

## Microstructure and Selected Properties of $\text{Si}_3\text{N}_4$ + SiC Composite

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The effects of strengthening phase in particulate ceramic composites on their properties were studied in presented paper. The experimental materials were a monolithic  $\text{Si}_3\text{N}_4$  and particulate ceramic composites consisting of  $\text{Si}_3\text{N}_4$  matrix with different additions of the SiC strengthening phase (10 and 20 vol.%). The microstructure, density, hardness and fracture toughness of  $\text{Si}_3\text{N}_4$  + SiC ceramic composite materials were compared with monolithic  $\text{Si}_3\text{N}_4$  based ceramic material. The addition of SiC particles into the  $\text{Si}_3\text{N}_4$  based matrix does not positively influence the phase transformation from  $\alpha$ - $\text{Si}_3\text{N}_4$  to  $\beta$ - $\text{Si}_3\text{N}_4$  in  $\text{Si}_3\text{N}_4$  + SiC ceramic composite materials, but it affects the growth of prismatic  $\beta$ - $\text{Si}_3\text{N}_4$  grains and contributes to the creation of fine-grained microstructure. The increase of SiC strengthening phase portion slightly increases relative density of  $\text{Si}_3\text{N}_4$  + SiC ceramic composite materials. The hardness of ceramic materials increased from 14.48 GPa at monolithic  $\text{Si}_3\text{N}_4$  ceramics to 16.99 GPa at ceramic composite with 20 vol.% SiC. The highest fracture toughness value of  $8.30 \text{ MPa}\cdot\text{m}^{1/2}$  was achieved for monolithic  $\text{Si}_3\text{N}_4$  ceramics, the lowest value of  $7.09 \text{ MPa}\cdot\text{m}^{1/2}$  was achieved for ceramic composite with 20 vol.% SiC.

**Keywords:** silicon nitride, strengthening phase, microstructure, mechanical properties

### 1 Introduction

Silicon nitride ( $\text{Si}_3\text{N}_4$ ) ceramics has been one of the most studied ceramic materials. It has been often used for demanding application because of its excellent strength, extraordinary wear, corrosion and creep resistance [1,2,3]. Because of these properties,  $\text{Si}_3\text{N}_4$  ceramics is utilised for high temperature applications and friction systems such as combustion engines, gas turbines and rolling bearings working in condition with abrasive, adhesive, erosive and corrosive wear [4-8].

Generally, ceramic materials are fragile materials with low reliability. The fragility is caused by their predominant covalent or ionic chemical bond. High attention has been focused on understanding of initiation and propagation of cracks in ceramic materials. The processes leading to the creation of so-called procedural zone in front of crack have been studied, because they could eventually increase the fracture toughness of ceramics.

Addition of secondary phases into ceramic matrix can be useful for enhancing of toughness of ceramic materials. The secondary phases can be added in form of ceramic micro and nano particles (SiC,  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ , TiN, TiC), whiskers, or fibres [9-12]. Invention of carbon nanomaterials (nanofibers, nanotubes, graphenes) caused progress in composite materials research because of utilising of their progressive properties [13-15].

Recently, properties of composite material type  $\text{Si}_3\text{N}_4$  + graphene nanoplatelets has been studied in

several works [16-19].  $\text{Si}_3\text{N}_4$  + SiC based ceramic composite materials have been studied in many research works. Physical, mechanical, and tribological properties of this type of composite have been evaluated. The effects of chemical composition of the composite, used processing method, process parameters and morphology of secondary phase on these properties have been studied [20-23]. The authors of work [23] evaluated processing method and properties of  $\text{Si}_3\text{N}_4$  + SiC composite which was densified using mullite. Prepared ceramic composite is a perspective material for application in solar cells because of favourable properties such as high thermal conductivity, good thermal shock resistance and oxidation resistance. The oxidation resistance of  $\text{Si}_3\text{N}_4$  + SiC composite was compared to monolithic  $\text{Si}_3\text{N}_4$  ceramics during 100 hours of exposition at a temperature of  $1300^\circ\text{C}$ . Sufficiently thick coating which contained mainly  $\text{SiO}_2$  and decelerated the oxidation speed was created on the surface of  $\text{Si}_3\text{N}_4$  + SiC composite during oxidation process of this composite material. Many cracks were observed on the surface of monolithic  $\text{Si}_3\text{N}_4$  ceramics, the surface of  $\text{Si}_3\text{N}_4$  + SiC composite was compact.

The decrease of relative density with increased portion of SiC thus the restriction of densification of composite material with addition of SiC was shown at studying of  $\text{Si}_3\text{N}_4$  based ceramic composites with a different portion of SiC submicron particles and SiC nano particles [21,24-28]. The presence of SiC particles in  $\text{Si}_3\text{N}_4$  matrix affects the portions of  $\alpha$ - $\text{Si}_3\text{N}_4$  and  $\beta$ - $\text{Si}_3\text{N}_4$  phase in the final microstructure. The refinement of microstructure with dominantly equiaxed  $\alpha$ -

Si<sub>3</sub>N<sub>4</sub> grains was observed too. These results show, that the presence of SiC strengthening phase in Si<sub>3</sub>N<sub>4</sub> matrix suppresses the transformation of  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> to  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phase and restricts the growth of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains.

Additionally, the effect of SiC nano particles on the fracture toughness and hardness of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite was studied. The addition of 5 and 10 % SiC has a positive effect on the fracture toughness of Si<sub>3</sub>N<sub>4</sub> + SiC composite compared to Si<sub>3</sub>N<sub>4</sub> monolithic ceramics. TEM observations showed that the increase of the fracture toughness was caused by the deflection of crack where SiC particle was the obstacle for the propagated crack. It is supposed that if the particles are adequately small in comparison to the critical length of the crack, their presence in the matrix does not decrease the fracture toughness of composite [21]. Significant decrease of the fracture toughness was measured at 20 % SiC, when the morphology of matrix grains proved its effect. At this portion of SiC, the matrix consists mainly of equiaxed  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> grains which unfavourably affects the fracture toughness. The prolongation of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains has a favourable effect on the fracture toughness. The fracture toughness of Si<sub>3</sub>N<sub>4</sub> + SiC composite is influenced mainly by the morphology of Si<sub>3</sub>N<sub>4</sub> matrix [24,28]. The HV hardness values increased with increased portion of SiC particles. Significant increase was proved at 20 % SiC. This fact could be predicted because the hardness of SiC ceramics is by (2.5-3.5) GPa higher compared to Si<sub>3</sub>N<sub>4</sub> ceramics [21].

As the SiC phase positively affects the properties of Si<sub>3</sub>N<sub>4</sub> based ceramic materials, the effects of SiC strengthening phase addition on microstructure of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composites and their properties such as density, hardness and fracture toughness were studied in the presented work.

## 2 Experiment

The experimental materials were a monolithic Si<sub>3</sub>N<sub>4</sub> and particulate ceramic composites consisting of Si<sub>3</sub>N<sub>4</sub> matrix with different additions of the SiC strengthening phase (10 and 20 vol.%). The experimental materials were prepared by hot pressing in nitrogen atmosphere at the temperature of 1680°C, and the pressure of 34 MPa. Sintering additives Y<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> in concentration that could set creating of 10 vol.% Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> garnet were added to enhance the sintering ability of the ceramics. The mean particle size of the Si<sub>3</sub>N<sub>4</sub> was 0.68  $\mu$ m, Y<sub>2</sub>O<sub>3</sub> was 1.0  $\mu$ m, Al<sub>2</sub>O<sub>3</sub> was

0.5  $\mu$ m, and SiC was below 3  $\mu$ m.

The densities of hot pressed ceramic samples were measured by the Archimedes' method. The hardness and the fracture toughness were determined by means of the Vickers indentation method with the loading force of 10 kg. The fracture toughness of ceramics materials was calculated using the formula (1):

$$K_{IC} = 0.0889 \cdot \sqrt{\frac{HV \cdot F}{4 \cdot l_p}} \quad (1)$$

Where:

HV... Vickers hardness[GPa],

F . . . loading force [N],

$l_p$  . . . average crack length measured on the impression [ $\mu$ m].

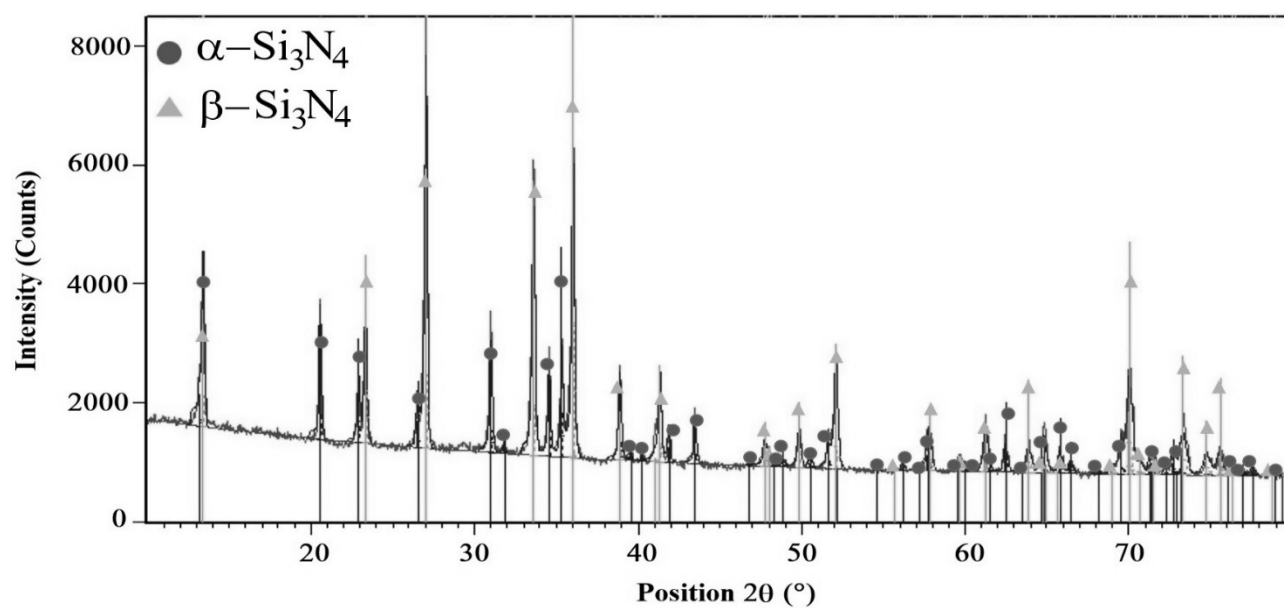
The phase analysis was done using an X-ray diffraction method with Panalytical Empyrean diffractometer with source of characteristic X ray of CuK $\alpha$ . The microstructures of hot pressed ceramics were observed using a scanning electron microscope JEOL IT- 300-LV.

## 3 Results and discussion

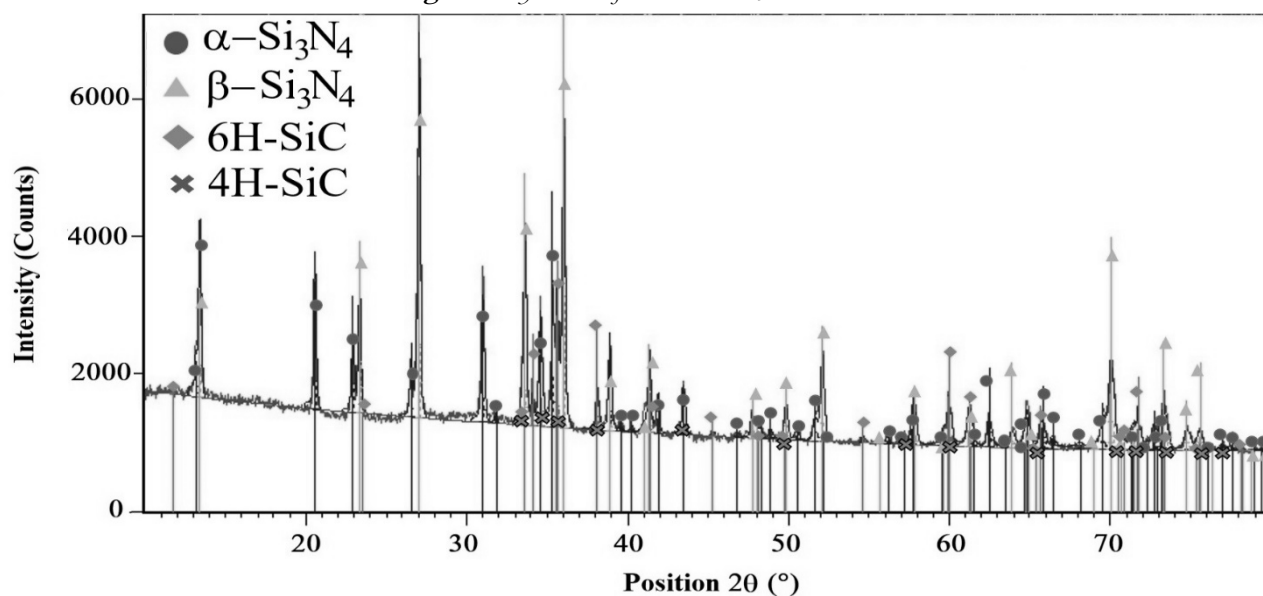
### 3.1 The effect of SiC addition on microstructure of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite materials

The identification of microstructural phases was done by X ray analysis and the records from this analysis are documented from Fig. 1 to 3. The  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> and  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phases were identified in all ceramic samples. The polytypes of  $\alpha$ - SiC 6H and 4H were identified in both Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite materials (Fig. 2 and 3). The effect of SiC addition on phase compositions of experimental materials is depicted in Fig. 4. The ratio of phases 6H-SiC and 4H-SiC was the same in all analysed ceramic materials.

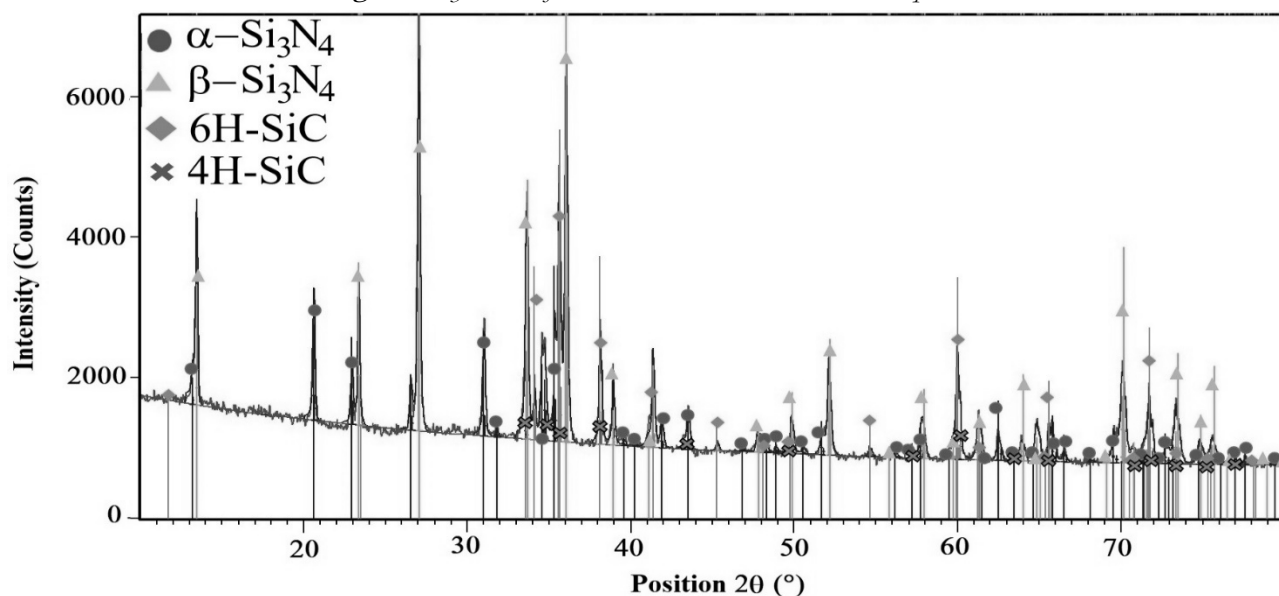
Phase transformation from  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> to  $\beta$ -Si<sub>3</sub>N<sub>4</sub> occurs during sintering process of Si<sub>3</sub>N<sub>4</sub> based ceramic materials [8]. The initial Si<sub>3</sub>N<sub>4</sub> powder consisted of 100 % of  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> phase. Fig. 5 documents the portion of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phase created during hot pressing process by phase transformation from the initial  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> phase. The highest portion of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phase (71.9 vol.%) was measured at adding of 20 % SiC into the initial powder mixture. However, the portion of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phase was 66.5 and 58.1 vol.% for monolithic Si<sub>3</sub>N<sub>4</sub> and ceramic composite with 10 % SiC, respectively. Based on the results presented in Fig. 5, the effect of SiC on the phase transformation from  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> to  $\beta$ -Si<sub>3</sub>N<sub>4</sub> was not positive.



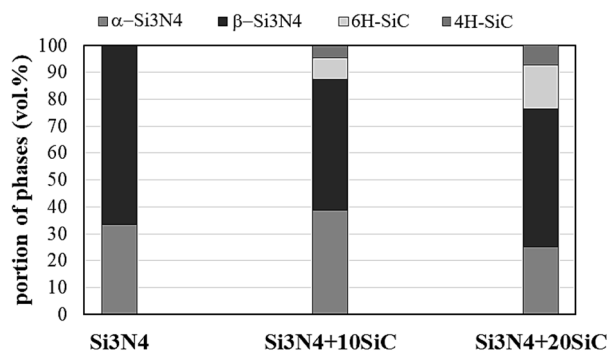
*Fig. 1 X-ray record of monolithic  $\text{Si}_3\text{N}_4$  ceramics*



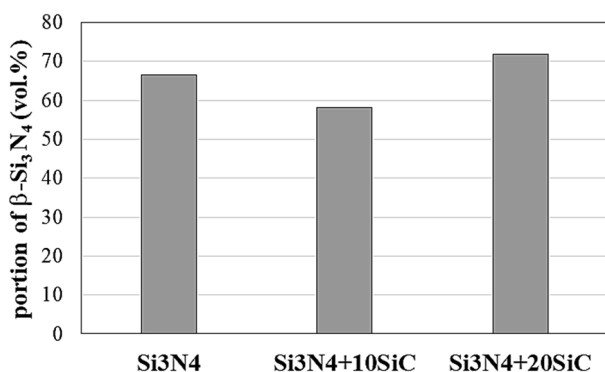
*Fig. 2 X-ray record of  $\text{Si}_3\text{N}_4$  + 10 vol.% SiC ceramic composite*



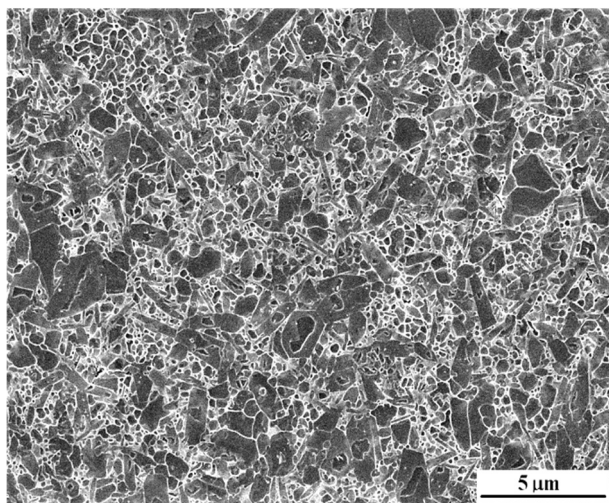
*Fig. 3 X-ray record of  $\text{Si}_3\text{N}_4$  + 20 vol.% SiC ceramic composite*



**Fig. 4** The effect of SiC addition on phase compositions of ceramic materials



**Fig. 5** The effect of SiC addition on  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phase portion in ceramic materials

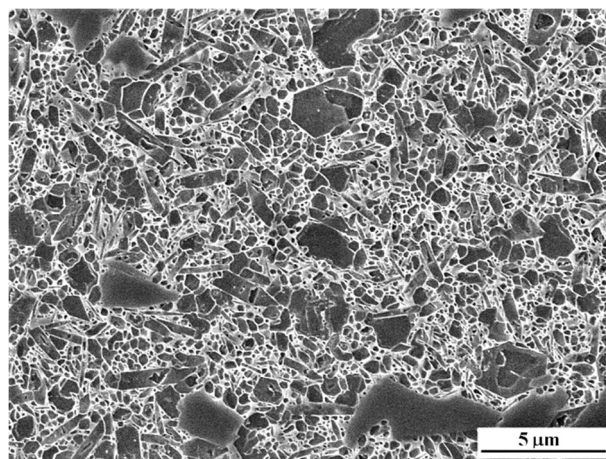


**Fig. 6** Microstructure of monolithic Si<sub>3</sub>N<sub>4</sub> ceramic

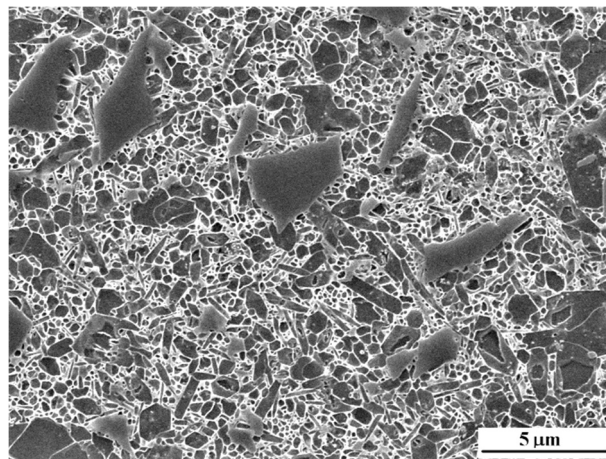
The microstructures of experimental ceramic materials are documented from Fig. 6 to 8. The microstructure of monolithic Si<sub>3</sub>N<sub>4</sub> in Fig. 6 consists of equiaxed  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> grains, prismatic  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains, and binding phase. Ceramic composite materials Si<sub>3</sub>N<sub>4</sub> + SiC types in Fig. 7 and 8 consist of matrix created by the same phases equiaxed  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> grains, prismatic  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains, and binding phase. In addition to that, SiC particles are irregularly distributed in this matrix. Prismatic  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains were fine-grained by adding of SiC into the Si<sub>3</sub>N<sub>4</sub> based matrix which can be seen in Fig. 7 and 8. Fine-grained microstructure of ceramic

composites with SiC particles could be the consequence of movement hindering of Si<sub>3</sub>N<sub>4</sub> grain boundaries by SiC particles or SiC clusters. The sample with more SiC content gave a relatively smaller Si<sub>3</sub>N<sub>4</sub> grain size because SiC particles limit the Si<sub>3</sub>N<sub>4</sub> grain growth by pinning and prohibiting the grain boundary movement.

Although addition of SiC particles into the Si<sub>3</sub>N<sub>4</sub> based matrix does not influence the phase transformation from  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> to  $\beta$ -Si<sub>3</sub>N<sub>4</sub>, it affects the growth of prismatic  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains and contributes to the creation of fine-grained microstructure.



**Fig. 7** Microstructure of Si<sub>3</sub>N<sub>4</sub> + 10 vol.% SiC ceramic composite

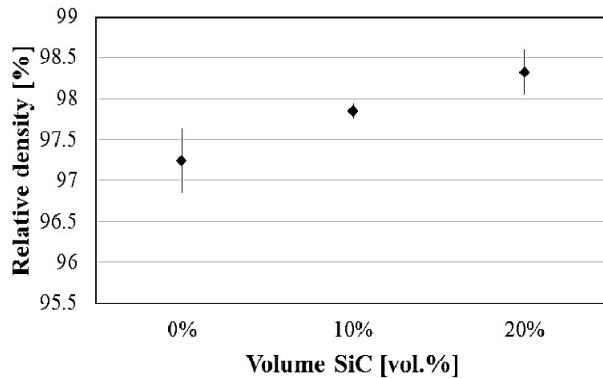


**Fig. 8** Microstructure of Si<sub>3</sub>N<sub>4</sub> + 20 vol.% SiC ceramic composite

### 3.2 The effect of SiC addition on density of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite materials

The effect of SiC on the relative density of ceramic materials is documented in Fig. 9. The maximal value of relative density 98.32 % was achieved at ceramic composite with addition of 20 vol.% SiC. The relative density was 97.85 % for ceramic composite with 10 vol.% SiC. Slight increase of relative density of ceramic materials with increase of SiC is evident from Fig. 9. The relative density of ceramic composite with 20 vol.% SiC is by 1.11 % higher compared to monolithic Si<sub>3</sub>N<sub>4</sub>

ceramics. The densities of samples are influenced mainly by their porosity which is affected primarily by sintering method and process parameters. Morphology and portion of secondary SiC phase could improve the densification of ceramic composite materials.



**Fig. 9** The effect of SiC addition on relative density of ceramic materials

### 3.3 The effect of SiC addition on hardness of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite materials

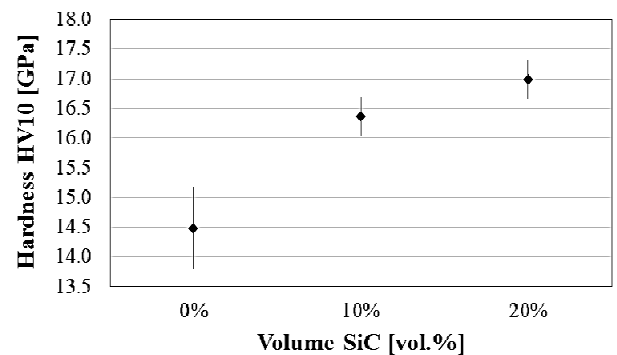
The effect of SiC additive on hardness of ceramic experimental materials is documented in Fig. 10. The highest hardness of 16.99 GPa was achieved at ceramic composite with 20 vol.% SiC. The hardness decreased with decreasing of SiC portion with the lowest value of 14.48 GPa measured for monolithic Si<sub>3</sub>N<sub>4</sub> ceramics which gives the hardness difference of 2.51 GPa compared to ceramic composite with 20 vol.% SiC. The ceramic composite with 10 vol.% SiC had the hardness of 14.77 GPa. The increase of hardness with increased SiC addition can be related to the higher hardness of SiC compared to Si<sub>3</sub>N<sub>4</sub>. The highest density measured for samples with the highest portion of SiC could positively influence the hardness values.

### 3.4 The effect of SiC addition on fracture toughness of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite materials

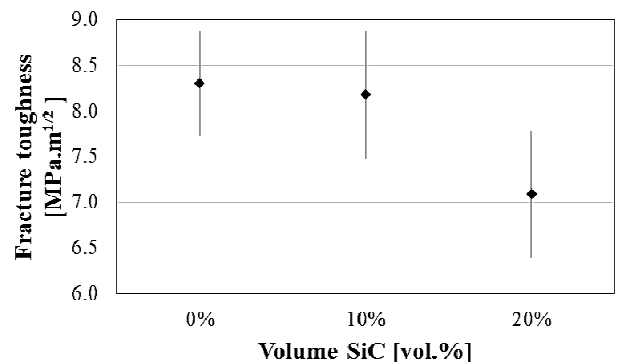
The effect of SiC addition on the fracture toughness of ceramic experimental materials is in Fig. 11. Palmqvist cracks were initiated in all samples at measuring of fracture toughness using Vickers indenter. The highest fracture toughness value of 8.30 MPa.m<sup>1/2</sup> was achieved for monolithic Si<sub>3</sub>N<sub>4</sub> ceramics. The fracture toughness was 8.18 MPa.m<sup>1/2</sup> for ceramic composite with 10 vol.% SiC. As the lowest fracture toughness value of 7.09 MPa.m<sup>1/2</sup> was achieved for ceramic composite with 20 vol.% SiC, the maximal measured difference between the monolithic Si<sub>3</sub>N<sub>4</sub> ceramics and the ceramic composite with 20 vol.% SiC was 1.21 MPa.m<sup>1/2</sup>. Decrease of fracture toughness with increased portion of SiC could be caused the morphology of the matrix [8,25,26]. Prismatic β-Si<sub>3</sub>N<sub>4</sub> grains, which increase the fracture toughness, could not reach the prolonged morphology, because SiC

particles limited the growth of Si<sub>3</sub>N<sub>4</sub> grains. The microstructure of monolithic Si<sub>3</sub>N<sub>4</sub> ceramics consists mainly of prolonged prismatic β-Si<sub>3</sub>N<sub>4</sub> grains with optimal grain prolongation and this is the reason for the highest fracture toughness of the monolithic Si<sub>3</sub>N<sub>4</sub> ceramic material.

Several effects such as morphology of Si<sub>3</sub>N<sub>4</sub> matrix, grain size of SiC particles and difference in thermal coefficient values of Si<sub>3</sub>N<sub>4</sub> and SiC could decrease the fracture toughness of studied ceramic composite materials. Because of these facts, residual stresses could be created at the interfaces between Si<sub>3</sub>N<sub>4</sub> matrix and SiC particles. These stresses will support propagation of cracks in composite materials and decrease their fracture toughness [25-27]. Presented experimental results showed that the addition of SiC particles into Si<sub>3</sub>N<sub>4</sub> matrix do not have a positive effect on the fracture toughness.



**Fig. 10** Effect of SiC addition on hardness of Si<sub>3</sub>N<sub>4</sub> + SiC composites



**Fig. 11** The effect of SiC addition on fracture toughness of Si<sub>3</sub>N<sub>4</sub> + SiC composites

## 4 Conclusions

The effects of SiC strengthening phase in particulate ceramic composites consisting of Si<sub>3</sub>N<sub>4</sub> matrix with different additions of the SiC on their microstructures and selected properties were studied in presented paper. The microstructure, density, hardness and fracture toughness of Si<sub>3</sub>N<sub>4</sub> + SiC ceramic composite materials were compared with monolithic Si<sub>3</sub>N<sub>4</sub> based ceramic material. From achieved results followed conclusions were derived:

- The addition of SiC particles into the  $\text{Si}_3\text{N}_4$  based matrix, does not positively influence the phase transformation from  $\alpha\text{-Si}_3\text{N}_4$  to  $\beta\text{-Si}_3\text{N}_4$  in  $\text{Si}_3\text{N}_4 + \text{SiC}$  ceramic composite materials, but it affects the growth of prismatic  $\beta\text{-Si}_3\text{N}_4$  grains and contributes to the creation of fine-grained microstructure.
- The increase of SiC strengthening phase portion slightly increases relative density of  $\text{Si}_3\text{N}_4 + \text{SiC}$  ceramic composite materials.
- The SiC additive has a positive effect on the hardness of  $\text{Si}_3\text{N}_4 + \text{SiC}$  ceramic composite materials. The hardness of ceramic materials increased from 14.48 GPa at monolithic  $\text{Si}_3\text{N}_4$  ceramics to 16.99 GPa at ceramic composite with 20 vol.% SiC. The increase of the hardness with increased SiC portion can be related to the higher hardness of SiC compared to  $\text{Si}_3\text{N}_4$  ceramics.
- The increase of SiC strengthening phase portion does not have a positive effect on the fracture toughness of  $\text{Si}_3\text{N}_4 + \text{SiC}$  ceramic composite materials. The highest fracture toughness value of  $8.30 \text{ MPa}\cdot\text{m}^{1/2}$  was achieved for monolithic  $\text{Si}_3\text{N}_4$  ceramics, the lowest value of  $7.09 \text{ MPa}\cdot\text{m}^{1/2}$  was achieved for ceramic composite with 20 vol.% SiC. Decrease of fracture toughness with increased portion of SiC could be caused by limited growth of prismatic  $\beta\text{-Si}_3\text{N}_4$  grains and by the size of SiC particles.

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