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On the nonlinear mechanical behavior of macroporous cellular ceramics under bending

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Abstract

This work presents the results of an experimental work on the mechanical behavior under bending of Si–SiC reticulated foams. Particularly it is here studied a transient regime after their linear elastic behavior and before their catastrophic failure. During this regime part of the mechanical work is dissipated by local struts failure. This behavior is interesting because, if well understood, could give important information on the prediction of reticulated ceramics' life time. Besides load cell and deflection readings, acoustical emission and electrical resistance were also recorded during the tests. Data were further processed using Weibull statistics.
Results: evidence that the duration and effects of this regime strongly depend on ligaments' microstructure and thus on the manufacturing technique. A correlation with the average acoustical events per sample and the Weibull modulus is also evidenced.

Keywords: Cellular ceramics; Nonlinear mechanical behavior; Silicon carbide; Acoustical emission; Electrical resistance monitoring

1. Introduction

Ceramic foams are highly porous materials (above 70% porosity) consisting of gas-filled pores, i.e. cells, whose edges are made of interconnected solid ligaments. As silicon carbide foams at high temperatures show good thermal conductivity, low thermal expansion, good thermo-mechanical and chemical resistance, they are more and more used in components for burners,[1] liquid metal filtering, thermal protection,[2] concentrated solar plants, and other high temperature applications.[3] The properties of the foam's constituent material always depend on the manufacturing method it was made with. Although different methods exist for manufacturing such materials,[4] they can be summarized as: replica, direct foaming, and fugitive pore forming.[5] Replica is the most industrialized method for manufacturing SiC foams: it utilizes the impregnation of polymeric foams with a ceramic slurry. In some cases silicon infiltration is performed to convert the residual carbons from the pyroloysis of the binder into β-SiC.[6] Due to presence of unreacted Si in their final microstructure, these foams are commonly called Si–SiC foams. Ceramic foams produced by replica are often employed in structural applications because of their high effective mechanical properties despite their porosity. Foams' effective properties are always a fraction of the value corresponding to the solid or monolithic material constituting their skeleton.[7]

It is well known that monolithic ceramics show a linear mechanical behavior during loading until they fail catastrophically by rapid cracks propagation. This assumption does not apply on the same material when it is highly porous. In present of pores a progressive damage can be observed based on the place and morphology of the pores. Merkert et al. observed the signs of prefracture microcracking in artificially porous alumina.[8] For highly porous ceramics where the pore spacing is lower than the pore diameter damage occurs prior to fracture by a so called "pore-crack linking". This phenomena has been observed experimentally[9] and numerically[11] in different papers. In foams made of fragile materials, cracks propagate by consequent breaking of ligaments,[13] turning their mechanical behavior from linear to nonlinear. This transition from brittle to "cellular" behavior was recently observed on cellular materials.[14,15] In particular

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Meille et al.[15] studied failure of cellular ceramics with 40–70% macro-porosity. They observed that as macroporosity exceeds 50%, the brittle behavior is gradually substituted by a cellular behavior.

This work shows that Si–SiC foams under bending experience first a linear elastic regime similar to their skeleton monolithic material but, beyond a deflection corresponding to ~40% of their deflection at failure, the weakest struts[12,16] start to fail, and a new regime appears. Struts then fracture until the whole foam separates in two halves. This study aims at shedding more light on this nonlinear regime showing how micro and macro-porosities lead to different fracture behaviors.

To monitor this mechanical behavior, specific sensors can be employed. Piezoelectric sensors and optical fibers have been already used for monitoring polymer composites, their use is expensive, complicated and unpractical for ceramic foams.[17] On the other hand, techniques like X-ray CT scans, ultrasonic C-scan and infrared thermography are difficult to integrate in the mechanical testing machine. Recently researchers have tried to use the electrical resistance (ER) of the material as a sensing technique of the damage in composites.[17–19] This method is inexpensive, simple, does not need any embedded sensor and can gather real time damage information. In this study ER monitoring is utilized with acoustical emission (ACE) to give in situ information about the damage in the foams.[20]

Since when testing ceramic materials, mechanical properties are significantly scattered, output data were analyzed using Weibull statistics.

2. Experimental approach

2.1. Samples preparation

The specimens used in this work were Si–SiC reticulated foams (ERBISIC-R Erbicol SA Balerna CH). Foams were made by the replica method[21] followed by Si infiltration.[6] The impregnation of polyurethane foams was performed with a slurry made of α-SiC powders, plastic binders and solvent. After drying, the green body was pyrolyzed in Argon above 1000 °C. Finally, the foam was infiltrated with molten Si at 1550 °C and 10^{-2} mbar residual pressure to form β-SiC through the reaction bonding between the polymer derived carbon, and the molten silicon (1) [22]:

$$Si_{liquid} + C_{solid} = SiC_{solid}[\Delta H = -73 \text{kJ mol}^{-1}] \quad (1)$$

Further properties of ERBISC foams can be found in our previous works.[3,23] Typically the amount of Si utilized during the infiltration is higher than what needed for the corresponding stoichiometric reaction. The unreacted Si fills the micropores by capillary forces[24] leading to a so called a Si–SiC foam.[3] Three batches were prepared for silicon infiltration with three different amounts of silicon, in respect of their weight after pyrolysis, in order to progressively fill their pores (Table 1).

The final product is a macroporous reticulated ceramic, microscopically made of reaction bonded β-SiC, α-SiC powders, Si, and a low amount of residual carbon (depending on how much Si was added).

Diamond tool cutting and milling was employed to machine bars $(14 \times 25 \times 170)\,mm^3 \pm 0.05$ mm from rectangular plates $(15 \times 145 \times 195)\,mm^3$.

Microstructure evaluations were performed with an optical microscope (Leica DMLM, Wetzlar, D)

2.2. Bending set up

Two copper plates $(5 \times 10 \times 0.5)\,mm^3$ were brazed at the ends of each foams (Cusil-ABA Paste, Morgan Crucible Company, UK) at 790 °C in Argon to ensure electrical connections (Fig. 1). Electrical resistance was measured with the four probe method.[17] A constant current of 500 μA was introduced into the foam through the copper plates, and the voltage measured every 0.513 s (1.95 Hz). Acoustical data were recorded by means of a microphone (MD321N Sennheiser, D) with a smooth frequency response ranging from 40 to 20,000 Hz particularly designed for industrial use and noise measurement. ACE signals were recorded at a 51,200 Hz frequency. The microphone was placed at the center of the specimens and as close as possible to them. The setup was acoustically insulated from the outside with a dedicated box. Four point bending tests were performed using a universal testing machine (Z050, Zwick/Roell, D) as depicted in Fig. 1. A 5 kN load cell (Zwick/Roell, D) and an LDT, with a repeatability of 2 μm (TR25, Novotechnik, Southborough, MA, USA) were used to measure respectively the load and the deflection of the beam. A crosshead speed of 0.1 mm min^{-1} and a preload of 10 N were selected as loading conditions.

Data from ACE, ER, LDT and the load-cell were recorded and synchronized by means of Labview (National Instrument, Austin, TX, USA). Data reduction was performed with MATLAB (MathWorks, Natick, MA, USA).

3. Results and discussion

3.1. Foams morphology

The three types of Si–SiC foams with different Si content were labeled as 30Si, 22Si and 12Si (Table 1). The samples

Table 1
Some physical properties of the foams. Samples were grouped by percentage of unreacted Si. All the foams present pore size of ~5 mm, struts with thickness of 0.83 ± 0.13 and macroporosity of 87%. Total porosity was calculated by adding calculated skeleton microporosity to the foams' macroporosity.

I.D.	Free Si (wt.%)	Number of specimens	Theoretical skeleton density (g cm^{-3})	Actual skeleton density (g cm^{-3})	Skeleton microporosity (%)	Total porosity (%)
30Si	30	21	2.95	2.68 ± 0.05	9.2	88.20
22Si	22	23	3.07	2.66 ± 0.07	13.3	88.73
12Si	12	20	3.10	2.55 ± 0.06	17.7	89.30