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Influence of silica aerogel filler on strength-to-weight ratio of carbon/epoxy composite made by vacuum resin infusion

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Abstract. Thermoset matrix composites are a competitive solution in high-performance applications due to their superior characteristics. Several previous studies have shown that the addition of particles to thermosetting resins - particularly epoxy resins, which are currently the most widely used - can improve many of the physical properties of composites. In this context, this study aims to investigate the effect of silica aerogel content on the mechanical and weight properties of composites made via vacuum infusion. The results have shown that the strength-to-weight ratio increases with increasing filler percentage. However, the experiments also made it possible to recognise process limits, which occur when the percentage of aerogel in the resin exceeds 2%.

Introduction

Over the years, the transport industry has tended increasingly to replace conventional materials with composite materials, as in the case of hybrid composites[1], mainly because of the gain in lightness without sacrificing mechanical performance. Furthermore, the use of this type of material allows their mechanical, thermal, electrical, acoustic, etc. properties to be modified in a very flexible manner by inserting additives into the matrix. Commonly used fillers include carbon nanotubes (CNTs)[2,3], graphene, metal nanoparticles[4,5], or flame-retardant additives in cases where reinforcements are of organic origin[6] and silica aerogels[7,8]. Recent studies have shown that the introduction of the latter within the polymer matrix increases the mechanical, thermal and soundproofing properties of the composite[9,10]. For instance, Mazlan et al.[11] found that the addition of silica aerogel in a concentration of 2 wt% increased the flexural strength and modulus by 8 and 11%, respectively.

Impact resistance also improves with the introduction of silica aerogel. For example, Riahipour et al.[12] estimated that a 10 vol% aerogel percentage increased impact strength by about 100%. This trend, however, tends to reverse when the volume percentage of aerogels increases above 10%.

However, the use of nanoscale fillers presents certain problems including the lack of a strong interfacial bond between the filler particles and the polymer chains[13]. By using porous fillers with larger particle sizes, not only is the interfacial interaction between the matrix and the filler improved due to the infiltration of the resin within the inorganic scaffold of the aerogel[14], but there is also a benefit in terms of weight since more porous fillers are also less dense.

The aim of this work is to study the influence of silica aerogel within an epoxy matrix composite reinforced with carbon-fibre fabrics produced by vacuum resin infusion. In particular, using a porous filler, the aim of the work is to establish how the strength-to-weight ratio of the finished product varies with the amount of filler.

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Experimental details

Material and processing. An epoxy resin (SX8 EVO by Mates Italiana srl, Segrate, MI, Italy) and a carbon fiber fabric of 220 g/m^2 (provided by Industria Tessuti Tecnici srl, Lesmo, MB, Italy), with densities of 1.11 g/cm^3 and 1.8 g/cm^3 respectively, were used to produce the composite laminates.

The test campaign involved the production of four panels. For each laminate, the weight ratio of fibre to total mass was kept constant at 40%, while the percentage by weight between resin and silica aerogel particles with a density of 0.074 g/cm^3, varied according to three different ratios 100:2, 100:5 and 100:10. In addition, a reference composite type with no aerogel was also fabricated for the sake of comparison.

Table 1 summarizes some of the characteristics of the samples produced, in particular the percentage weight content of each material.

Table 1 – Denomination and composition of the different laminated panels

Sample denomination	Fibre content [%]	Epoxy content [%]	Silica aerogel content [%]
REF	40.00	60.00	0.00
AER2	40.00	58.82	1.18
AER5	40.00	57.14	2.86
AER10	40.00	54.55	5.45

Each composite laminate was made by vacuum bag technique from 510 mm (length) x 350 mm (width) x 2.5 mm (thickness) laminates, overlaying 7 layers of carbon fabric, using a flat glass plate as the bottom mould. A mesh of polymeric material was also placed on the upper carbon layer to improve resin distribution, which was otherwise made difficult by the low permeability of the bidirectional carbon fabric. The whole was then enclosed in a polymer material bag and epoxy resin infusion was then carried out.

Fig.°1 shows the final experimental set-up for the manufacture of the infused panels with the carbon fabric layers at the bottom, then the various film like the peel ply and the infusion mesh under the polymer bag.

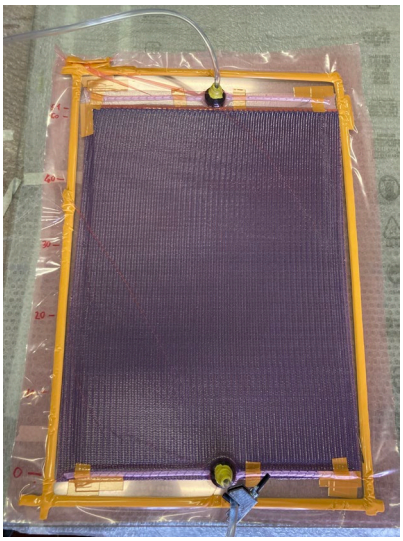

Fig.°1 – Experimental set-up for the infusion process

For laminates named REF and AER2, the infusion process was successful in achieving full impregnation of the carbon fabrics in about 90 min, by applying a vacuum pressure of 0.9 bar. The complete cross-linking of the resin was achieved after 1 day at T=23 °C and RH=50% and 15 h at

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T=60 °C and RH=50%. The processing time was high due to the low permeability of the carbon fabric and the presence of the aerogel, which decreased the fluidity of the resin. However, for the laminates named AER5 and AER10, impregnation was not successful because after about 30 min, the introduced amount of aerogel caused the formation of clots that prevented the resin from advancing. These results were consistent with those obtained from researches by other authors, which have shown that high percentages of filler are better in the case of hydrophobic material. Conversely, in the case of hydrophilic fillers, such as the one used in this paper, the best results are obtained with low percentages[15].

Fig.°2 shows the clots formed in the silicone connector for the resin inlet. Note the resin feed stop line in green.

Fig.°2 – Clots in the connector circled in red

Experimental methods. From the only two laminates that could be produced by vacuum infusion (REF and AER2), ten specimens each were extracted: five for the tensile test and five for the three-point bending test. The specimens were mechanically characterized in order to assess whether and how much the addition of the filler affected the bonding properties between the fibre and the matrix and thus the mechanical performance. To do this, a universal testing machine (Instron 8802) equipped with a 50 kN load cell was used.

Specifically, tensile tests were performed according to ASTM D3039 on rectangular specimens without tabs on the ends measuring 250 mm (length) x 25 mm (width) x 2.5 mm (thickness). The speed of the moving crosshead was set at 1 mm/min.

The three-point bending test was performed according to ASTM D790 on rectangular specimens with dimensions of 100 mm (length) x 14 mm (width) x 2.5 mm (thickness). The distance between the support rollers was set at 40 mm with a moving crosshead speed set at 1.2 mm/min.

Each test was conducted under standard laboratory conditions (T=23 °C and RH=50%) and 5 replicates (N=5) were tested per case. Fig.°3 shows the shape of one of the tensile specimens cut from the laminate.

Fig.°3 – Example of specimen extracted from the composite panel

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In addition, specific parallelepiped samples were obtained from the REF and AER2 infused laminates for density evaluation. Each sample was weighed three times and, in this way, together with the dimensions of each sample, the average density of these laminates was obtained.

Result and discussion

Fig.°4 shows tensile stress–strain characteristic. The values of stress and strain were calculated according to the ASTM standard.

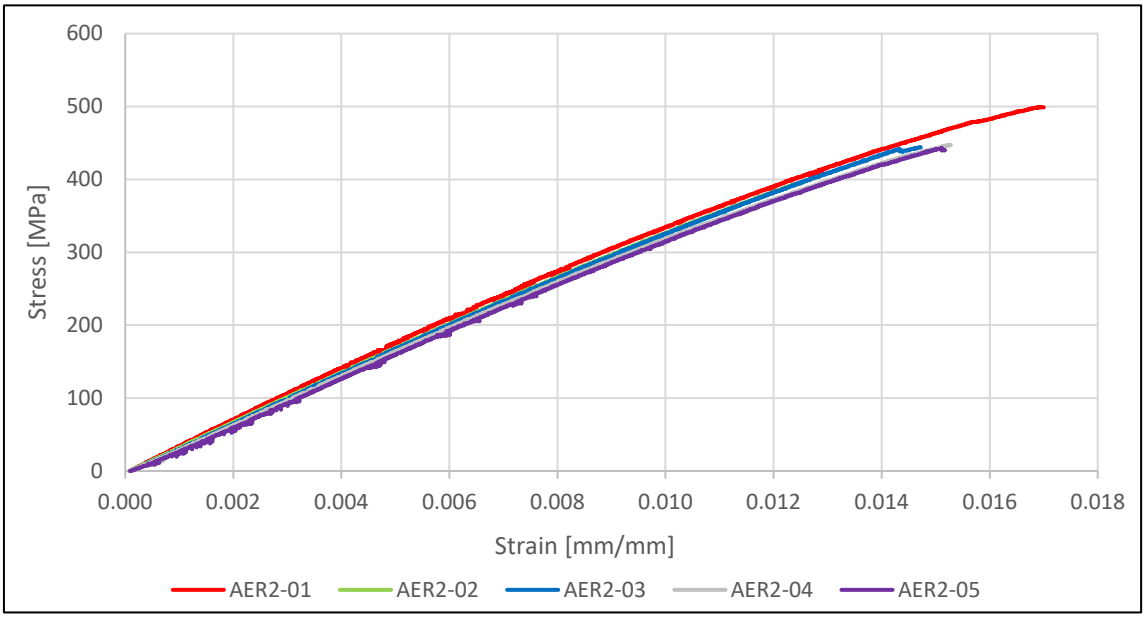

Fig.°4 – AER2 stress-strain graph for tensile test

Each test was considered valid because the failure of the specimens occurred in a section of the specimen within the gauge length and as can be seen from the graph, each test is practically superimposed on the other four.

Fig.°5 shows the fracture zone of one of the specimens after tensile testing.

Fig.°5 – Section of sample breakage AER2-01

The mechanical tests conducted on the materials have produced the results summarised in Table 2 and Table 3.

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Table 2 – Results obtained on the REF panel

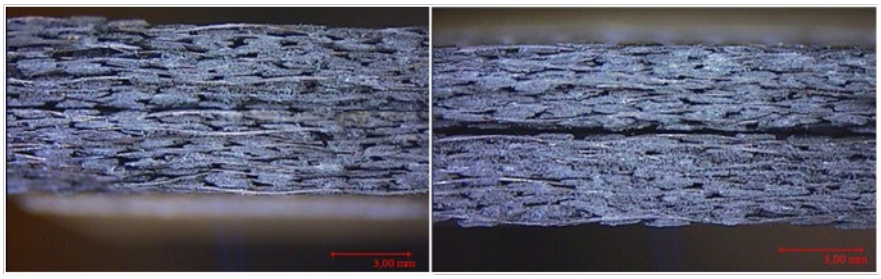
	Tensile resistance [MPa]	*Elongation at break [%]*	*Tensile modulus [GPa]*
REF-01	*413*	*1.6*	*33.7*
REF-02	*456*	*1.5*	*33.5*
REF-03	*374*	*1.5*	*32.9*
REF-04	*456*	*1.5*	*33.5*
REF-05	*439*	*1.4*	*35.4*
Average Value	*428*	*1.5*	*33.8*
Standard Deviation	*32*	*0.1*	*0.8*

Table 3 - Results obtained on the AER2 panel

	Tensile resistance [MPa]	*Elongation at break [%]*	*Tensile modulus [GPa]*
AER2-01	*499*	*1.7*	*35.3*
AER2-02	*400*	*1.3*	*34.9*
AER2-03	*444*	*1.5*	*35.7*
AER2-04	*448*	*1.5*	*34.2*
AER2-05	*443*	*1.5*	*32.3*
Average Value	*447*	*1.5*	*34.5*
Standard Deviation	*32*	*0.1*	*1.2*

After the tensile tests, the fracture surfaces were observed under 10x to 40x magnification to assess which fracture mechanism affected the section.

Fig.°6 shows images of both fracture surfaces placed one on top of the other, of one of the reference specimens and one of the specimens with 2 wt% aerogel.



(a) (b)

Fig.°6 – Sections of samples breakage at 10x magnification: REF-05 (a) and AER2-05 (b)

What can be seen from the images is that, on average, the fracture surface of the reference specimens is characterised by many inter-laminar cracks, which is less noticeable in the fracture surfaces of the specimens containing the aerogel; these are characterised by reinforcement failure or pull-out phenomena. This is indicative of a superior bond between the matrix and the reinforcement due to the presence of the additive.

Fig.°7 shows the stress-strain graph in the case of three-point bending tests on REF specimens.

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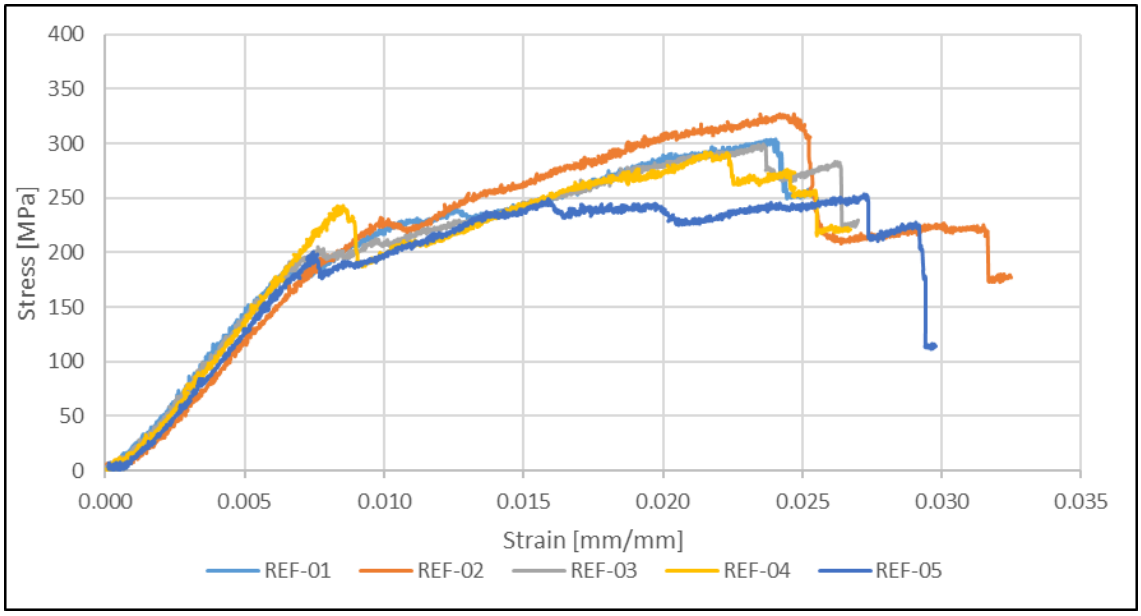

Fig.°7 – REF stress-strain graph for three-point bending test

The tension and strain values were derived from the force and displacement data output by the sensors using the formulae given in the standard.

Each test was considered valid since the failure occurred near the middle section of the specimen. A photo of test specimen AER-01 and its fracture section is shown as an example in Fig.°8.

Fig.°8 – Section of sample breakage AER2-01

Tables 4 and 5 summarise the results of the three-point bending tests conducted on the REF and AER2 laminates.

Table 4 – Results obtained on the REF panel

	Deflection at maximum load [mm]	*Maximum load [N]*	*Strain at maximum tension [%]*	*Flexural strength [MPa]*
REF-01	*3.33*	*575.2*	*2.4*	*303*
REF-02	*3.37*	*612.8*	*2.4*	*327*
REF-03	*3.32*	*536.8*	*2.4*	*299*
REF-04	*2.99*	*533.7*	*2.1*	*292*
REF-05	*3.79*	*459.2*	*2.7*	*254*
Average Value	*3.36*	*543.5*	*2.4*	*295*
Standard Deviation	*0.28*	*57.1*	*0.2*	*26*

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Table 5 - Results obtained on the AER2 panel

	Deflection at maximum load [mm]	Maximum load [N]	Strain at maximum tension [%]	Flexural strength [MPa]
AER2-01	3.61	676.6	2.6	324
AER2-02	3.81	591.4	2.7	337
AER2-03	3.53	545.5	2.5	294
AER2-04	3.36	609.3	2.4	357
AER2-05	3.38	561.5	2.4	308
Average Value	3.54	596.9	2.5	324
Standard Deviation	0.19	51.1	0.1	25

Furthermore, Tables 6 and 7 summarise the volume, average mass and density obtained for each sample of the REF and AER2 panels.

Table 6 - Volume, average mass and density of REF panel samples

	Volume [cm^3]	Average mass [g]	Density [g/cm^3]
REF-01	4.63	5.22	1.13
REF-02	4.58	5.17	1.13
REF-03	4.46	5.12	1.15
REF-04	4.44	5.07	1.14
REF-05	4.40	5.02	1.14
Average Value	4.50	5.12	1.14
Standard Deviation	0.10	0.08	0.01

Table 7 - Volume, average mass and density of AER2 panel samples

	Volume [cm^3]	Average mass [g]	Density [g/cm^3]
AER2-01	5.22	6.02	1.15
AER2-02	4.35	4.99	1.15
AER2-03	4.57	5.24	1.15
AER2-04	4.18	4.80	1.15
AER2-05	4.56	5.17	1.13
Average Value	4.58	5.24	1.14
Standard Deviation	0.40	0.47	0.01

Conclusion

The aim of this work was to evaluate the influence of silica aerogel in a carbon/epoxy composite produced via vacuum infusion.

The effect of the silica aerogel content on the tensile mechanical behaviour is not very relevant as the increases shown in the table are to be related to the relative sample standard deviation values. Even from the point of view of flexural behaviour, the performance increases obtained are in any case less than the uncertainty due to errors in the measurement chain.

What was certainly expected was a decrease in the density of the finished product, which was not achieved.

However, the results are nevertheless encouraging as it is important to emphasise that the tensile and flexural characteristics of the aerogel-added composite did not decrease, an event that could have occurred as the addition of an additive does not always maintain the adhesion between fibre

and matrix. Furthermore, the addition of a quantity of aerogel makes it possible to obtain a product whose thermal characteristics could improve, an aspect that will be investigated after this work.

These results, therefore, confirm that the introduction of composite materials in the transport sector can be a viable alternative to traditional structural materials such as aluminium alloys as they have very similar tensile strengths despite having lower deformability and being less rigid, with Young's modulus values of approximately half. To the advantage of composite materials, however, due to their lower density compared to aluminium alloys, weight can be further reduced for the same size of the finished product. Furthermore, it has been shown how the introduction of additives can further increase the strength-to-weight ratio of composite materials.

It remains to be explored how the dynamic behaviour of composites is modified by the introduction of silica aerogels.

Since the results obtained with the addition of up to 2 wt% aerogel did not produce any performance gains in mechanical terms, it was decided to continue the research by further increasing the amount of aerogel within the epoxy matrix. However, an increase of more than 2 wt% created problems regarding the feasibility of impregnation by vacuum infusion.

To fully impregnate the reinforcements, it is necessary to adopt different production techniques, such as hand lay-up, with the knowledge that purely manual techniques such as the one mentioned above make it difficult to control the uniform distribution of filler that should be homogeneous.

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