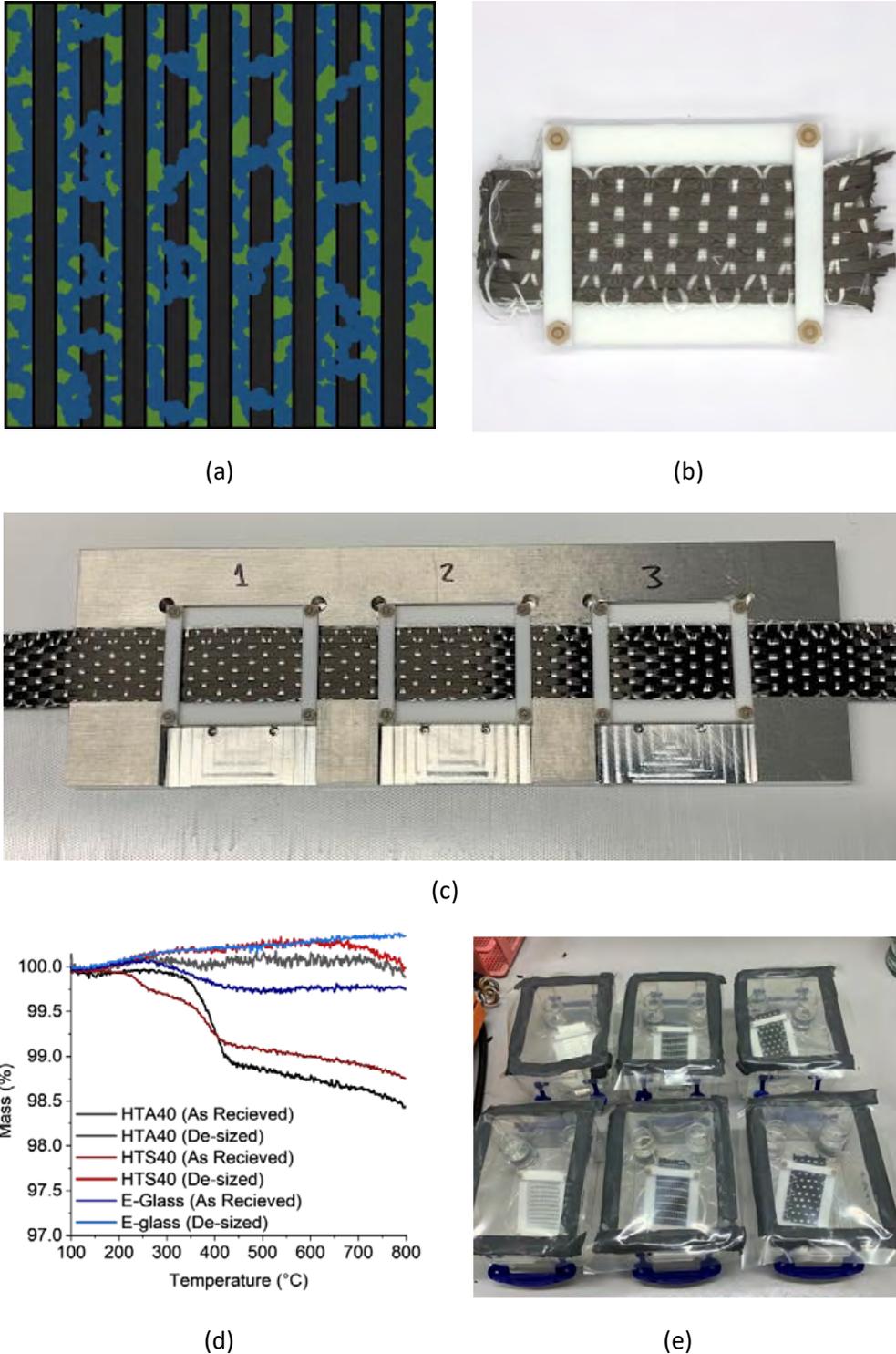


Figure 1. (a) Schematic representation of silica (blue) – epoxy (green) bicontinuous nanocomposite reinforced with continuous unidirectional carbon fibre (black, not to scale). (b) Carbon fibre – silica aerogel composite specimen held in PTFE clamping frame. (c) Assembly for alignment and clamping of carbon fibre tape layups. (d) TGA mass loss plot for carbon and glass fibre tapes with and without

removal of sizing. (e) Apparatus for coating woven tape specimens with continuous silica aerogel under a saturated ethanol atmosphere (6 shown).

Table 3. Fibre reinforcement content of silica aerogel – fibre composites formed with various fibre reinforcement materials. Error given as measurement uncertainty.

Fibre	HTA40 As Received	HTA40 De-sized	HTS40 As Received	HTS40 De-sized	E-Glass As Received	E-Glass De-sized
Fibre wt.%	67.9 ± 0.2	81.6 ± 0.3	88.7 ± 0.5	75.9 ± 0.2	87.8 ± 0.4	72.8 ± 0.2
Fibre vol.%	20 ± 2	35 ± 4	49 ± 6	27 ± 2	46 ± 5	24 ± 2

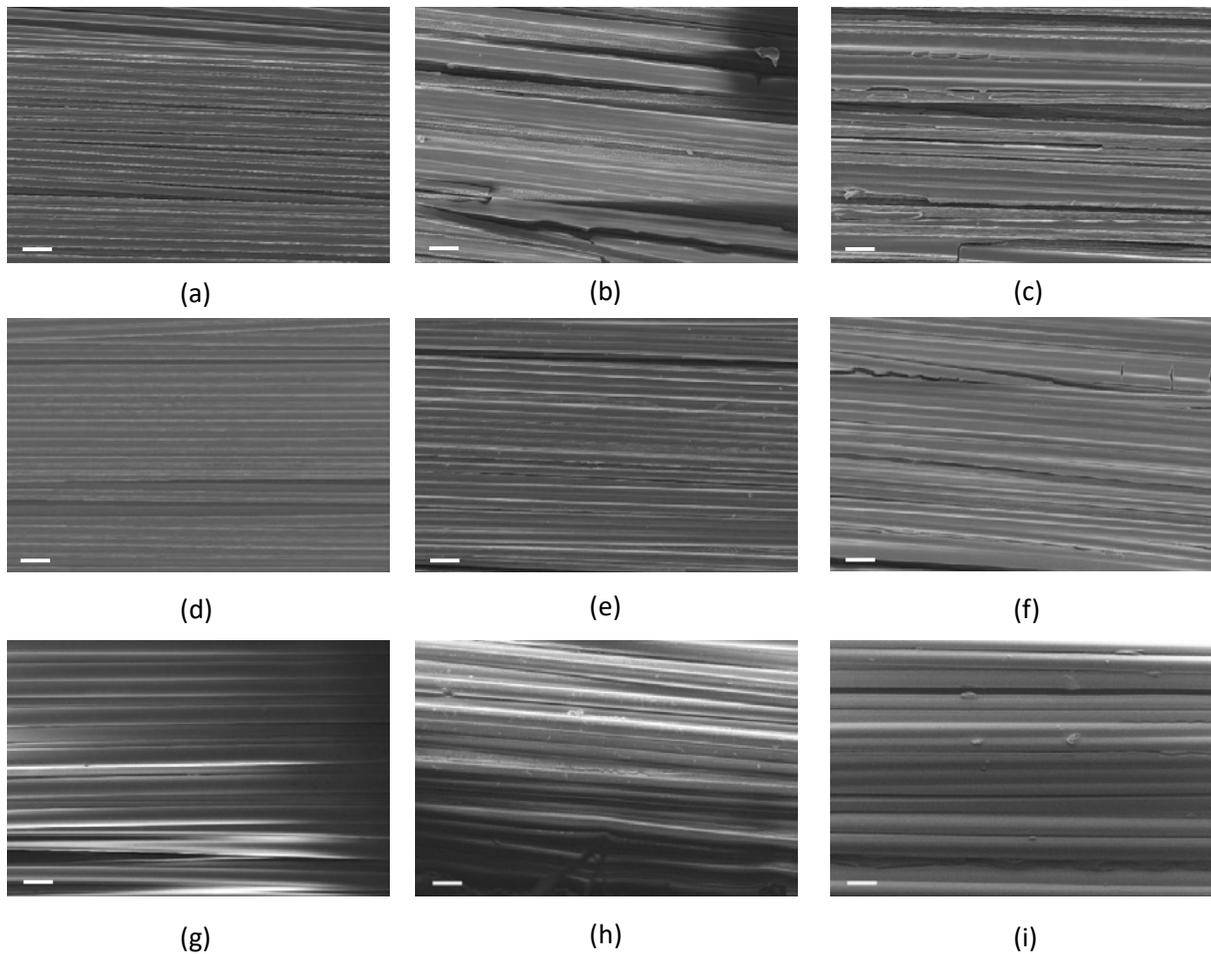


Figure 2. Representative SEM images for the following materials: HTA40 carbon fibre tape shown (a) as received, (b) with continuous silica aerogel coating and (c) with continuous silica aerogel coating applied after removal of sizing. HTS40 carbon fibre tape shown (d) as received, (e) with continuous silica aerogel coating and (f) with continuous silica aerogel coating applied after removal of sizing. E-Glass tape shown (g) as received, (h) with continuous silica aerogel coating and (i) with continuous silica aerogel coating applied after removal of sizing. All scale bars = 20 µm.

4. Conclusions

A capillary rise method has been devised for application of a macroscopically continuous silica aerogel coating to woven, aligned carbon or glass fibre tape. The effect of the fibre surface on the quantity and integrity of the aerogel coating applied to such fibres has been studied. For carbon fibre reinforcement a polyurethane-based sizing gives the most appropriate fibre volume fraction when compared epoxy-sized and de-sized alternatives. For glass fibre reinforcement a greater fibre volume fraction (lower aerogel excess) results when the fibres are treated with a pluricompatible size in comparison to de-sized fibres. In future, these findings will be applied to the fabrication of multi-ply composites of silica aerogel with carbon or glass fibre. These intermediate composites will subsequently be infused with epoxy resin to give hierarchical fibre composites with a bicontinuous silica-epoxy matrix.

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