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Fabrication and Strength Properties of LPS-SiC based materials

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Abstract. This paper dealt with the LPS process for the development of high performance SiC materials, based on the detailed analysis of their microstructure and mechanical properties. The submicron SiC powder was used for the fabrication of LPS-SiC materials. A mixture of Al_2O_3 and Y_2O_3 particles was also used as a sintering additive in the LPS process. LPS-SiC materials were fabricated at different temperatures, using various additive composition ratio (Al_2O_3/Y_2O_3). The total amount of additive materials ($Al_2O_3+Y_2O_3$) was fixed as 10 wt%. The characterization of LPS-SiC materials was investigated by means of SEM, XRD and three point bending test. The LPS-SiC material represented a relative density of about 98 % and a flexural strength of about 800MPa, when it was fabricated at the temperature of 1820°C and the additive compositional ratio of 1.5.

Introduction

In the last decade, the energy industrial field makes many efforts to develop the advanced energy conversion systems for providing the high efficiency energy under a high purity environment. SiC fiber reinforced SiC matrix composite (SiC_f/SiC) have been extensively studied as a promising candidates for structural materials in fusion power plant systems such as first wall or divertor coolant channel [1]. SiC materials have excellent properties such as high temperature strength, fracture toughness, thermal conductivity and irradiation resistance. The development researches of SiC materials are extensively progressing for high temperature gas turbine and automobile ceramic engine systems [2,3]. Especially, the production of high dense SiC matrix with a high purity is considered as an important issue in the R & D fields for high performance SiC_f/SiC composites, since it can provide a good oxidation resistance and a high thermal conductivity at the elevated temperature. The liquid phase sintering (LPS) process can be recognized as an attractive method for the high density of SiC materials. This process has an advantage for the reduction of the consolidation temperature, if the additive materials can be transformed into some eutectics between SiC particles [4,5]. The mechanical properties of LPS-SiC materials are greatly affected by the preparation conditions such as the type or compositional ratio of additive materials, the size of starting SiC powder and the consolidation temperature. The oxide additive materials including Al_2O_3, Y_2O_3 and SiO_2 and its mixture were also used for the consolidation of LPS-SiC materials. [5-7] However, the production conditions for LPS-SiC materials must be further improved through the optimization of starting powders. Moreover, both the type and the amount of additive materials must be limited for providing the irradiation efficiency of LPS-SiC materials in the application area of fusion power plants. The approach of ultra-fine SiC particle for the control of grain size also needs to examine for the development of high performance LPS-SiC materials.

The purpose of present study is to investigate the effect of fabricating temperature and additive composition ratio (Al_2O_3/Y_2O_3) on the mechanical properties of LPS-SiC materials, in conjunction with the detailed analysis of their microstructure. The fracture surface is also observed to explain the characterization of LPS-SiC materials.

2. Experimental procedures

A commercial SiC powder (β-type, Ibiden Corp., Japan) with average size of about 0.3 μm was used for the fabrication of LPS-SiC ceramics. The SiC particles contain SiO_2 (0.39 wt %), C (1.08 wt %), Al (153 ppm), Fe (173 ppm) and Ca (3 ppm). The additive materials were commercial Al_2O_3 and Y_2O_3 particles (High Purity Chemical Corp., Japan), which have an average size of about 2 μm, respectively. The complex mixture containing SiC, Al_2O_3, Y_2O_3 and accetone was prepared by a ball milling process. The blending speed and its holing time of the complex mixture were 160 rpm and 12 hours, respectively. The compositional ratios of additive materials (Al_2O_3/Y_2O_3) in the complex mixture were 0.4, 0.7, 1.5 and 2.3, respectively. The total amount of Al_2O_3 and Y_2O_3 particles in the complex slurry were constant as 10 wt %. LPS-SiC materials were fabricated at the sintering temperatures of 1780 ℃, 1800 ℃ and 1820 ℃, using rectangular compact preforms of dried SiC complex mixture. The sintering temperature for the formation of secondary phase like an yittrium-aluminum-garnet (YAG) between SiC particles was determined from the phase diagram of the Al_2O_3-Y_2O_3 system. The applied pressure and its holding time for the consolidation of LPS-SiC materials were 10 MPa and 2 hours under the vacuum atmosphere, respectively. The dimension of an as-pressed sample was 3×40×40 mm^3.

The bulk densities of LPS-SiC materials were determined by the immersion method, according to Archimedes' principle. The microstructure of LPS-SiC materials was analyzed using SEM and XRD. The surface of LPS-SiC ceramics was etched to observe the microstructure, after the mechanical polishing by diamond powders with an average size of 0.1 μm. The three point bending test was carried out at room temperature, in order to examine the mechanical properties of LPS-SiC materials. The dimension of all samples for three point bending test was 3×4×25 mm^3. The span length and the crosshead speed were 18 mm and 0.5 mm/min, respectively.

3. Results and discussion

Fig. 1 shows the microstructure of LPS-SiC materials fabricated at the temperature of at 1820 ℃, using an additive compositional ratio of 1.5. The results of XRD analysis for LPS-SiC materials fabricated with different composition ratios of additive materials were shown in this figure. LPS-SiC materials represented a dense morphology without sintering defects, even if there were some amount of pores. LPS-SiC materials also showed the same peak such as SiC, Al_2O_3 and YAG, regardless of additive composition ratios. Especially, the secondary oxide phase like YAG was created in the morphology of LPS-SiC materials, due to the chemical reaction of Al_2O_3 and Y_2O_3. It was found that the morphology of LPS-SiC materials was composed of SiC, secondary oxide phases and micropores.

Fig. 2 shows the effect of fabricating temperature on the density and the flexural strength of LPS-SiC materials. LPS-SiC materials were fabricated with the additive compositional ratio of 1.5. The relative density of LPS-SiC materials was determined as a ratio of measured density to the theoretical density, which was calculated by the rule of mixture, considering the density and the volume fraction of starting powders. The flexural strength of LPS-SiC materials greatly increased

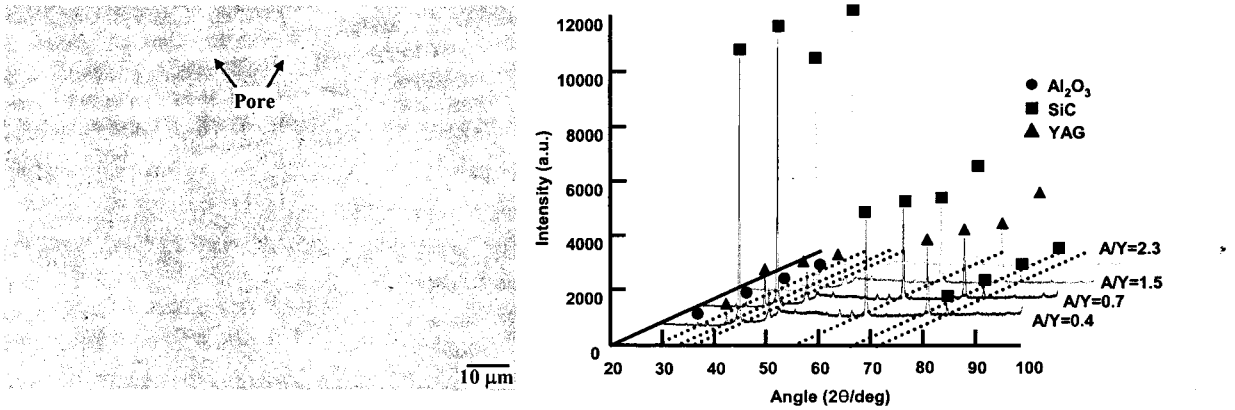

Fig. 1 Microstructure and XRD analysis result of LPS-SiC materials

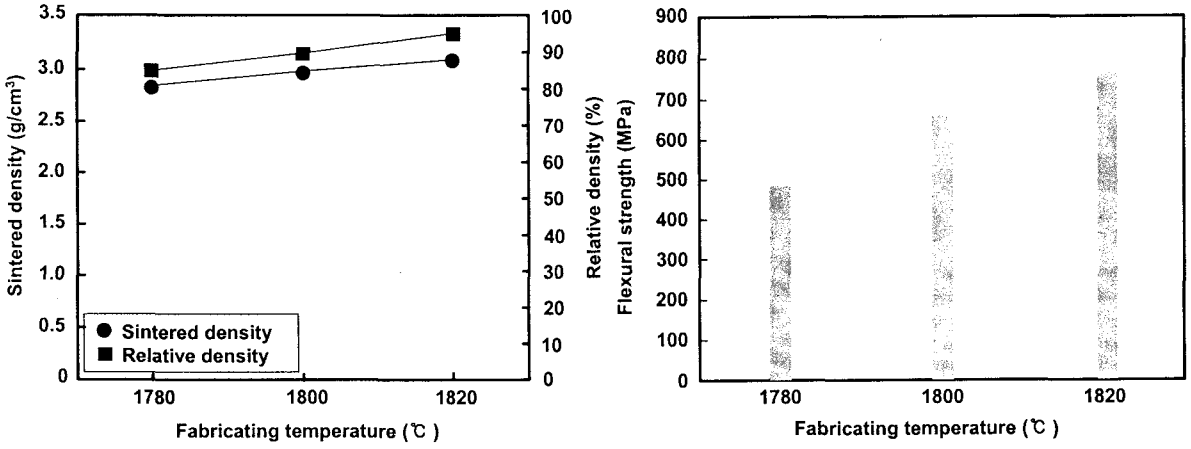

Fig. 2 Effect of fabricating temperature on the characterization of LPS-SiC materials

with increasing the fabricating temperature, accompanying the improvement of sintered density. Especially, LPS-SiC materials possessed an excellent density of about 3.2 g/cm^3, which corresponding to about 98 % of theoretical density, when fabricated at the sintering temperature of 1820 ℃. LPS-SiC materials represented a good flexural strength of about 800 MPa at the fabricating temperature of 1820 ℃. This is maybe because the increase of fabricating temperature accelerates the creation of secondary phases and its connection between the grains of SiC particles for the densification of SiC morphology.

Fig. 3 shows the effect of additive composition ratio on the density and the flexural strength of LPS-SiC materials. LPS-SiC materials were fabricated at the temperature of 1820 ℃. The sintered density of LPS-SiC materials tended to increase with increasing the compositional ratio of additive materials. LPS-SiC materials also maintained the similar density level (about 3.2 g/cm^3) at the additive composition ratio higher than 1.5. This is maybe because the amount increase of Al_2O_3 with a melting point lower than that of Y_2O_3 easily fills pore or vacancy between SiC particles through the sufficient formation of secondary phases. On the contrary, the flexural strength of LPS-SiC materials was affected by the compositional ratio of additive materials. LPS-SiC materials represented an excellent flexural strength (about 800 MPa) at the additive composition ration of 1.5, accompanying the increase of sintered density and the enhancement of grain boundary between SiC particles by the proper formation of secondary phases. However, the flexural strength of LPS-SiC materials greatly decreased at the additive composition ratio of 2.3, even if it had a similar density level to that of the additive composition ration of 1.5. This is related with the grain growth and the dispersion of additive materials in the morphology of LPS-SiC materials. (See Fig. 4)

Fig. 4 shows the fracture surface of LPS-SiC materials containing the additive composition ratios of 1.5 and 2.3. LPS-SiC materials were fabricated at the temperature of 1820 ℃. LPS-SiC materials

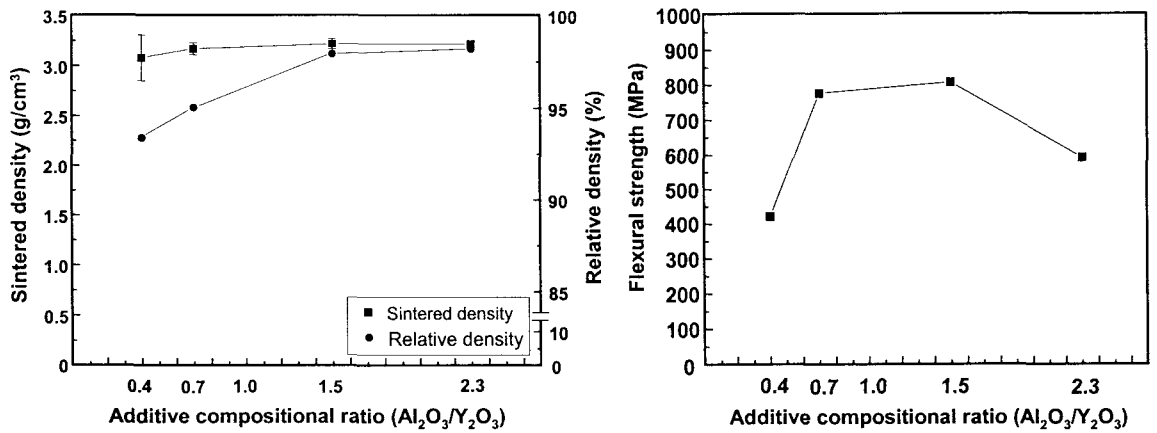

Fig. 3 Effect of additive composition ratio on the characterization of LPS-SiC materials

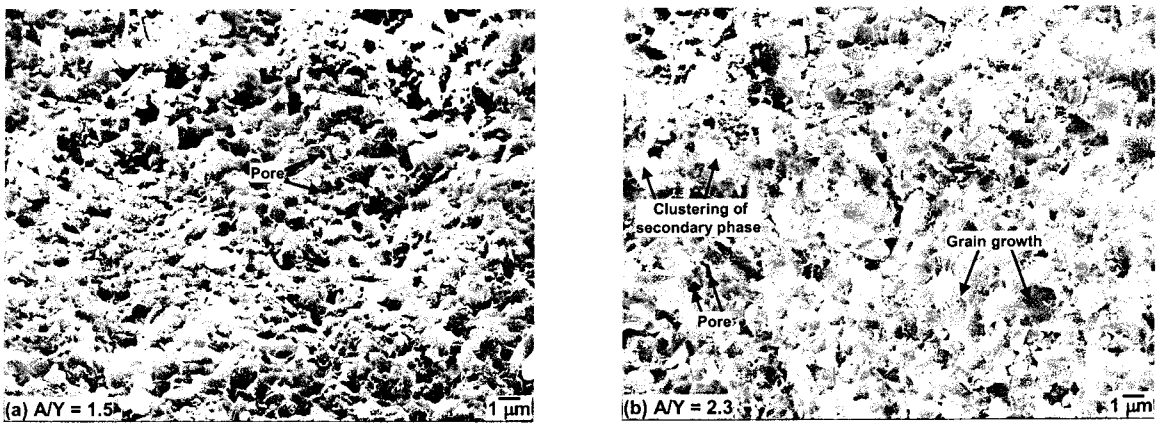

Fig. 4 Fracture surface of LPS-SiC materials

fabricated with the additive composition ratio of 1.5 represented a dense fracture surface with a constant grain size, even if there were small amounts of pores. On the other hand, both the growth of grain size and the sintering defects such as some amounts of pores and clustering of secondary phases were entirely revealed in the fracture surface of LPS-SiC materials containing the additive composition ratio of 2.3. It was found that such a microstuctural defects led to the reduction of flexural strength in high dense LPS-SiC materials.

Summary

The characterization of LPS-SiC materials were greatly affected by the fabricating temperature and the compositional ratio of additive materials (Al_2O_3/Y_2O_3). The density and the flexural strength of LPS-SiC materials largely increased with the increase of fabricating temperature, even if the experiment was conducted at the limited temperature region. The YAG phase by the chemical reaction of additive materials was created in the morphology of LPS-SiC materials, regardless of the variation of additive composition ratio. LPS-SiC materials had an excellent density of about 3.2 g/cm^3 and a good flexural strength of about 800 MPa, accompanying a dense fracture morphology with a constant grain size, when it was fabricated at the temperature of 1820 ℃ and an additive composition ratio of 1.5. However, the extreme composition of additive materials (Al_2O_3/Y_2O_3:2.3) largely decreased the flexural strength of LPS-SiC materials, due to the growth of grain size and the interfacial softening by the clustering of secondary phases.

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