MECHANICAL PROPERTIES OF SUBMICRON SIZED OF SiC CERAMICS

A. Vysocká, J. Špaková, J. Dusza, M. Balog, P. Šajgalík

Abstract

The aim of the present contribution is the study of the microstructure of liquid phase sintered SiC and SiC+Si_3N_4 composites. The basic mechanical properties, namely macro/micro hardness, indentation fracture toughness, Young's modulus, and fracture strength at room temperature of the experimental materials were investigated. The influence of heat-treatment on microstructure and mechanical properties of SiC materials is compared.

The microstructure of SiC+Si_3N_4 composite (hot pressed) consisted of fine equiaxed grains of SiC, Si_3N_4 and a grain boundary intergranular glassy phase. For material annealed at 1650°C, the microstructure was fine with equiaxed grains with average size of SiC grain of 450 nm, as well. The microstructure was changed after post-sintering high temperature treatment at 1850°C/5 hours. It consisted of elongated grains with average size of SiC of 2.25 μm. It is obvious from measurements that a heat treatment performed at higher temperatures and/or longer times had a positive effect on the microhardness and fracture toughness values. With an increasing amount of Si_3N_4 in SiC/Si_3N_4 composite the fracture strength was increased, but with a rising annealing temperature from 1650°C to 1850°C characteristic strength was decreasing.

Keywords: silicon carbide, SiC+Si_3N_4 composite, heat treatment, micorstructure, mechanical properties

INTRODUCTION

Advanced silicon carbide ceramics are leading candidate materials for a wide variety of applications in the aeronautics, energy, electronics, nuclear and transportation industries [1]. Silicon carbide has been recognized as an important structural ceramic because of its unique combination of properties such as high-temperature strength, resistance to wear and corrosion, and thermal shock resistance [2]. These properties depend strictly on density and grain size, and because of the difficulty to obtain SiC parts with high density and small grain size, in recent years methods to sinter silicon carbide with the aim to improve its mechanical properties (fracture strength and toughness) were studied [3]. Silicon carbide is difficult to densify without sintering additives because of the covalent nature of the Si-C bond and the low self-diffusion coefficient. For mechanically demanding structural applications, favourable properties may be accepted especially in liquid-phase sintered materials (LPS-SiC) because, compared to conventionally sintered SiC (SSiC), very homogeneous and fine-grained microstructures can be obtained due to lower sintering

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temperatures and to the presence of the liquid phase [4]. For LPS-SiC various sintering additives have been reported such as Al_2O_3, Al_2O_3-Y_2O_3, yttrium-aluminum garnet (YAG), AlN-Y_2O_3 [5].

The most common method of adjusting the microstructures and mechanical properties of LPS-SiC materials is to vary the α-SiC/β-SiC ratio of the starting powders. The microstructure of SiC obtained from an α-SiC strating powder, is composed of fine equiaxed grains and is usually brittle. SiC obtained from a β-SiC powder consists of coarse, elongated grains, which are the result of the grain growth that accompanies the β-SiC to α-SiC phase transformation during fabrication. Larger elongated grains increase the fracture toughness of SiC by crack bridging or crack deflection. However, phase transformation and resultant microstructure are strongly dependet on the sintering and annealing conditions [6].

MATERIAL PREPARATION AND METHODS

β-SiC powder (HSC-059, Superior Graphite) was mixed with Si_3N_4 (AlY-3/54, Grade C, Plasma & Ceramic Technologies Ltd.), Al_2O_3 (A 16 SG, Alcoa), and Y_2O_3 (grade C, H.C. Starck). The Si_3N_4 powder contains Y_2O_3 and Al_2O_3 sintering additives in a weight ratio 6:3. The weight ratio of nonoxide matrix to oxide sintering additives SiC (+Si_3N_4) : Y_2O_3+Al_2O_3 was kept constant, 91:9. The weight ratio of particular oxides Y_2O_3:Al_2O_3 was 6:3 for all compositions. The final chemical compositions of the studied samples are listed in Table 1.

Tab.1. Chemical composition of SiC samples.

Samples	Composition [wt. %]			
	SiC	Si_3N_4	Y_2O_3	Al_2O_3
SC-N-0	91	0	6	3
SC-N-5	86.5	5	5.7	2.8
SC-N-10	81.9	10	5.4	2.7

The powder mixtures were ball milled in isopropanol with SiC balls for 24 hours. The suspensions were dried, subsequently sieved through a 25 μm sieve screen in order to avoid hard agglomerates. The samples were hot pressed at 1850°C/1h under mechanical pressure of 30 MPa in N_2 atmosphere. Hot pressed samples were subsequently annealed under various time/temperature conditions given in Table 2.

Tab.2. Heat-treatment regimes of samples.

Sample	Heat-treatment
SC-N-A*	HP: 1870°C/1h + AN: 1650°C/5h
SC-N-B	HP: 1870°C/1h + AN: 1850°C/5h
SC-N-G	HP: 1870°C/1h

* SC-N-A5 is SiC/Si_3N_4 composite with 5% Si_3N_4 annealed at 1650°C/5 hours

The hot-pressed samples for microstructural investigation were prepared by the standard ceramographic procedure of multistep grinding and polishing with subsequent chemical etching in Murakami´s reagent for structural elements revelation. The materials were studied by scanning electron microscopy (SEM) and by transmission electron microscopy (TEM). TEM foil preparation was performed by standard techniques involving grinding, dimpling and ion-beam thinning. The SEM micrographs were quantitatively analyzed by image analysis. The thickness of each grain was determined directly from the

shortest grain dimension, the apparent length of each grain was obtained from the largest dimension.

Macro/micro hardness of SiC+Si_3N_4 composites was investigated by using a Vickers indenter at contact load from 2-100N. The indentation of Young`s modulus were obtained by depth-sensing test at load 1 N. The fracture toughness (K_{IC}) was estimated by measuring lengths of cracks generated by a Vickers indenter. The following equation, proposed by Anstis et al. [7], was used for the calculation:

$$K_{IC} = 0{,}016\left(\frac{E}{H}\right)^{1/2}\left(\frac{P}{c^{3/2}}\right) \quad (1)$$

where K_{IC} – fracture toughness [MPa.$m^{1/2}$]; 0.016 – material-independent constant for Vickers-produced radial cracks [-]; E – Young's modulus [GPa]; H – Vickers hardness [GPa]; P – indentation load [N]; c – half-length of the radial crack [m].

The specimens with dimensions 3x4x45 mm were tested in a four point bending test. They were ground and polished by a 15 μm diamond grinding wheel before testing. The two edges on the tensile surface were rounded with a radius of about 0.15 mm in order to eliminate a failure initiated from an edge of the specimen. The specimens were broken in four point bending test (inner span of 20 mm and an outer span of 40 mm) with a crosshead speed of 0.5 mm/min in ambient air. The characteristic strength and Weibull modulus were computed using two-parameter Weibull distribution.

RESULTS

The microstructures of all the hot pressed and annealed materials are shown in Fig.1 The microstructure of hot pressed ceramic (Fig.1.a) was fine, with equiaxed SiC grains. For material annealed at 1650°C (Fig.1.b), the microstructure consisted of fine equaixed SiC grains with an average size of SiC grain of 450 nm, as well. The microstructure was changed after post-sintering high temperature treatment at 1850°C/5 hours (Fig.1(c)). It consisted of elongated grains with an average size of SiC of 2.25 μm and with an average length of 4.14 μm. The mean grain size of the grains of SiC are shown as a function of heat treatment in Fig.2. The grain shape changed from substantially globular to elongated, and the mean aspect ratio increased from 1.086 (SiC +Si_3N_4 composite annealed at 1650°C) to 4.43 (SiC +Si_3N_4 composite annealed at 1850°C). Heat treatment at higher temperature and a longer time results in the β-α transformation, which is accompanied by grain growth.

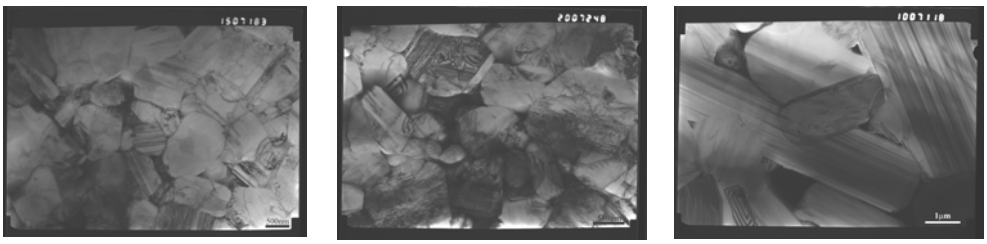

Fig.1. TEM micrographs SiC/Si_3N_4 composite a) hot pressed at 1850°C/1h (SC-N-G5); b) annealed at 1650°C/5h (SC-N-A5); c) annealed at 1850°C/5h (SC-N-B5).

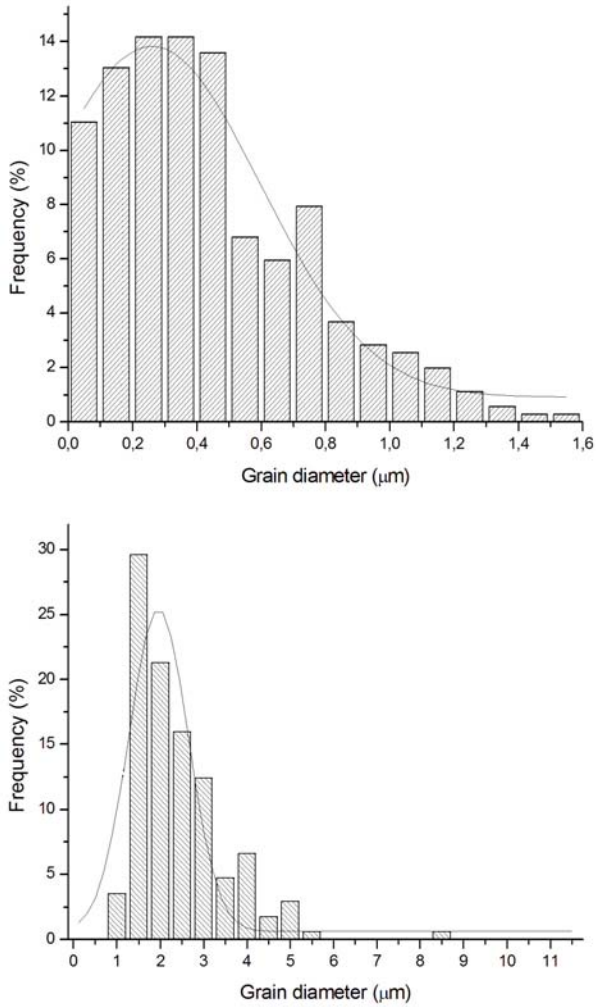

Fig.2. Size distribution of SiC particles of a) annealed at 1650°C/5h; b) annealed at 1850°C/5h.

Microhardness and indentation of Young´s modulus were determined by the depth–sensing indentation method at load 1 N. It was found that the microhardness values of composites, Tab.2, are from 25.82 to 26.87 GPa. Hardness and indentation fracture toughness were estimated at different load (Vickers indentation). Results are reported in Fig.3. From Figure 3a it is possible to see that microhardness shows a slight decrease when indentation load is increased. Fracture toughness resulted from 2.9 to 4.5 MPa.m$^{1/2}$. Results of the indentation toughness measurement, Fig.3b, revealed that the material does not exhibit rising fracture toughness with increasing the indentation load. Fracture toughness increased as the annealing temperature increased (from 1650°C to 1850°C).

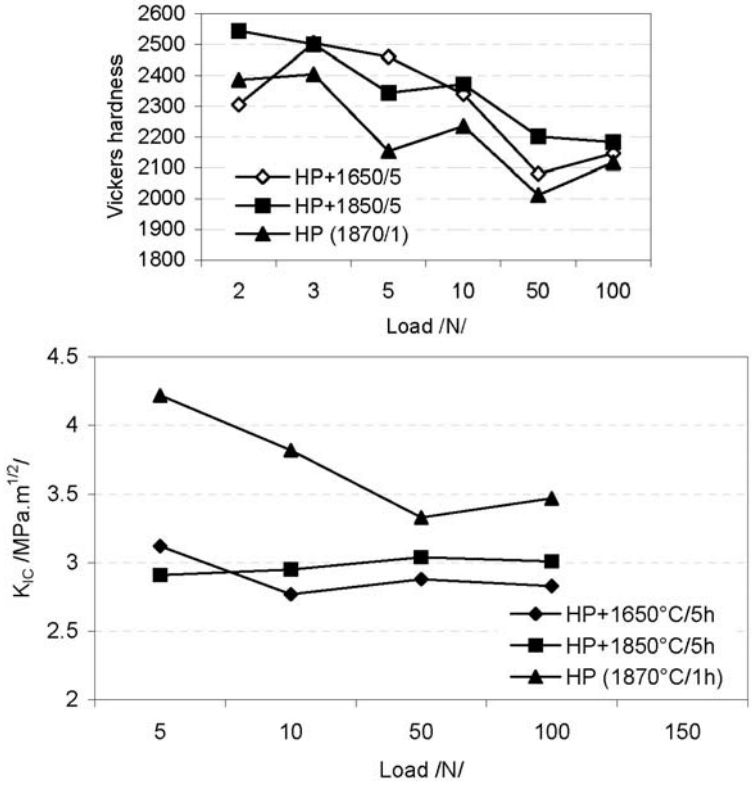

Fig.3. Dependence of Vickers hardness (a) and indentation fracture toughness (b) on applied load.

Tab.3. Microhardness and Young ´s modulus of SiC/Si_3N_4 composites.

Material	Mechanical properties	
	Hv [GPa]*	E [GPa]
SC-N-G5	26.31±0.65	351.20±13.27
SC-N-A5	25.82±0.28	373.30±13.09
SC-N-B5	26.87±1.71	312.75±57.57

The variation of the characteristic strength with a different amount of silicon nitride for hot pressed material (SC-N-G) is reported in Fig.4a. With an increasing amount of Si_3N_4 in SiC/Si_3N_4 composite the characteristic strength was increased from 387 to 584 MPa. In Figure 4b are compared hot pressed and annealed SiC/Si_3N_4 composites with 5% Si_3N_4. The composite SC-N-B5, annealed at 1850°C/5h has the lowest characteristic strength σ_0=386.83 MPa and the highest Weibull modulus m=10.21 when comparing with other composites. However, Weibull´s modulus was low for all experimental materials. The low values of Weibull´s modulus suggest a high scatter of bending strength.

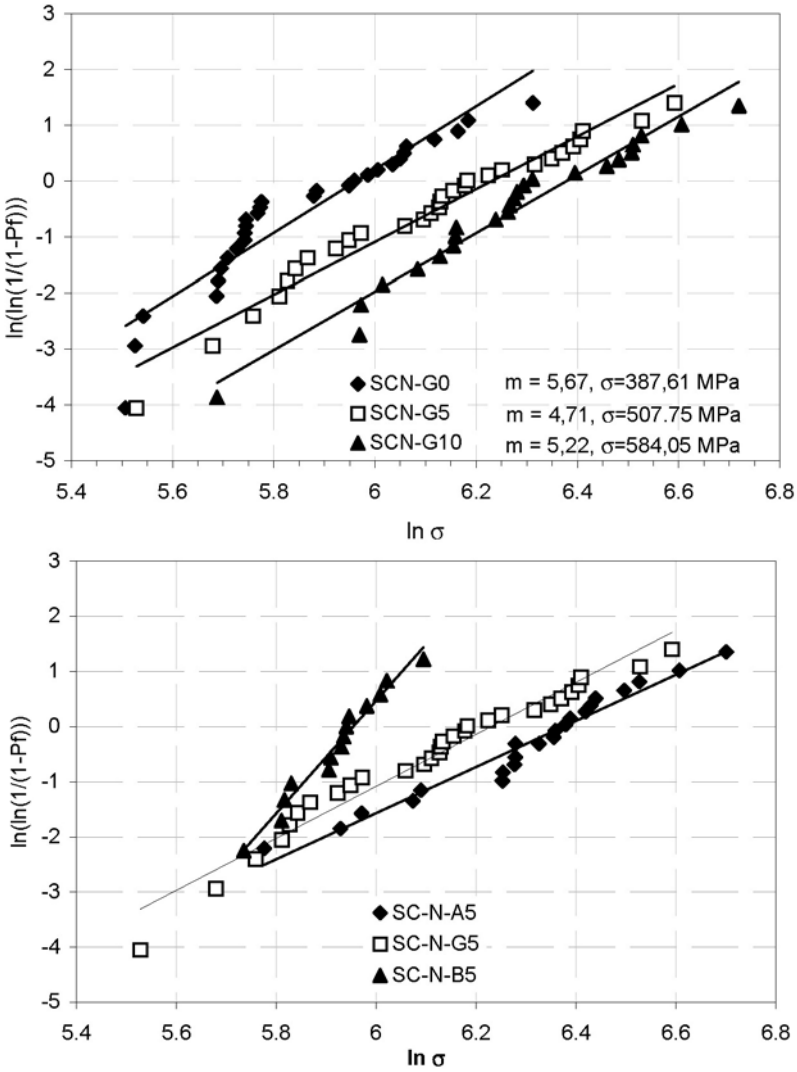

Fig.4. Weibull plot of (a) hot pressed material with different amount of Si_3N_4 ; (b) hot pressed and annealed SiC/Si_3N_4 composites with 5% Si_3N_4.

CONCLUSION

In all SiC materials, and also in $SiC+Si_3N_4$ composites, a load size effect was found during the testing of their hardness. Microhardness of SiC/Si_3N_4 material is in interval from 25.82 to 26.87 GPa. It is obvious from these measurements that a heat treatment performed at higher temperatures and/or longer times had a positive effect on the microhardness and fracture toughness values. However, the indentation fracture toughness is low (2.9-4.5 $MPa.m^{1/2}$). With an increasing amount of Si_3N_4 in SiC/Si_3N_4, composite the bending strength increased from 387 to 584 MPa. With rising annealing temperature the values of characteristic strength decreased.

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