



Reaction bonded silicon nitride foams: The influence of iron disilicide on microstructure and mechanical strength

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Received 25 November 2014; received in revised form 10 December 2014; accepted 10 December 2014

Available online 17 December 2014

Abstract

In this study open-cell reaction bonded silicon nitride (RBSN) foams with high and controlled level of porosity have been produced based on the wet processing of Si-PMMA suspensions in the presence of iron disilicide (FeSi_2) as an additive. The investigation focuses on the effect of FeSi_2 on the mechanical strength, nitriding mechanisms, and microstructure of the RBSN foam. The nitriding reactions and the foam microstructures and properties were investigated under various nitriding conditions and for different FeSi_2 contents.

It was observed that 1 wt% FeSi_2 significantly increases the strength of the RBSN foams regardless of the nitriding temperature and atmosphere. The nitriding conditions have a strong influence on RBSN foam strength. The maximum strength was achieved under a N_2 atmosphere at 1390 °C with a foam porosity of 71 vol%. The high porosity level of the foam significantly affects the nitriding mechanisms and consequently the foam microstructure, compared to “dense” RBSN ceramics. FeSi_2 promotes the formation of both α - and β - Si_3N_4 phases depending on its concentration and its addition also increases the whisker content of the foam microstructure due to an increase in SiO and Si vapor formation.

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Keywords: B. Porosity; C. Strength; Silicon nitride; Reaction bonding; Iron disilicide

1. Introduction

Ceramic foams have recently been found to be very attractive since they have opened up many new applications including filters, membranes, catalyst supports, interpenetrating composites, and bioimplants [1–3]. Among the engineering ceramic materials, Si_3N_4 foams have not received sufficient attention due to the difficulties of working with Si_3N_4 powder and also the high cost of the material [4]. It must be mentioned that working with Si_3N_4 powder as the starting material requires dealing with its very high sintering temperature and also large linear shrinkage (15–20%) which makes costly post-sintering machining steps necessary [5]. Si_3N_4 suspensions have also shown poor and inconsistent rheological behavior which is a major issue from the industrial point of view [6,7]. Therefore, it is of great interest to fabricate Si_3N_4 foams with tailored properties based on a cost-effective fabrication procedure avoiding the issues of

working with Si_3N_4 powder. The procedure must also be capable of producing Si_3N_4 foams with homogeneous open-cell structures and a high level of porosity which can be precisely controlled. All the above-mentioned advantages can be achieved through gel-casting of Si suspensions containing sacrificial phases combined with subsequent reaction bonding. This newly developed and near net-shape fabrication procedure is capable of producing RBSN foams which benefit from controlled and high levels of porosity (between 41 vol% and 87 vol%), pore interconnectivity, ease of fabrication, and low production cost. It must be mentioned that there are only a few studies on the fabrication and characterization of RBSN foams [8–11]. The RBSN foams in these studies have been produced based on dry-processing techniques such as uniaxial pressing resulting in closed-cell structures with low levels of porosity (maximum porosity < 60 vol%).

Design and optimization of the RBSN foam microstructure and consequently its properties are not possible unless the reaction bonding phenomenon of the porous foam is completely understood. Although reaction bonding in “dense” RBSN

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ceramics has been studied in the past, there is still disagreement in the literature regarding the nitriding reactions and mechanisms. Moreover, it must be stated that nitriding of porous foams is far more complicated than the nitridation of “dense” RBSN ceramics. There are two foam properties, namely a high level of porosity and also pore interconnectivity, which markedly influence the nitriding mechanisms and consequently the morphology of the Si_3N_4 phases formed during reaction bonding of RBSN foams. These microstructural features affect the nitridation of RBSN foams in terms of Si volatilization rate, the free space required for Si_3N_4 formation and growth, morphology of the Si_3N_4 phases, and the nitridation rate. The creation of local atmospheres including Si, SiO, and atomic/molecular nitrogen within the porosity network of the RBSN foams also influences the dominant nitriding reactions. Therefore, it is of great importance to study the nitridation process of the RBSN foams where the foam microstructural features play a significant role.

Another important parameter which significantly affects the nitriding reactions and consequently the properties of the final RBSN foam is the presence of iron compounds in the system either as an impurity or as an additive. The effect of Fe compounds was always of great concern in “dense” RBSN ceramics since it has a substantial influence on the nitriding reactions. It must be mentioned that commercial Si powder has approximately 0.9 wt% of iron [12,13] and even in high purity Si powder, the presence of minor amounts of iron impurity is almost unavoidable [14]. Iron may influence both the dominant nitriding mechanism and the ratio of α - to β - Si_3N_4 phases. It has also been mentioned that Fe significantly deleteriously affects the mechanical strength of the “dense” RBSN ceramics [12,13]; therefore, it has always been recommended to avoid even minor concentrations of Fe in “dense” RBSN ceramics. However, the influence of Fe on the nitriding reactions of highly porous RBSN can be quite different compared to “dense” RBSN ceramics. Understanding the effect of Fe compounds on nitridation of RBSN foams also makes it possible to utilize commercial grades of Si powder. This results in much more cost-effective RBSN foams compared to the employment of high-purity grades of Si powder. Therefore, investigating the role of Fe compounds on the nature of the nitriding reactions and consequently on the RBSN foam properties is significantly important. To the best of the authors' knowledge, there is no investigation on the influence of FeSi_2 on the microstructure, mechanical strength, and properties of highly porous RBSN foams fabricated by wet processing of Si-PMMA suspensions. This investigation aids the understanding of the nitriding mechanisms of the porous RBSN foams in the presence of FeSi_2 . The mechanical strength of the RBSN foams has been tested under different nitriding conditions including different temperatures, atmospheres, and FeSi_2 contents. The Si_3N_4 phases formed during nitriding, and their morphologies, have also been investigated in depth via XRD and SEM analysis and the nitriding mechanisms are thoroughly discussed. Finally, the influence of FeSi_2 on the foam microstructure and the formation of α - and β - Si_3N_4 phases has been extensively studied.

2. Experimental procedure

2.1. Foam fabrication

RBSN foams with open-cell structures and interconnected porosity were fabricated based on a combination of gel-casting, sacrificial template, and reaction bonding techniques. Suspensions containing high purity Si powder (ABCR, 99.995%, $-8\ \mu\text{m}$), PMMA beads (Microbeads, 10–40 μm), and FeSi_2 (Alfa Aesar, 99.9%) were transformed into a two-phase solid body via aqueous gel-casting. The gel-casting system was based on acrylamide (AM, Sigma-Aldrich, $\text{C}_2\text{H}_3\text{CONH}_2$) as the monomer and N,N'-methylbisacrylamide (MBAM, Sigma-Aldrich ($\text{C}_2\text{H}_3\text{CONH}_2$) $_2\text{CH}_2$) as the crosslinker with the weight ratio of AM to MBAM of 15. The monomer and the cross-linker were dissolved in water followed by the addition of Si powder and PMMA beads. PMMA beads play the role of sacrificial phases and leave residual porosity in the structure after polymer pyrolysis. DS001 (Polymer Innovations Inc.) as a deflocculant, Poly(acrylamide) (Acros Organics, M.W.=5,000,000–6,000,000) as a binder, and DF002 (Polymer Innovations Inc.) as an antifoaming agent were also added to the suspension. The suspension pH was adjusted to 8.5 followed by ball milling and homogenization. The addition of a catalyst (ammonium persulfate, Sigma-Aldrich, $(\text{NH}_4)_2\text{S}_2\text{O}_8$) and an initiator (N,N,N',N'-tetramethylethylenediamine, Sigma-Aldrich, $\text{C}_6\text{H}_{16}\text{N}_2$) started the polymerization process followed by casting in silicone rubber molds. Drying and polymer pyrolysis were performed as the next steps in order to remove water and the sacrificial phases from the cast bodies, respectively. At this stage, a silicon foam has been produced and the Si to Si_3N_4 conversion was finally performed through the reaction bonding process. Fig. 1 shows the flowchart of the fabrication procedure. More details of the RBSN foam fabrication procedure, the foam characterization, and its properties can be found in our previous study [15].

Since this study investigates the nitridation and microstructure of the RBSN foams containing FeSi_2 , various nitriding conditions including temperatures ranging from 1350 °C to 1450 °C and static atmospheres of N_2 and N_2 -4% H_2 were utilized. The heating rate during nitriding was 5 °C/min up to 1350 °C, 1 °C/min up to 1390 °C, and 0.3 °C/min up to 1450 °C. The dwell time was about 6 h at the maximum nitriding temperature. The temperature range used in this study covers nitriding temperatures both below and above the silicon melting point (1410 °C) which can be used to study the influence of Si melting on the foam strength. In order to study the effect of Ar-sintering on the microstructure and strength of the foams, some samples were sintered under Ar-4% H_2 for 2 h at 1200 °C prior to the nitriding step. Based on the nitriding conditions, specific codes have been used for sample identification. In the sample codes used in the rest of the study, ArH shows the performance of a short sintering step under the Ar-4% H_2 atmosphere, NH shows nitriding under N_2 -4% H_2 , and N represents nitriding under the N_2 atmosphere. The final nitriding temperature can also be found in the last part of the sample code. As an example, ArH-NH1400 indicates a sample that has been sintered under Ar-4% H_2 and then nitrided under N_2 - H_2 at 1400 °C.

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