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Abstract

SiC foams sandwiched between two Ceramic Matrix Composite (CMC) skins are of interest for several high temperature applications ranging from aeronautics to energy production.
In this paper, SiC foams were joined to C/SiC composites by the ''Mo-wrap" method to obtain sandwich structures. The Mo-wrap method is a recently developed joining technique: it consists of wrapping Si foils inside a Mo wrap in order to prevent molten silicon leaking from the joined area and infiltrating SiC foam and C/SiC during the joining process.
Compression and thermal shock resistance tests were performed on the C/SiC – SiC foam – C/SiC sandwich obtaining sound results.

1. Introduction

Light-weight high temperature stable structures are highly demanded in aerospace and energy applications. [1]. Ceramic sandwich structures offer weight reduction and high insulation potential: in particular ceramic foam/CMC sandwich structures are produced by joining two stiff/strong CMC skins with a porous ceramic foam core [2]. The core material is typically of lower strength and lower elastic modulus than the skins [3]. The ideal core material can withstand high working temperatures, thermal shocks and mechanical loads: materials with a tailored porosity have exceptional properties which can be designed for a specific application and are not possible with conventional materials [4]. Ceramic foams (i.e. SiC foams) have a great potential in high temperature aerospace and energy applications as core materials for sandwich structures.

The sandwich structure proposed in this work is made of CMC skins (i.e. Chemical Vapour Deposition, CVD-SiC Coated C/SiC) which are light weight, thermal resistant and stable in harsh environments. SiC foams used as core material, are potentially suit- able for applications between 1300 and 1400 °C, with an excellent lifetime in combustion environment [2,5].

It is quite challenging to produce strong sandwich structures with a high porosity (>80%) because of the few connecting points between the skin and the core; moreover, joining material can infiltrate into the pores, thus causing weak joints, ineffective insu- lation and low heat/energy recovery.

A wide range of joining materials have been used so far for join- ing the [6] CMC but only a few techniques are reported to join cellular structures to CMC skins [3,7]. In the past, NASA [5] manufactured a sandwich of C-SiC/SiC skins joined to a SiC core, obtained by integral densification of the CMC and the SiC foam; in this case no joining process was required. The process of joining skin to foam by *in situ* joining via impregnation and pyrolysis allows sandwich structures to be assembled in any shape, but requires autoclave treatment and the repetition of the densifica- tion processes [2].

In this study a CVD-SiC coated C/SiC is joined with a SiC foam to produce a sandwich structure (referred to as *sandwich* in the text) by using the new brazing technique called ''Mo-wrap". The joining material is a composite, made of an *in-situ* formation of molybde- num

disilicide ($MoSi_2$) particles in a silicon matrix. Mo-wrap braz- ing is a cost-effective pressure-less joining technique which requires a non-reactive atmosphere [8].

2. Experimental part

Keraman® C/SiC composites (density = 1.7–2.5 g/cm^3) were manufactured at MT Aerospace (Germany) using the standard gra- dient Chemical Vapour Infiltration process and supplied as flat samples; the 2D composites were coated with a protective CVD-SiC layer (5–15 mm).

Molybdenum foil (thickness of 25.4 mm, Alfa Aesar Germany, 99.95% purity) and silicon foils (thickness of 584 mm, MEMC Elec- tronic Materials, Italy, 99.95% purity) were used as joining materials.

The core was a Si-SiC 3D foam (EngiCer SA, Balerna, Switzer- land); it is an isotropic network of hollow ligaments with an open cell porous ceramic structure (porosity = 88%) produced by the replica method [4,9,10]. The foam characteristics are reported in ref [2].

Two skins of C/SiC were cut into samples of about 25 12 mm to match the dimensions of the foam block and then placed on the top and the bottom of the foam to produce sandwiches, as shown in Fig. 1(a) using Mo and Si foils as joining materials, according to the sketch in Fig. 1(b).

The joining process was performed at 1450 °C for 5 min (heat- ing rate of 1000 °C/h) under Ar flow. No external pressure was applied to the sandwich assembly, except for the 30–50 g over weight to avoid the sample misalignment during the thermal treat- ment. Titanium sponges were used as an oxygen getter. Sand- wiches were cross-sectioned to analyze the joint morphology by scanning electron microscopy (SEM, QUANTA INSPECT 200, Zeiss SUPRA TM 40), with Energy Dispersion Spectroscopy (EDS EDAX 9900).

The thermal shock tests were performed in air according to [11]. The sandwiches were introduced into a furnace at 1100 °C and held at this temperature for 2 min, then cooled in air to simulate the atmospheric re-entry real conditions; the thermal cycle was repeated three times. The cooling rate was 4 K/s.

The sandwiches were tested under compression at room tem- perature with a crosshead displacement rate of 1 mm/min using a universal mechanical testing machine (SINTEC D/10) equipped with a 5 kN load cell. The compression tests were performed on sandwiches, applying the load orthogonally to the joined inter- faces. Special fixtures were designed to clamp the specimen into the loading frame, by pressing the two skins of C/SiC. At least 3 samples were tested both for bare foam and skin-foam joints.

3. Results and discussion

3.1. Joining

The microstructure of the joints manufactured using the Mo- wrap method is shown in Fig. 2. The $MoSi_2$/Si composite joining material is well distinguishable in Fig. 2a); it spreads across the whole joined area showing a uniform thickness. As expected, the highly porous SiC foam is joined to the CMC skin in few connecting points.

The temperatures involved in the joining process are above Si melting point; as a consequence, Si infiltration into the foam pores is an issue. Controlling the silicon infiltration and at the same time producing strong joints is quite challenging. Four Mo-Si based join- ing procedures were adopted in our earlier study in order to avoid the infiltration into substrates [8], resulting in the development of the one-step, cost effective and pressure-less ''Mo-Wrap" joining technique. The joining material itself is an *in-situ* composite mate- rial ($MoSi_2$ particles embedded in a Si matrix), with a thickness of 170–200 mm, as observable in Fig. 2a). Silicon low viscosity at its melting point is beneficial for wetting the surfaces to be joined, but it is difficult to control, particularly with porous substrates. In the Mo-Wrap technique, silicon provides enough liquid phase to achieve substrate wettability while molybdenum wrap confines the silicon to the joining area, thus avoiding infiltration.

The continuous joint interface is evident in the cross section micrograph (Fig. 2) for both interfaces of SiC foam and C/SiC. No free Mo was found nor any compound containing C was

detected; due to faster diffusion of Si in refractory metals (i.e. Mo, Ta, Nb), silicides are formed instead of carbides [8]. In this study, only $MoSi_2$ was formed as a reaction product between Mo and Si; this is particularly beneficial for the oxidation stability behavior of the sandwich, since $MoSi_2$ has a very high melting point and shows excellent stability in an oxidative environment. Foam ligaments are encompassed into the joining material and the skin facing the joining material is partially infiltrated (Fig. 2a and b), thus lead- ing to a strong sandwich structure. Moreover, no degradation of the SiC foam was detected; the observable cracks in the SiC foam ligaments were also present in the as-received foams.

3.2. *Mechanical and thermal shock tests*

Compression tests were performed on C/SiC – foam joined spec- imens and the as-received SiC foam. The joints gave an average value of 710 N, comparable with that of the as-received SiC foam (700 N), but with larger displacement at fracture, thus confirming the soundness of the sandwich structure. Regardless of the brittle character of the bare SiC foam, the sandwich structures experi- enced a certain toughening behavior, as shown in Fig. 3. The curves show a typical pseudo-plastic behavior as expected for these sand- wiches, compared to the more brittle one of the bare SiC foam; every drop in the graph corresponds to a foam cell strut failure [9], thus demonstrating that the weak point of the sandwich structure lies in the foam structure itself and not in the joined area. Fracture surface analysis revealed that the failure occurred within the SiC foam and not at the SiC foam-C/SiC interface; the connecting points remain firmly attached on both skins. This demonstrates the effec- tiveness of the Mo-wrap to join not only flat skins, but also highly porous structures to uneven surfaces such as the C/SiC ones, despite having very few connecting points.

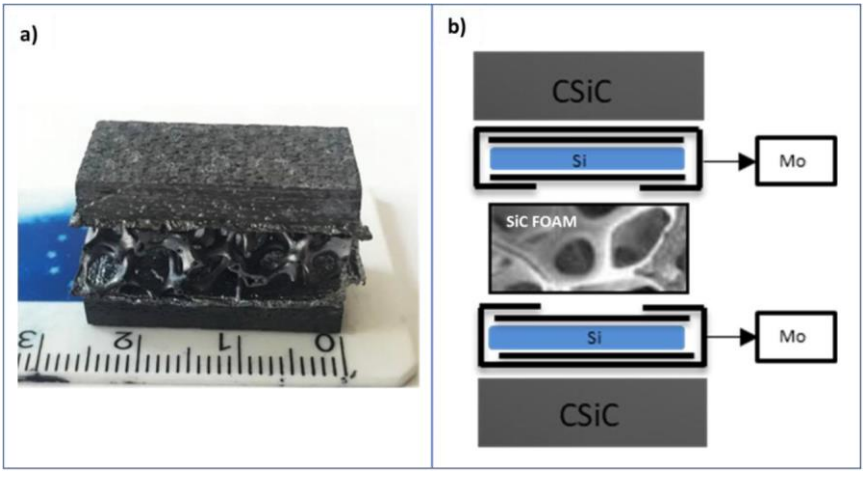

Fig. 1. C/SiC-SiC foam sandwich structure (a) prepared by the Mo-Wrap joining technique (b).

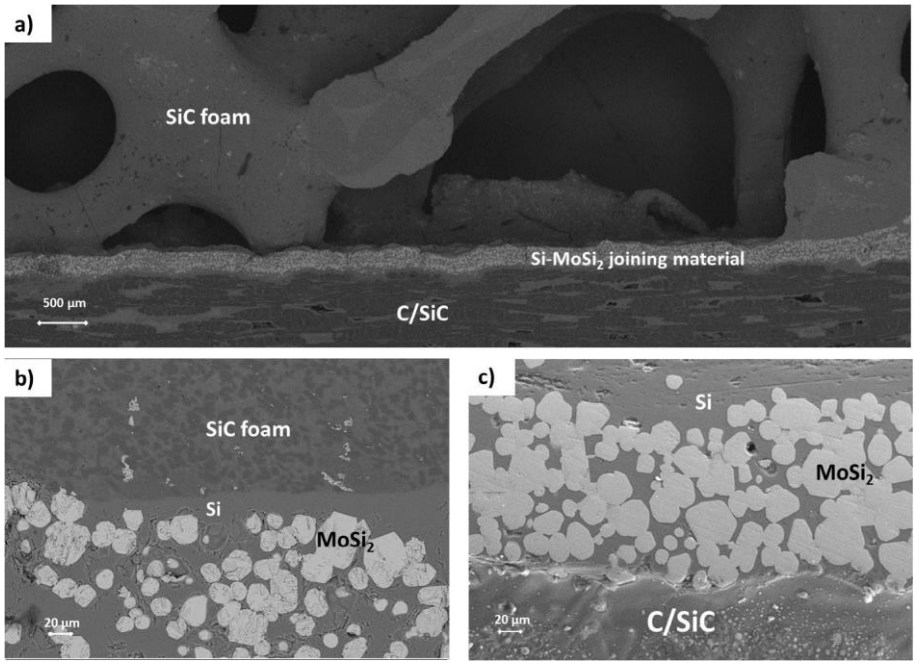

Fig. 2. SEM cross section of a C/SiC-SiC foam sandwich (a) prepared by means of the Mo-Wrap joining technique; magnification of the joined area of a C/SiC-SiC foam sandwich before (b) and after thermal shock test at 1100 °C in air (c).

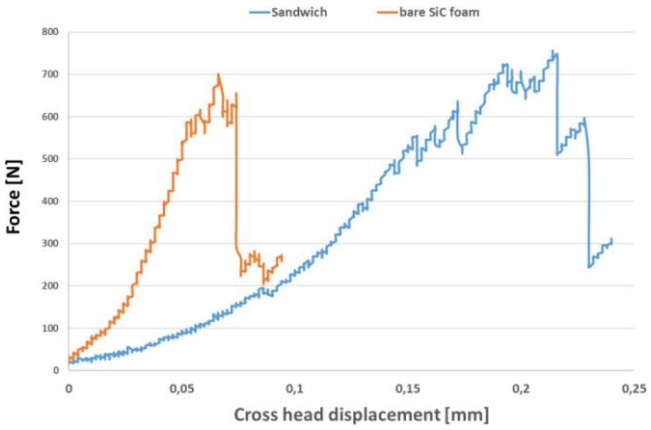

Fig. 3. Force-displacement curves of a SiC foam and a joined C/SiC-SiC foam sandwich tested under compression.

Thermal shock tests were performed on the joined samples by cycling three times in air up to 1100 °C. The primary observations revealed that the specimens showed no visible damage and the sample surface was not oxidized. Neither the interface Mo-wrap/ foam, nor the Mo-wrap itself changed morphology (Fig. 2c) or com- position (checked by EDS analysis, not reported here) after thermal shock tests.

4. Conclusions

C/SiC – SiC foam – C/SiC sandwich structures were successfully manufactured using the pressure-less ''Mo-Wrap" joining tech- nique, based on a $MoSi_2$/Si composite joining material. This tech- nique was effective in joining highly porous SiC foams to C/SiC composites and avoiding detrimental infiltration. Compression tests carried out on the sandwiches confirmed the soundness of the sandwich structure; moreover, the sandwich structures were able to withstand thermal shock from 1100 °C to RT.

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