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ScienceDirect
Journal of the European Ceramic Society 31 (2011) 1821–1826
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An integrated assembly method of sandwich structured ceramic matrix composites

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Received 5 November 2010; received in revised form 28 February 2011; accepted 7 March 2011
Available online 2 April 2011

Abstract

Sandwich structured composites have been widely studied and applied at ambient temperature in aeronautical, automobile and naval applications. For high temperature applications, an integrated ceramic sandwich structure could take advantage of multiple functions such as skin stiffness and core insulation. For thermo-structural applications, skins must be made of ceramic matrix composites (CMC) because of their strength, their resistance to high temperatures (beyond 1000 °C), and their low densities. Concerning foam cores, some carbides (e.g. SiC) are, for their outstanding thermo-mechanical properties, the most appropriate. These foams can withstand long oxidative exposing conditions with low material degradation. This paper presents an assembly method of SiC based sandwich structured CMC. It is performed during sandwich manufacturing in an integrated fashion and allows the production of complex shapes at low costs. Produced flat sandwich panels, characterized by three point bending tests, showed a marked toughening behaviour.

Keywords: Sandwich structures; Joining; Composites; SiC; Structural applications

1. Introduction

A sandwich structured composite is typically produced by joining two thin but stiff skins to a lightweight but thick core.[1] The core is usually made of a lower strength material, but its thickness offers to the overall structure high flexural stiffness with low density. These structures are widely employed at ambient temperatures for aeronautical, automobile, naval applications.

For high temperature applications sandwich structured composites become extremely interesting because they can combine weight reduction and stiffness with heat insulation.

In the aerospace field, insulating structures incorporating, ribs and stiffeners have been already manufactured. Pichon et al. developed a concept called "shingle" in which the sandwich was divided into two sets of elements: some with mechanical functions (shell, fasteners, and stand-offs), and others with thermal functions (inner insulation layers, seals and insulating washers).[2]

Integrated sandwich structured composites for high temperature applications have been studied with the goal of reduction of weight and number of parts to be assembled. A core material that can withstand high temperatures, thermal shocks and mechanical loads is represented by ceramic foams produced with the replica method.[3]

Reticulated SiC foams bear high thermal loads[4] and, due their high porosity (>80%), they have rather low effective thermal conductivities.[5,6] In comparison with reticulated vitreous carbon (RVC) foams, if passive oxidation conditions are met, SiC foams can operate for long time at high temperatures (∼1400 °C) in oxidative environments.[4,7]

A sandwich in which skins were made of SiC tape casted bulk sheets and the core of expanded poly(silsesquioxane) co-fired after sandwich assembly was produced by Hoefner et al.[8] During bending tests, the brittle behaviour of the skins caused sandwich crushing.

For thermo-structural ceramic sandwiches, ceramic matrix composite (CMC) skins together with structural ceramic foams need to be employed. The first attempt to realize a high temperature structured sandwich made of CMC skins and a chemical vapour infiltration (CVI) SiC foam core was reported by Fisher in 1985.[9] Recently NASA developed a sandwich made with

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doi:10.1016/j.jeurceramsoc.2011.03.010

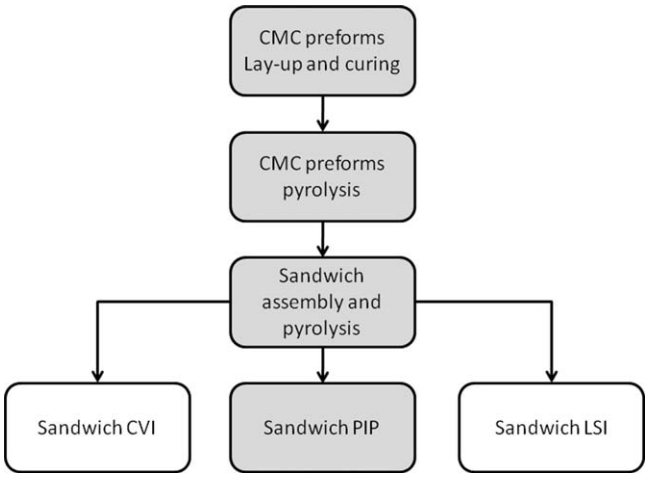

Fig. 1. CMC SiC sandwich manufacturing procedure.

C_f–SiC_f/SiC_m skins and a SiC core, processed through integral densification of the CMC and foam core.[10] In this work SiC is deposited by CVI both onto fibres and the core preform. These structures do not present a joining layer. Authors point out that skins and core coefficient of thermal expansion must be similar for both processing and operation.

If bonding between skins and core has to be employed, during bending, because stresses in the composite material changes rapidly between the core and the skin, the joining layer experiences some degree of shear stress. Thus skin to core bonding must be carefully optimized in order to avoid local delamination.

CMC have been bonded with: mechanical fasteners,[2,11] glasses,[12,13] brazing alloys,[14–16] and pre-ceramic polymers.[17] Krenkel et al. applied in situ preform joining with additional densification via liquid silicon infiltration.[18]

The present work illustrates a procedure to assemble ceramic sandwich structures in any shape with a low-cost manufacturing technique employed in polymer matrix composite fabrications. Skins and core are assembled at the very beginning of the sandwich manufacturing. The assembled sandwich structured composite "preform" can be further densified with the common techniques employed for CMC manufacturing (Fig. 1). Polymer impregnation and pyrolysis (PIP) was employed.

2. Materials

For the CMC laminates, fibres employed were SiC plain weave (200 g/m^2) Tyranno LOXM (UBE, J), fibre density 2.48 g/cm^3, fabrics were compacted with phenolic novolac resin (Hexion Specialty Chemicals). For CMC densification a polysilazane preceramic polymer Ceraset 20 (Kion Speciality Polymers, Clariant, CH) was utilised.

A slurry with two SiC powders of different grain size and Ceraset polymer was prepared to bond CMC preform skins to the ceramic core. The fine SiC powder is UF-15 α-SiC (H.C. Starck, D), it has a specific surface area of 15 m^2/g with a particle size distribution D90% = 1.2 μm. The coarse α-SiC powder (Washington Mills, USA) has grit size F800.

ERBISIC-R foams, produced with the replica method, (Erbicol SA, CH) were used as porous ceramic core. The foam bulk material is made of α-SiC powder in a β-SiC/silicon matrix. Bulk average density is 2.83 g/cm^3. The reticulated foam is an isotropic three-dimensional network of hollow ceramic ligaments. Pore sizes are ∼5 mm (equivalent to 10 pores per inch (PPI)). Foams characteristics are reported in Table 1.

3. Experimental

SiC_f preforms were obtained by laying up fabric plies, pre-impregnated with a small amount of phenolic resin (2% of the fabric weight), and curing (T = 150 °C, p = 1.5 bar) in an autoclave. Laminates with different thicknesses were obtained by lying up 4 (S1) and 12 (S2) fabrics. Wider laminates were realized from which, panels were cut with the same in-plane dimensions of the SiC foams.

Composites were pyrolysed in flowing argon with the heat ramp shown in Fig. 3. Laminates were then measured and weighted, their characteristics are reported in Table 2.

On the pyrolysed porous preforms, a slurry (30 wt% fine SiC powders, 30 wt% coarse SiC powders, and 40 wt% Ceraset) was deposited onto one side of each laminate. Due to the slurry viscosity (ν = 8100 mPa s) it not fully infiltrated SiC_f preforms, acting as a thick bonding layer (Fig. 7). The two laminates were placed on the top and the bottom of each foam, as shown in Fig. 2, and cured in the autoclave with the same cure cycle pre-

Table 1
ERBISIC-R foam dimensions and weight.

Sample ID	L [cm]	W [cm]	t [cm]	Volume [cm^3]	Mass [g]	Density [g/cm^3]	Porosity [%]
ErbisicR1	18.5	13.6	1.5	377.40	134.31	0.36	87
ErbisicR2	18.4	13.6	1.5	375.36	141.30	0.38	86

Table 2
Composite preform properties after curing and first pyrolysis.

Laminate	Number of layers	Thickness [cm]	Volume [cm^3]	Mass [g]	Porosity [%]
S1 – top	4	0.10	25.39	27.77	55
S1 – bottom	4	0.10	25.39	27.75	56
S2 – top	12	0.28	70.06	84.50	51
S2 – bottom	12	0.28	70.06	84.40	51

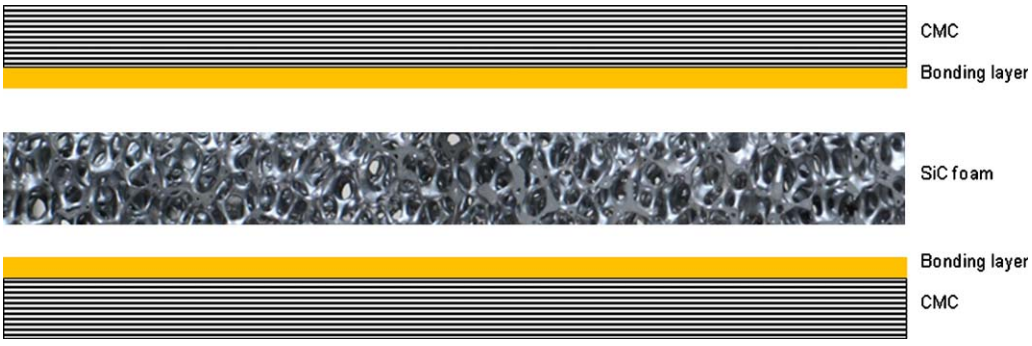

Fig. 2. Sandwich assembly.

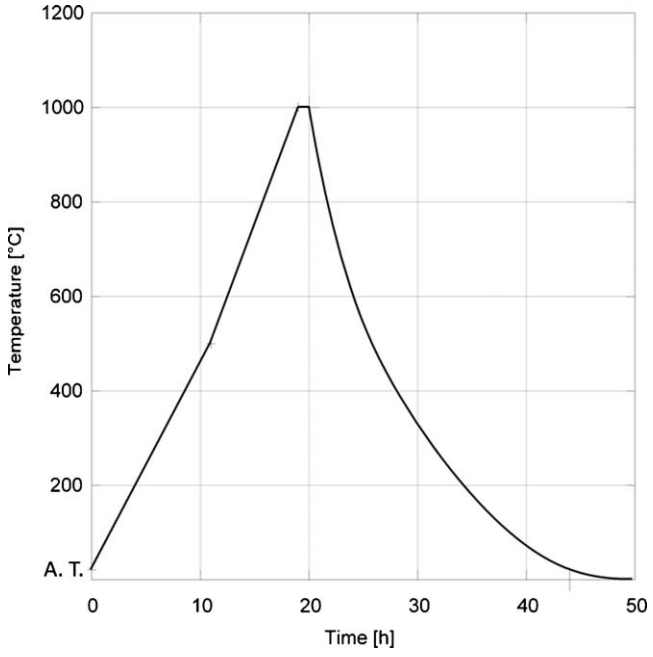

Fig. 3. Pyrolysis thermal cycle (Ar atmosphere).

viously described. Once cured, the sandwich was placed into the furnace and pyrolysed (Fig. 3). The assembled sandwiches were then impregnated on both sides of the sandwich skins. Impregnations with the Ceraset polymeric precursor were performed using a brush. Sandwiches were then placed into a furnace and pyrolysed with the heat ramp shown in Fig. 3. After each pyrolysis weights were recorded, data are reported in Table 3.

Table 3
Sandwiches S1 and S2 weights and weight gains, after each processing step.

Operation	S1		S2	
	Weight [g]	Gain [%]	Weight [g]	Gain [%]
Sandwich assembly and pyrolysis	240.50	–	360.75	–
PIP 1	254.97	6.0	403.18	11.8
PIP 2	263.31	3.3	421.2	4.5
PIP 3	266.50	1.2	432.9	2.8
PIP 4	271.55	1.9	438.7	1.3
PIP 5	272.71	0.4	449.2	2.4

PIP was repeated 5 times, since after the fifth cycle the CMC densification was judged to be sufficient to attain adequate skins strength for bending tests.[22]

Sandwich panels were cut with a diamond saw in five pieces with the following in plane dimensions: 139 mm × 25 mm.

4. Characterization

Three point bending tests (Fig. 4) were performed with an universal testing machine (Zwick/Roell Z050, Germany) using a span of 100 mm. Cross head speed was set to 0.01 mm/s. Set up and dimensions were chosen to compare current data with previous works.[4]

Fig. 5 shows that sandwich structured composites exhibit an average flexural strength which is roughly 6 times higher the experimental data[4] and the corresponding analytical value. The last one (2.64 MPa) calculated with the Gibson and Ashby formula $\sigma = \sigma_0 C(\rho/\rho_0)^{(3/2)}$[19] using a bulk material flexural strength σ_0 of 280 MPa (CarSIK-G, Schunk, D), a relative density ρ/ρ_0 of 0.14 and a constant C of 0.18.[20] Fig. 5 also shows that skin thicknesses, and thus number of layers, do not to affect sandwich overall flexural strength. Despite the fragile behaviour of bare foams, sandwich structures experienced a marked toughening behaviour (Fig. 6). Flexural strength, after reaching a maximum, dropped in both sandwiches due to foam ligament failures. Essentially every drop into the chart of Fig. 6 corresponded to one or more ligament tears. CMC, with uncoated SiC fibres, did not break since they were loaded below their stress limit.[23] A bending curve of a bear foam taken from previous experiments[4] was added in Fig. 6 to show its fragile behaviour.

By measuring the area under the stress–strain curves of S1 and S2 samples in Fig. 6, one can assume that the resilience of the S2 sandwich is higher than S1.

Samples, cut from S1 and S2 sandwiches, were milled and polished to be analysed by optical microscopy. Observation was performed using a optical microscope (Leica DMLM, Germany). Images were acquired using a digital camera (Leica DFC 280, Germany).

Fig. 7 shows that foam struts are stuck into the bonding layer as a result of the previously described integrated assembly method. Fig. 8 illustrates also that the bundles of the external CMC lamina facing the core, have been infiltrated by the slurry during its deposition. Bonding layer thickness is uniform along

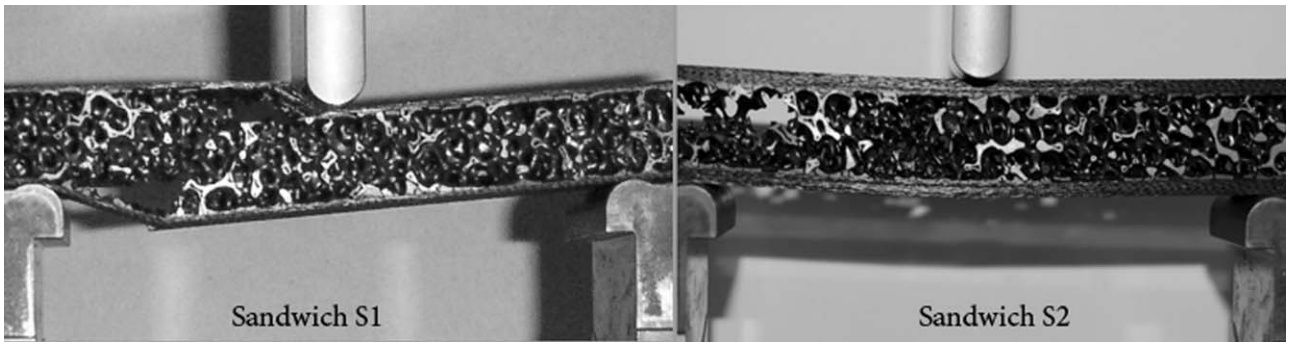

Fig. 4. Three point bending test at 2% strain. S1 sandwich (4 layers) and S2 sandwich (16 layers).

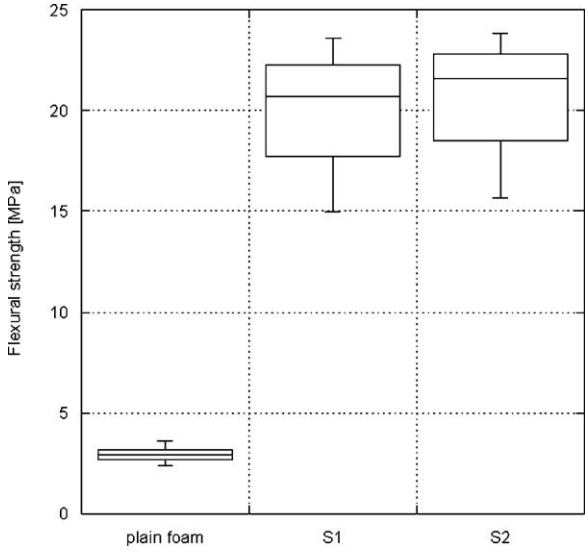

Fig. 5. Flexural strength of S1, S2 sandwiches and a plain foams with equivalent porosity (86%).

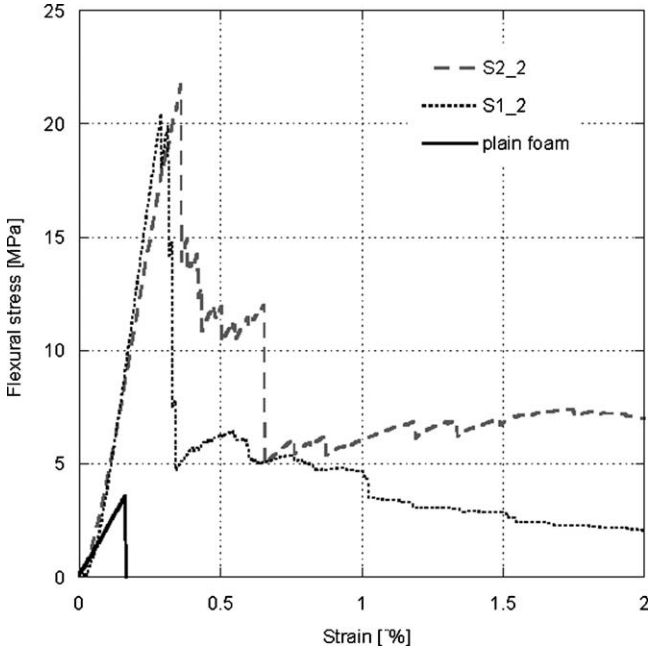

Fig. 6. Stress–deflection curve under three point bending of sandwiches S1 (sample #2) and S2 (sample #2) and a plain foam with equivalent porosity (86%).

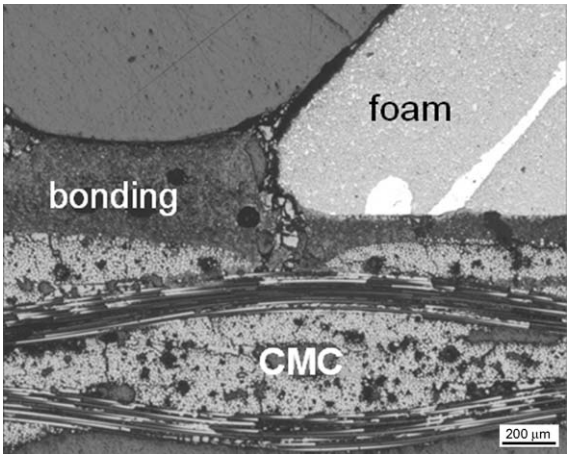

Fig. 7. Bonding layer wrapping the foam ligament.

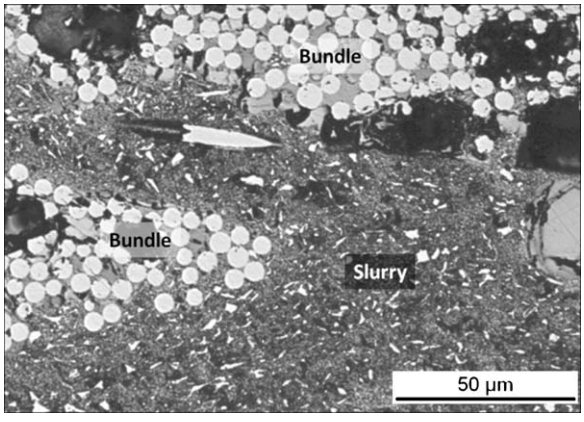

Fig. 8. Partial CMC preform bundles infiltration by the bonding slurry.

the sandwich (Fig. 9) but, due to slurry shrinking during pyrolysis, it presents an uniform pattern of cracks (Fig. 10) which are filled by the following PIP cycles.

5. Discussion

Joining skins to ceramic core before their actual densification allows a strong grip of the bonding layer both to the CMC laminates and to the foam. Both CMC and foam are embedded into the polymer derive ceramic layer and thus mechanically fastened to it. The layer presents several cracks

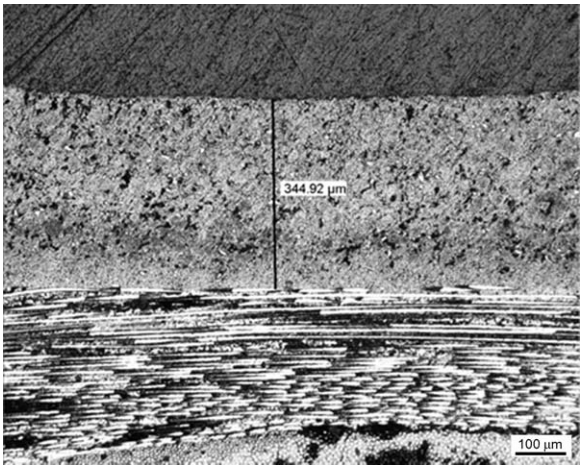

Fig. 9. Bonding thickness.

trough the thickness due to its shrinking during its first pyrolysis. Nevertheless it remains sufficiently compact and thus allows load transfer from the skins to the core. Indeed, in all the bending tests, no failure was observed into the bulk bonding layer; foam struts were always the first to fail. Struts embedding into the bonding layer effectively works as a joint between the CMC and the foam (Fig. 7) but, in order to promote joining, skins and core thermal expansions upon heating and cooling should be similar. Different coefficients of thermal expansion lead, during heat treatments, to weak joints or debonding.[21] Even when de-bonded, sandwiches still present a sort of toughness due to foam strut-by-strut breaking behaviour.[21]

Since the slurry, due to its controlled viscosity, was able to infiltrate the CMC perform bundles and few fibres inside the bundles (Fig. 8) the relevant mechanical joint is strong.

Bending strength was similar in thinner (S1) and thicker (S2) skin sandwiches. In both samples an angled crack ($\theta > 30^\circ$) was observed (Fig. 4). Sandwich cores broke because shear stresses were becoming predominant.[23] Crack position was different from sandwich to sandwich in agreement with the fact that, in three-point bending, unlike normal stresses, shear is approximately constant throughout the core volume.[23]

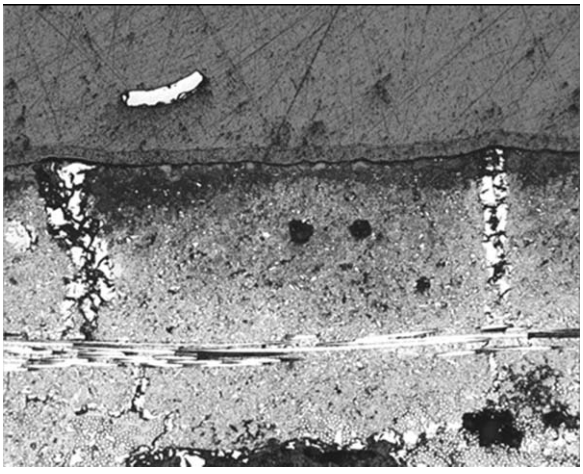

Fig. 10. Bonding crack formation during pyrolysis and further filling by PIP.

All tested sandwiches exhibited a marked toughening behaviour under bending. This can be ascribed to foam strut-by-strut failure mode, evidenced by load drops in the stress–strain curves of Fig. 6 and by acoustic emissions. This failure mode was found to be similar to the fibre pull out behaviour in CMC fracture mechanics.[23]

6. Conclusions

An integrated assembly method of silicon carbide sandwich structured composites was presented. The assembly was performed during component manufacturing in an integrated fashion. Sandwich strengths higher than that of bare foams, prevented that CMC and core coefficient of thermal expansion are similar. After their maximum stress these ceramic structures did not experience a catastrophic failure. Further work needs to be done in order to: understand their mechanical behaviour at ambient and high temperatures and see how sandwich structured ceramic matrix composites, densified with the other techniques shown in Fig. 1, behave.

References

1. Wikipedia contributors. Sandwich structured composite. Wikipedia, The Free Encyclopedia; 29 October 2010, Web. 2 November 2010.
2. Pichon T, Barreteau R, Soyris P, Foucault A, Parenteau JM, Prel Y, et al. CMC thermal protection system for future reusable launch vehicles: generic shingle technological maturation and tests. *Acta Astronaut* 2009:165–76.
3. Colombo P. Conventional and novel processing methods for cellular ceramics. *Philos Trans R Soc A* 2006;**364**:109–24.
4. Ortona A, Pusterla S, Fino P, Mach FRA, Delgado A, Biamino S. Aging of reticulated Si–SiC foams in porous burners. *Adv Appl Ceram* 2010;**109**(4):246–51.
5. Ortona A, Pusterla S, Valton S. Reticulated SiC foam X-ray CT, meshing, and simulation advances in bioceramics and porous ceramics III. In: Narayan R, Colombo P, editors. *Ceramic engineering and science proceedings*. Wiley; 2011.
6. Xu W, Zhang H, Yang Z, Zhang J. The effective thermal conductivity of three dimensional reticulated foam materials. *J Porous Mater* 2009;**16**:65–71.
7. Mach RA, Issendorff v, Delgado F, Ortona AA. Experimental investigation of the oxidation behavior of Si–SiC-foams. *Adv Bioceram Porous Ceram: Ceram Eng Sci Proc* 2009:299–311.
8. Hoefner T, Zeschky J, Scheffler M, Greil P. Light weight ceramic sandwich structure from preceramic polymers. *Ceram Eng Sci Proc* 2004:559–64.
9. Fisher R, Burkland C, Busmante W. Ceramic composite thermal protection systems. *Ceram Eng Sci Proc* 1985;**6**(7/8):806.
10. Hurwitz FI, Steffier W, Koenig, Kiser J. Improved fabrication of ceramic matrix composite/foam core integrated structures. *NASA Tech Briefs* 2009:36.
11. Kochendorfer R, Lutzenburger N, Weihs H. Joining techniques for fibre ceramic structures. *Adv Compos Lett* 2004:59–69.
12. Ferraris M, Salvo M, Isola C, Appendino Montorsi M, Kohyama A. Glass–ceramic joining and coating of SiC/SiC for fusion applications. *J Nucl Mater* 1998:1546–50.
13. Ferraris M, Salvo M, Casalegno V, Ciampichetti A, Smeacetto F, Zucchetti M. Joining of machined SiC/SiC composites for thermonuclear fusion reactors. *J Nucl Mater* 2008:410–5.
14. Riccardi B, Nannetti CA, Petrisor T, Woltersdorf J, Pippel E, Libera S, et al. Issues of low activation brazing of SiC_f/SiC composites by using alloys without free silicon. *J Nucl Mater* 2004:562–6.

15. Riccardi B, Nannetti CA, Petrisor T, Sacchetti M. Low activation brazing materials and techniques for SiC_f/SiC composites. *J Nucl Mater* 2002:1237–41.
16. Riccardi B, Nannetti CA, Woltersdorf J, Pippel E, Petrisor T. Brazing of SiC and SiC_f/SiC composites performed with 84Si–16Ti eutectic alloy: microstructure and strength. *J Mater Sci* 2002:1237–41.
17. Colombo P, Riccardi B, Donato A, Sfarini G. Joining of SiC/SiC_f ceramic matrix composites for fusion reactor blanket applications. *J Nucl Mater* 2000:127–35.
18. Krenkel W, Henke T, Mason, Fuentes N, Martinez Esnaola M, Daniel JM. In-situ joined CMC components. In: *CMMC 96 – first international conference on ceramic and metal matrix composites. Key engineering materials*. 1997. p. 313–20.
19. Gibson L, Ashby M. *Cellular solids: structure and properties*, 3. NY: Pergamon; 1988.
20. Brezny R. Mechanical behavior of open cell ceramics. PhD thesis. PA, USA: Penn State University; 1990.
21. Ortona A, Pusterla S, Gianella S. C_f/SiC_m CMC–SiC foam sandwich preparation and characterization. In: *Conference proceedings HTCMC7*. 2010.
22. Sherwood WJ. Composite fabrication and CMCs. In: Colombo P, Riedel R, Sorarù GD, Kleebe H-J, editors. *Polymer derived ceramics*. Lancaster: DEStech Publications, Inc.; 2010.
23. Van Voorhees EJ, Green DJ. Failure behavior of cellular-core ceramic sandwich composites. *J Am Ceram Soc* 1991;**74**:2747–52.