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Ablation resistance of precursor derived Si-Hf-C-N(O) ceramics

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ABSTRACT

Polymer derived Si-Hf-C-N(O) foams were produced by the pyrolysis of polysilazane containing varying amounts of hafnium tetra *n*-butoxide. X-ray tomography was used to determine the cell size distribution. The ablation resistance of the foams was studied by subjecting it to different oxyacetylene flame temperatures. After ablation, fragmentation was not observed and the mass ablation rate was found to be minimal for the foams produced from highest vol% of hafnium tetra *n*-butoxide. The foams remained X-ray amorphous after ablation and the presence of Hf—O and Si—O bonds were confirmed using ATR spectroscopy. For comparison, a reasonably dense spark plasma sintered pellet was also ablated and the evolution of monoclinic hafnia was confirmed using X-ray diffraction. The microstructural characterization exhibited three different zones on the ablated surface for sintered sample and the ablation mechanisms were understood using thermodynamic calculations.

1. Introduction

The ablation resistance of materials is a major concern in the aerospace industry since several components of a hypersonic spacecraft are subjected to temperatures as high as 1650° C [1] due to aerodynamic heating during re-entry. Concomitantly, the design demands the material to possess high strength in addition to low density. Recent studies [2,3] focus on the usage of sandwich structured composites ie., ceramic foam positioned between ceramic matrix composite panels. The ceramic foam which forms the core of the composite must withstand high temperature, mechanical load and thermal shock resistance [4]. A detailed review on the optimal design of foams suggests that micro-architected cellular materials possess superior mechanical properties [5]. Recently, in SiC foams, it was shown that the pore and strut diameter affects the heat transfer (thermal radiation) [6]. The usage of these foams leads to reduction in weight of the structural components, thus increasing the fuel efficiency. Currently, some of the established methodologies for producing foams are by using a replica [7], use of sacrificial templates [8,9], direct foaming and reaction techniques [10–12]. In the replica method, the polymeric solution will be coated/immersed in a polyurethane sponge, which upon pyrolysis leads to a porous ceramic. The sacrificial template method envisages the use of a sacrificial template into which the polymeric precursors will be impregnated followed by pyrolysis to remove the template. The direct foaming method involves the creation of bubbles in the polymeric solution so as to produce a foamy structure where as in the reaction technique the foamy structure is produced as a result of the reaction that takes place between the polymers and fillers. In this study, the initial precursor mixture when subjected to pyrolysis leads to the formation of bubbles. It is believed that the since the viscosity of the mixture is high, the bubbles do not break and moves upwards, eventually leading to the foam formation. The release of butanol and gases such as CH_4, NOx, CO_2 and CO during pyrolysis is expected to be the reason for pore formation.

Several studies were carried out to develop material systems that possess high oxidation resistance, thermal shock resistance and chemical stability at higher temperatures. The material system being studied involves C/C composites [13–15], ultra high temperature ceramics (UHTC's) coated C/C composites [16–21] and C_f/Si_3N_4 [22]. However, the debonding of these coatings at high temperatures was a major concern. Recently, ablation resistance studies were performed on SiBCN based ceramic composites with fiber reinforcements such as SiC, C [23] and their combinations [24]. The fabrication of the above mentioned material systems were carried out using several methods comprising of chemical vapor infiltration [25,26], reactive melt infiltration [27], slurry infiltration by hot pressing [28], and precursor infiltration and pyrolysis (PIP) [29]. In this study, we have adopted polymer derived ceramic

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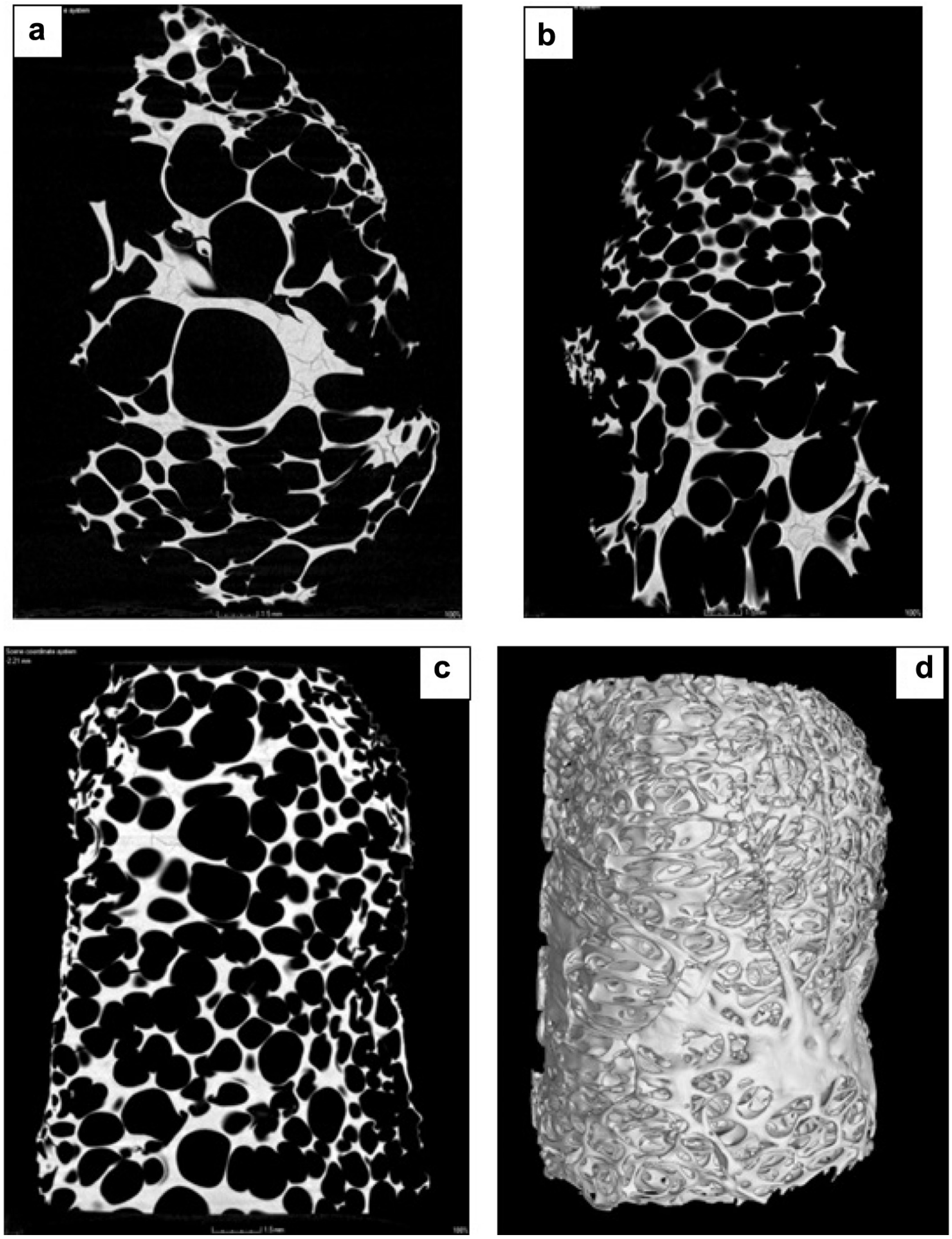

Fig. 1. 2D slice of front view of the foams produced from (a) 5 vol% HfTb (b) 15 vol% HfTb (c) 25 vol% HfTb and (d) 3D CT-X ray tomographic image foam derived from 25 vol% HfTb.

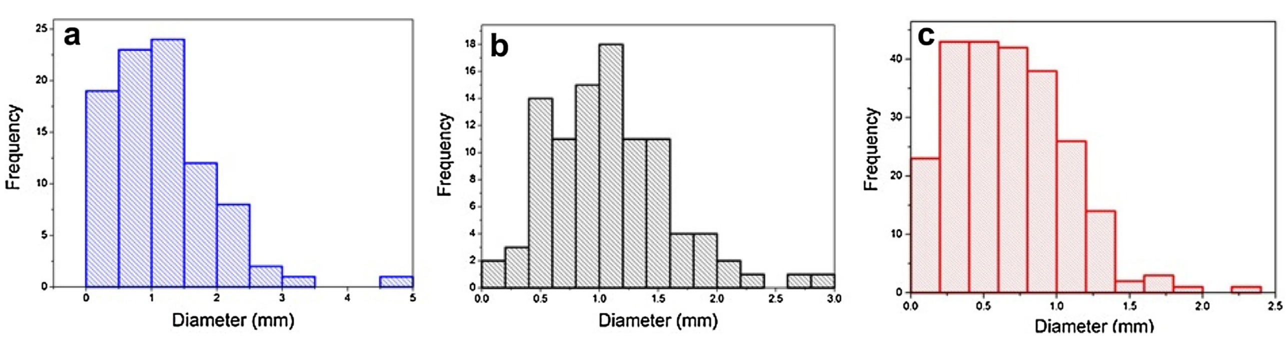

Fig. 2. Average cell size distribution of the foams produced from (a) 5 vol% HfTb (b) 15 vol% HfTb and (c) 25 vol% HfTb.