Key Engineering Materials
ISSN: 1662-9795, Vols. 317-318, pp 693-696
doi:10.4028/www.scientific.net/KEM.317-318.693

Online: 2006-08-15

Fabrication and Characteristic of Porous Alumina Developed with SiC Nano-fiber or Nano-whisker

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Keywords: Porous alumina, SiC, Gel-casting, Precursor

Abstract. Porous alumina green bodies were fabricated by a gel-casting method, for which the slurries of alumina (AKP-30) and PMMA (polymethylmetacrylate) bead were mixed and dispersed at 1:1, 1:2, 1:3, and 1:4 volume ratios. PMMA bead as precursor of carbon source was used for growing SiC nano-fiber or nano-whisker during a VLS (vapor-liquid-solid) reaction at 1450°C for 9hrs. PMMA beads are converted to carbon particles after calcination at 1300°C for 9hrs in static argon (Ar) atmosphere. Finally, carbon particles remain in pores of alumina bodies connected by continuous pore channels. Fe solution was infiltrated into the porous alumina bodies, which are absorbed on the carbon particles in pores. Fe precursor functions as a seed to develop the SiC nano-fiber or nano-whisker in the porous alumina bodies. The liquid droplets formed at the end of the SiC fiber or whisker are evident for the typical VLS mechanism. The microstructure of the SiC fiber or whisker grown by the VLS reaction was observed by SEM. The porosity was measured by mercury porosimeter. The formation behavior of SiC fiber or whisker is dependent on volume ratio of carbon converted from PMMA bead.

Introduction

SiC materials have been employed in a variety of application, such as catalyst support and hot-gas filter, because of their low thermal-expansion coefficient, good thermal shock resistance, and chemical stability at elevated temperature [1-4]. Recently, a shape memory synthesis (SMS) technique has been widely used to fabricate SiC materials with the desired morphology [5]. This technique is CVD-modified process by a vapor-solid (VS) reaction between gaseous SiO and carbon. For the synthesis of SiC materials, we selected carbon as a solid precursor, which can be obtained from calcinations of polymethylmetacrylate (PMMA) in argon (Ar) atmosphere, and Si-SiO powder mixture as a $SiO_{(vapor)}$ precursor. The aim of this work is to propose the porous alumina (Al_2O_3) developed with SiC nano-fiber or nano-whisker, which can enlarge the surface area of Al_2O_3 materials. In this paper, we describe the fabrication parameters for proposing the porous alumina with SiC nano-fiber or nano-whisker, and its mechanism is discussed.

Experimental procedure

Alumina (α-Al_2O_3, AKP-30, Sumitomo Co., Japan) and PMMA bead (SUNPMMA-LD100C, Sunjin Chemical CO., LTD. Republic of Korea) were used as starting materials for preparation of porous bodies. First, green bodies were fabricated by a gel-casting with the mixed slurries of α-Al_2O_3 and PMMA bead, which were prepared at 1:1, 1:2, 1:3, and 1:4 volume ratios. Green bodies were calcined to convert PMMA beads to carbon particles at 1300°C for 9hrs in Ar atmosphere. Carbon particles remain in pores after calcinations in argon (Ar) atmosphere, due to nature of PMMA. Fe precursors that functions as a seed for the SiC nano-fiber or nano-whisker growth was infiltrated into porous alumina bodies with carbon particles. Silicon (Si, Deajung Chemicals & Metals Co., Ltd., Republic of Korea) and silica powders (SiO_2, Junsei, Japan) were used as starting materials with reagent grade, which are employed as precursors for the synthesis of the SiC nano-fiber or nano-whisker in a vapor-liquid-solid (VLS) reaction. The SiC nano-fiber or nano-whisker was synthesized by the VLS reaction between SiO (gas) and carbon particles in porous alumina at a given reaction temperature on the basic of following reactions:

$$Si_{(solid)} + SiO_{2(solid)} \rightarrow 2SiO_{(vapor)} \uparrow$$

$$SiO_{(vapor)} + 2C_{(solid)} \rightarrow SiC_{(solid)} + CO_{(vapor)} \uparrow$$

The $SiO_{(vapor)}$ was generated by heating the mixture of Si and SiO_2 powders. The porous alumina bodies were heat-treated at 1450°C for 9hrs, with a heating rate of 5°C/min in a dynamic gas flow of 200 sccm (ml/min) in a tubular mullite furnace (0.05m dimeter). Formation of SiC whisker or fiber was confirmed by scanning electron microscopy (SEM, JEOL: JMS-840).

Results and discussion

According to volume ratio of PMMA bead and alumina, the sintered samples show the different porosity. Fig. 1 shows microstructures of the calcined alumina bodies. After calcination of green bodies at 1300°C for 9hrs in Ar atmosphere, PMMA beads are converted to carbon particles in pores of alumina bodies. These carbon particles are employed as a carbon source to synthesize SiC nano-fiber or nano-whisker. Also, the quantity of carbon particles is dependent on the addition amounts (volume rate) of PMMA bead.

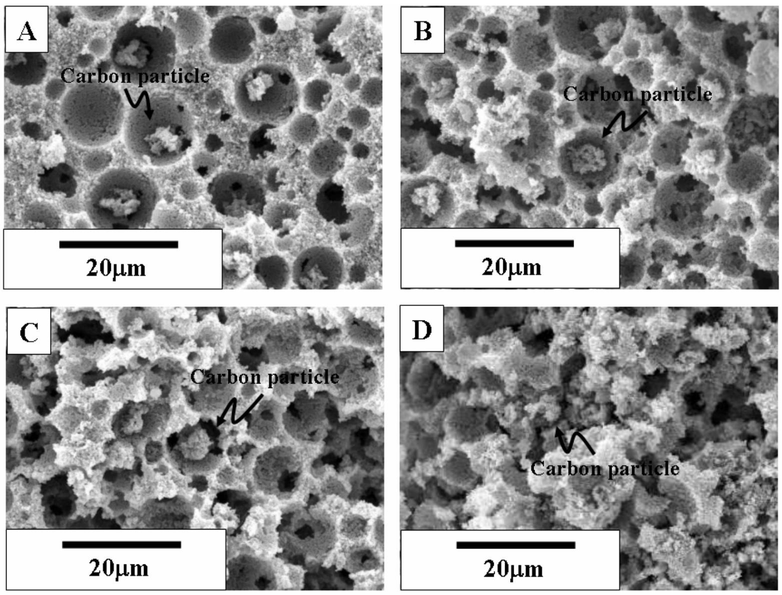

Fig. 1. Micrographs of fractured surfaces of green bodies after calcinations at 1300°C for 9hrs in Ar atmosphere, with different volume ratios in alumina and PMMA bead mixture: (A) 1:1 volume ratio, (B) 1:2 volume ratio, (C) 1:3 volume ratio, and (D) 1:4 volume ratio

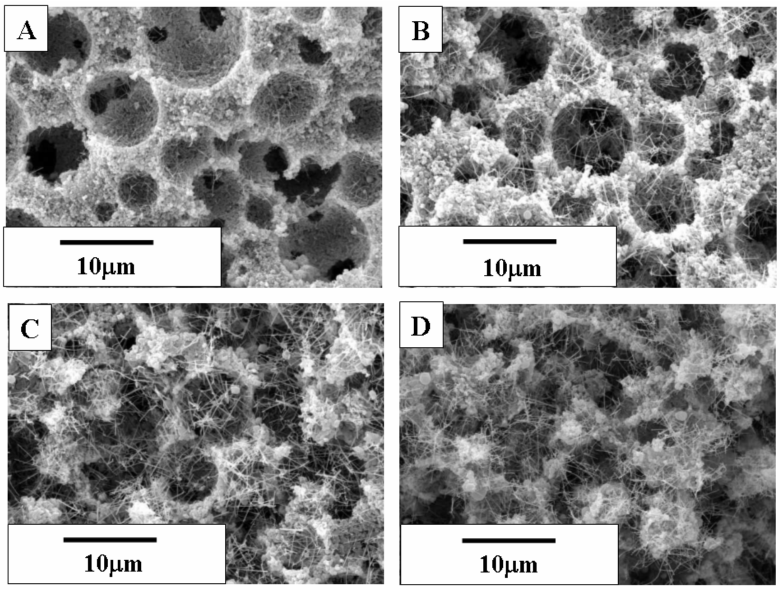

Fig. 2. Micrographs of fractured surfaces of alumina developed SiC nano-fiber or nano-whisker after sintering at 1450°C for 9hrs in Ar atmosphere, with different volume ratios in alumina and PMMA bead mixture: (A) 1:1 volume ratio, (B) 1:2 volume ratio, (C) 1:3 volume ratio, and (D) 1:4 volume ratio

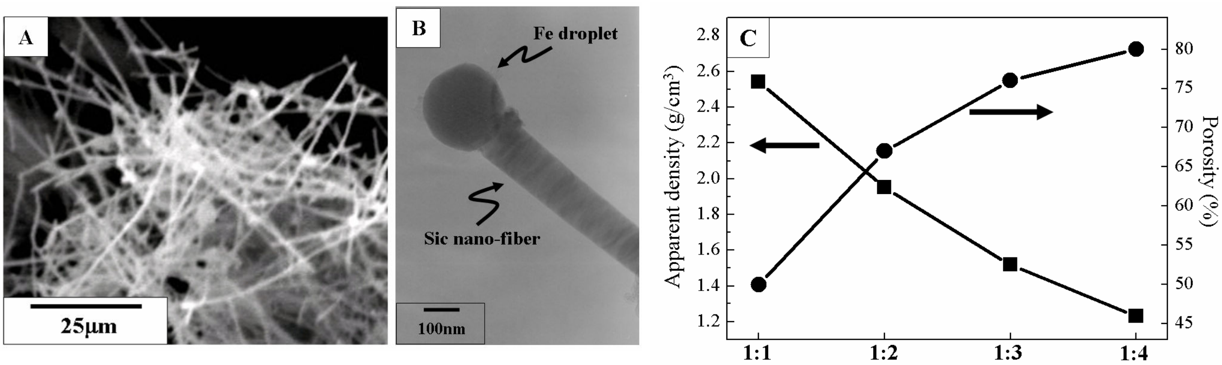

Fig. 3. (A) SEM, (B) HRTEM of SiC nano-fiber or nano-whisker, and (C) apparent density and porosity of porous alumina developed with SiC nano-fiber or nano-whisker synthesized at 1450°C for 9hrs.

Fig. 2 indicates micrographs of the sintered alumina at 1450°C for 9hrs in Ar atmosphere. The synthesized SiC nano-fiber or nano-whisker is produced by the VLS reaction. Reaction of gaseous SiO and carbon particles is more active with an increase of carbon amount converted from PMMA bead by calcination, resulting in increasing the synthesized amount of SiC nano-fiber or nano-whisker. The SiC nano-fiber or nano-whisker grown out of Fe droplet is shown in Figs. 3(A) and 3(B), which thickness is about 100 nm.

The Fe-assisted SiC fiber growth model would be proposed as follows: The mixture of $Si_{(solid)}$, $SiO_{(vapor)}$, and carbon($C_{(solid)}$) has large activation energy at high temperature. The $SiO_{(vapor)}$ molecules contact with the surface of Fe liquid droplets. It decomposes into gaseous Si and oxygen on the surface of Fe liquid droplets. The decomposed Si atoms diffuse and penetrate into the Fe liquid droplets, resulting in the formation of a Fe-Si solution. The successive supply of $SiO_{(vapor)}$ makes it possible to precipitate Si atoms from the Fe-Si solution. The precipitated Si atoms might diffuse into carbon fiber at the liquid-solid interface following the growth of SiC sub-micron sized fiber by the

VLS growth mechanism [6]. The SiC sub-micron sized fiber, the reaction product, begins to grow by tip-growth. Generally, SiC fiber or whisker grown by the VLS reaction has stacking faults [7, 8], which deteriorate the properties of SiC fiber. According to an increase of PMMA bead volume ratio, porosity is increased, but apparent density is decreased as shown in Fig 3(C).

Conclusions

1. Alumina bodies with various porosities are fabricated with different volume ratio in alumina and PMMA bead mixtures, using gel-casting process.
2. The porous bodies developed with SiC nano-fiber or nano-whisker are fabricated by the reaction between gaseous SiO and carbon particles in Ar atmosphere.
3. The SiC nano-fiber or nano-whisker is grown out of Fe liquid droplets. Thickness of SiC nano-fiber or nano-whisker is approximately 100 nm. The amount and thickness of synthesized SiC nano-fiber or nano-whisker are increased and decreased as PMMA bead volume ratio increases, respectively.
4. By the vapor-liquid-solid (VLS) growth mechanism, The SiC nano-fiber or nano-whisker is grown out of Fe liquid droplets.

Acknowledgement

This work was financially supported by MOCIE through EIRC program.

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