Effect of Mechanical Properties on Wear Resistance of Si_3N_4 – SiC Ceramic Composite

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ABSTRACT

The effect of mechanical properties on wear resistance of Si_3N_4 – SiC composite materials with different portions of SiC strengthening phase was investigated. Properties of monolithic silicon nitride were compared to ceramic composites consisting of Si_3N_4 matrix with 10 and 20 vol.% SiC. The SiC strengthening phase had a positive effect on the hardness of Si_3N_4 – SiC ceramic composite materials. Wear resistance of tested ceramic materials was mainly influenced by their fracture toughness. The highest wear resistance value was achieved for material with the highest fracture toughness. Worn surfaces of all experimental ceramic materials were damaged by both microcutting and microcracking mechanism. Microcracking was the predominant wear mechanism mainly at ceramic composites. The wear resistance of SiC-Si_3N_4 ceramic composites can be described by the model $W \sim HV/K_{IC}$.

Keywords: silicon nitride, strengthening phase, microstructure, mechanical properties, wear resistance

INTRODUCTION

Mechanical components with reciprocal motion are essential parts of many industrial applications. These components are often exposed to the impact of hard abrasive particles which is the consequence of their surface damage or leak of these components. Intensive wear concentrated in relatively small areas of mechanical components can lead to the destruction of whole apparatus. In apparatuses working in such demanding conditions, hard engineering ceramics have been applied [1–5].

Silicon nitride (Si_3N_4) is an important representative of promising engineering ceramic materials. Si_3N_4 based ceramics are characterized by high strength, high hardness, excellent wear resistance and corrosion resistance [6–9]. However, silicon nitride and ceramic materials in general have low fracture toughness compared to metallic materials. Therefore, engineering ceramic research is concentrated on enhancing facture toughness values in order to increase the reliability of ceramic components [10, 11].

Wear mechanisms are important at evaluation of wear of silicon nitride in friction couple either ceramic-ceramic or ceramic-metal. Some theoretical models were proposed [12,13] and subsequently specified and verified experimental [14–16]. Abrasive wear is the most common wear of Si_3N_4 based ceramic materials. Surface damage at this wear can be described be two basic mechanisms – microcutting and microcracking [5,12,17]. Microcutting can be expected as dominant wear mechanism at good fracture toughness and high hardness of worn ceramics but which is lower compared to abrasive material. Microcraking is dominant wear mechanism for material with low fracture toughness. This will lead to a decrease of wear resistance of material still at high hardness. The transfer from microcutting to microcracking can lead to a decrease of wear resistance because of high hardness of ceramic materials. Inclination to microcracking depends mainly on the fracture toughness of worn ceramic material and load conditions.

When abrasive wear is a predominant wear mechanism, parameter ratio K_{IC}/HV can be used for prediction of wear of fragile materials [18, 19]. Based on the value of the K_{IC}/ HV ratio, we can define the predominant wear mechanism in the contact. At high value of this ratio, plastic micro-cutting mechanism predominates, and hardness of worn material has a supreme effect on wear which can be described by the relation W~HV^{-1}. At low value of this ratio, microcracking mechanism predominates and wear extent can be described by the relation W~HV/ K_{IC}.

Research in the field of ceramic materials has shown that addition of suitable secondary phase into silicon nitride matrix can significantly influence physical, mechanical and tribological properties of created ceramic composite materials. The secondary phases can be added in form of ceramic micro and nano particles (SiC, Al_2O_3, ZrO_2, TiN, TiC, hBN…), whiskers, or fibres [6,20–22]. Invention of carbon nanomaterials (nanofibers, nanotubes, graphenes) caused progress in composite materials research because of utilising of their progressive properties [23,24]. The addition of secondary phase can induce favorable stress conditions in matrix of ceramic composite [25–27]. Compressive stress can be induced in the matrix because of different thermal coefficients of matrix and secondary phase and this strain can influence the final ceramic composite properties [28, 29].

Si_3N_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 [10,30–34]. The results of the works show that the mechanical properties depend on the microstructure of ceramic material. The addition of 5 and 10 vol.% SiC has a positive effect on the fracture toughness of Si_3N_4 – SiC composite compared to Si_3N_4 monolithic ceramics.

The increase of the fracture toughness was caused by the deflection of crack where SiC particles were the obstacles for the propagated cracks. It is supposed that if the particles are adequately small in comparison to the critical length of the cracks, their presence in the matrix does not decrease the fracture toughness of composite [6,31]. For composite samples with addition of 20 vol.%

SiC, the decrease of fracture toughness was observed, and this decrease is the consequence of the matrix morphology on the fracture toughness. Prolongation of β-Si_3N_4 grains has a positive effect on the fracture toughness of silicon nitride. However, the Si_3N_4 matrix with 20 vol.% SiC was predominantly built by equiaxed β-Si_3N_4 grains which negatively influenced the fracture toughness values.

The HV hardness values increased with increased portion of SiC particles. Significant increase was proved at 20 vol.% SiC. This fact could be predicted because the hardness of SiC ceramics is by (2.5–3.5) GPa higher compared to Si_3N_4 monolithic ceramics [31].

Two monolithic ceramics SiC and Si_3N_4 and two ceramic composites SiC-10%TiB$_2$ and Si_3N_4–10%SiC were studied in work [25]. The work was concentrated on the effect of residual stresses induced in composite matrix on mechanical properties and wear of ceramic composites. Compressive residual stresses in the Si_3N_4 matrix were calculated in both types of composite materials. The compressive residual stresses value was by 500% higher for SiC-10%TiB$_2$ composite compared to Si_3N_4–10%SiC composite material. This led to an increase of bending strength and fracture toughness and to the assumption of positive effect on wear resistance. The wear resistance was evaluated using Miller test. The experimental results showed that monolithic Si_3N_4 had higher wear resistance by 32% than monolithic SiC. No difference in wear resistance was measured between monolithic SiC ceramics and SiC-10%TiB$_2$ ceramic composite, although the highest compressive residual stresses value was calculated for this type of composite material. The highest wear resistance was measured for Si_3N_4–10%SiC TiB$_2$ ceramic composite although the lowest compressive residual stresses value was calculated for this composite. The wear resistance did not correlate with mechanical properties. Ceramic materials with the highest hardness (SiC, SiC-10%TiB$_2$) and highest fracture toughness (SiC-10%TiB$_2$) achieved the lowest wear resistance.

From research works it arises, that the effect of mechanical properties of Si_3N_4 – SiC composite on its wear resistance has been not investigated sufficiently so far. In some works, increased wear resistance was observed with increasing of K_{IC}/HV ratio [35, 36], but in another works the wear resistance do not correlate with mechanical properties [25, 37]. Therefore, this paper is

concentrated on the effect of mechanical properties on wear resistance of Si_3N_4 – SiC composite material with different portion of SiC strengthening phase.

EXPERIMENTAL MATERIALS AND METHODS

The experimental material was a monolithic silicon nitride and a particulate ceramic composite with a Si_3N_4 matrix with 10 and 20 vol.% of the SiC phase strengthening. In the paper they are marked SN, SN10SC and SN20SC, respectively. The mean particle size of the SiC was 3 µm and Si_3N_4 was 0.68 µm. The morphologies of used powders are documented in Figures 1 and 2. The Si_3N_4 in Figure 1 is spherical with small flakes and agglomerates, whereas morphology the SiC is irregular (Fig. 2).

The experimental materials were prepared by hot pressing in nitrogen atmosphere. The hot pressing of the experimental materials was performed on a laboratory hot press with the temperature of 1680°C, and pressure of 35 MPa. Sintering additives Y_2O_3 and Al_2O_3 in concentration that could set creating 10 vol.% $Y_3Al_5O_{12}$ garnet (YAG) were added for improving densification of our ceramic samples. This phase contributed to the sintering ability of the ceramics. The mean particle size of the Y_2O_3 was 1.0 µm and Al_2O_3 was 0.5 µm.

Densities of the hot pressed ceramics were measured by the Archimedes' method. The hardness and fracture toughness were measured by the indentation method using a Vickers indenter with load of 10 kp (HV10) and indentation time of 15 s. The fracture toughness of ceramics materials were calculated using the formula (1) [38]:

$$K_{IC} = 0.0889. \sqrt{(HV.F)/(4.l_p)}, \left[MPa.m^{1/2}\right] \quad (1)$$

where: HV is the hardness,
  F is the loading force and
  l_p is the average crack length measured on the impression.
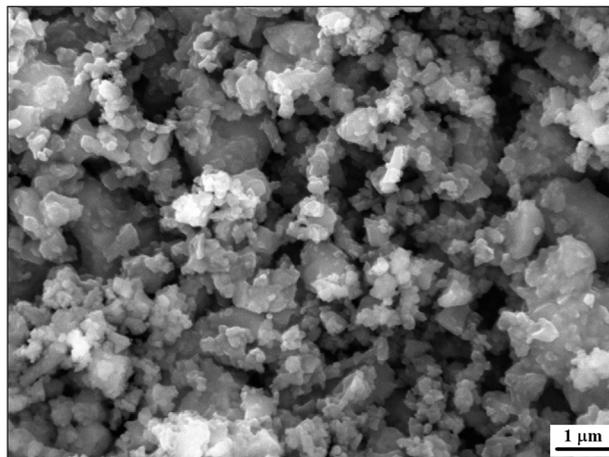
Fig. 1. Morphology of the Si_3N_4 powder
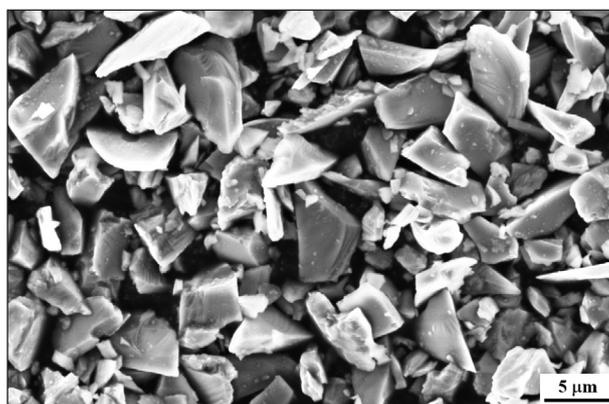
Fig. 2. Morphology of the SiC powder

The phase analysis was done using an X-ray diffraction method with Panalytical Empyrean diffractometer with source of characteristic X ray of CuKaα. Step size was 0.0260 °2Th and scan step time was 1.0 s. The wear resistance was evaluated by means of grinding the sample using a pin on disk method, Figure 3. Test samples with diameters of 8.4 mm and a height of 10 mm were placed in contact with corundum grinding paper with a graininess of 120 μm. The grinding trajectory was 125 m and the pressure 1.5 MPa. Maximal sliding speed was 0.5 m.s⁻¹, and radial movement 1.5 mm per revolution during dry friction of ceramic samples. The wear resistance was determined based on the volume loss of the samples relative to the grinding trajectory according to equation (2):

$$V_{V/s} = \frac{\Delta m}{\rho.l} \left[mm^3.m^{-1} \right], \qquad (2)$$

where: $V_{V/s}$ is volume loss of the samples,
Δm is weight loss of the samples,
ρ is density of the sample and l is grinding path of the sample. The microstructures of the hot pressed ceramics and surfaces after wear tests were observed using a scanning electron microscope JEOL IT- 300-LV.

RESULTS AND DISCUSSION

Microstructure and densification

The microstructures of experimental ceramic materials are documented from Figure 4 to 6. The microstructure of monolithic Si_3N_4 in Figure 4 consists of equiaxed α-Si_3N_4 grains, prismatic β-Si_3N_4 grains, and binding phase. Ceramic composite materials Si_3N_4 – SiC types in Figure 5 and 6 consist of matrix created by the same phases equiaxed α-Si_3N_4 grains, prismatic β-Si_3N_4 grains, and binding phase. In addition to that, SiC particles are irregularly distributed in this matrix. Prismatic β-Si_3N_4 grains were fine-grained by adding of SiC into the Si_3N_4 based matrix which can be seen in Figure 5 and 6. Fine-grained microstructure of ceramic composites with SiC particles could be the consequence of movement hindering of Si_3N_4 grain boundaries
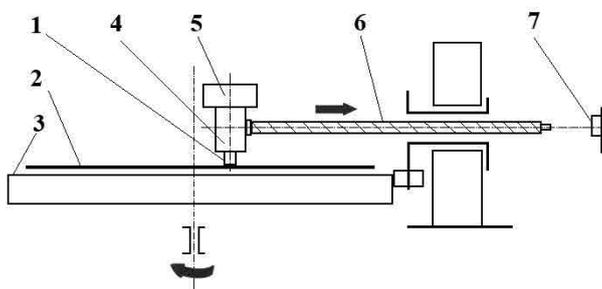
Fig. 3. Scheme of pin on disk method 1 – sample (pin), 2 – abrasive cloth (disk), 3 – board, 4 – clamping head, 5 – weight, 6 – sliding mechanism, 7 – power switch
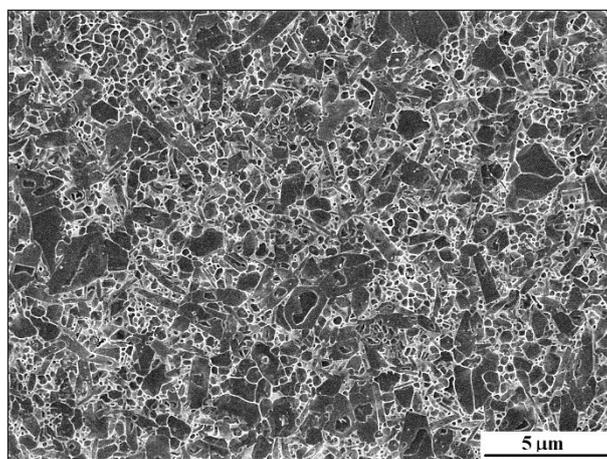
Fig. 4. Microstructure of SN sample
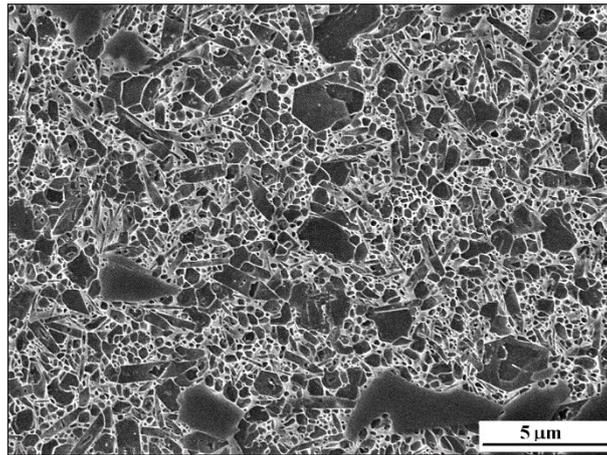
Fig. 5. Microstructure of SN10SC sample
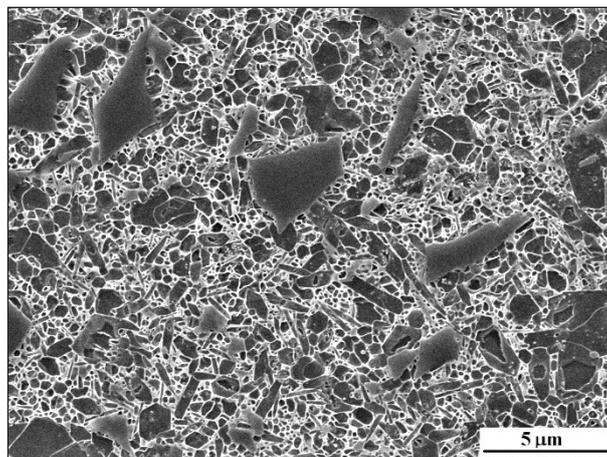
Fig. 6. Microstructure of SN20SC sample

by SiC particles or SiC clusters. The sample with higher SiC content gave a relatively smaller Si_3N_4 grain size because SiC particles limit the Si_3N_4 grain growth by pinning and prohibiting the grain boundary movement.

The maximal value of relative density 98.32% was achieved at the ceramic composite with addition of 20 vol.% SiC (SN20SC). The relative density was 97.85% for the ceramic composite with 10 vol.% (SN10SC). The relative density of the ceramic composite SN20SC is by 1.11% higher compared to monolithic Si_3N_4 ceramics. That means improving of densification of ceramic composite materials with SiC portion.

The identification of microstructural phases was done by X ray analysis and the records from this analysis are documented from Figure 7 to 9. Monolithic Si_3N_4 ceramic in Figure 7 consisted only of two phases α-Si_3N_4 and β-Si_3N_4. As the $Y_3Al_5O_{12}$ garnet (YAG) phase was amorphous

it could not be identified. The α-Si_3N_4 and β-Si_3N_4 phases were identified in all ceramic samples. The polytypes of α- SiC 6H and 4H were identified in both ceramic composite materials (Fig. 8 and 9). The ratio of phases 6H-SiC and 4H-SiC was the same in all analysed ceramic composites.

Phase transformation from a-Si3N4 to β-Si3N4 occurs during sintering process of Si3N4 based ceramic materials [19]. Figure 10 documents portions of phases in experimental materials after hot pressing process. The initial Si3N4 powder consisted of 100 % of a-Si3N4 phase. The highest portion of β-Si3N4 phase (66.5 vol.%) was measured for monolithic Si3N4. For SN20SC ceramic composite, β-Si3N4 portion decreased to the value of 51.3%. The lowest β-Si3N4 portion of 48.9 vol.% was measured for SN10SC ceramic composite.
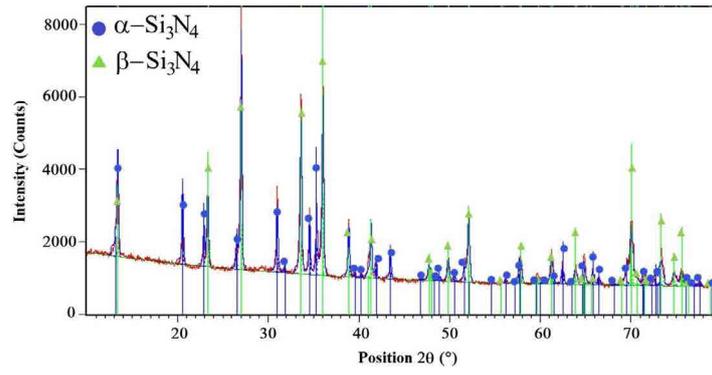
Fig. 7. X-ray record of SN sample
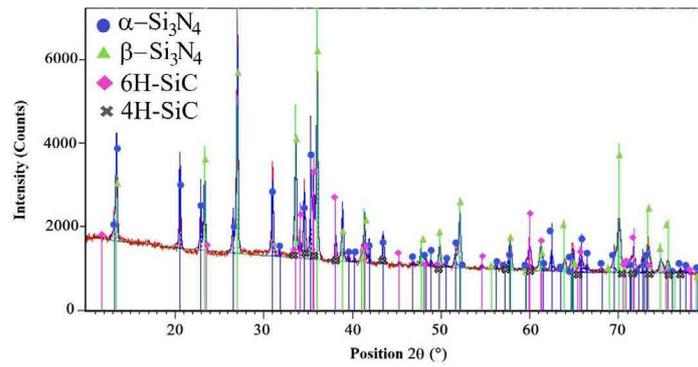
Fig. 8. X-ray record of SN10SC sample
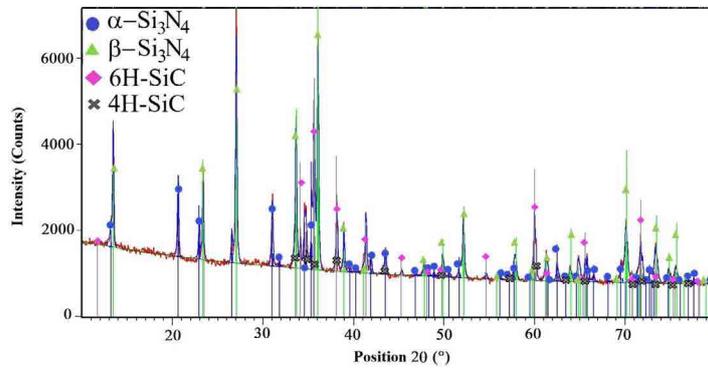
Fig. 9. X-ray record of SN20SC sample
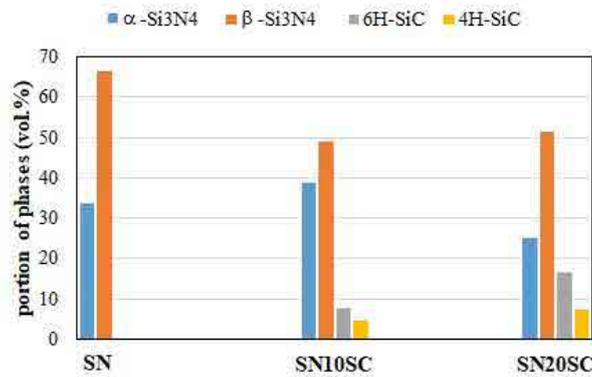
Fig. 10. The effect of SiC addition on phases portion in ceramic materials

Mechanical properties and wear

Average values and standard deviations of hardness and fracture toughness of experimental ceramic materials are summarized in Table 1.

From values in Table 1 it is clear, that the highest hardness was achieved at addition of 20 vol.% SiC into Si_3N_4 matrix. The lowest hardness value was measured for monolithic Si_3N_4 ceramics. The increase of hardness with increased SiC addition can be related to the higher hardness of SiC compared to Si_3N_4. The highest density measured for samples with the highest portion of SiC could positively influence the hardness values. From Table 1 the decrease of fracture toughness with increased portion of SiC can be seen. This effect could be caused the morphology of the matrix [19,32]. Prismatic β-Si_3N_4 grains, which increase the fracture toughness, could not reach the prolongated morphology, as the SiC particles

limited the growth of Si_3N_4 grains. The microstructure of monolithic Si_3N_4 ceramics consists mainly of prolongated prismatic β-Si_3N_4 grains with optimal grain prolongation and this is the reason for the highest fracture toughness of the monolithic Si_3N_4 ceramic material. Several effects such as morphology of Si_3N_4 matrix, grain size of SiC particles and difference in thermal coefficient values of Si_3N_4 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_3N_4 matrix and SiC particles. These stresses will support propagation of cracks in composite materials and decrease their fracture toughness. The fracture toughness of our specimens is in a good agreement with works [25, 39–42] where the values from 3 to 12 $MPa.m^{1/2}$ were measured for monolithic Si_3N_4. For Si_3N_4 composite with SiC particles the fracture toughness values were from 5.2 to 10.5 $MPa.m^{1/2}$.

The effect of SiC portion on wear of experimental material is presented from Figure 11 to 13. The volume loss of samples increased with increased portion of SiC secondary phase (see Fig. 11). The highest volume loss was recorded for

Table 1. Mechanical properties of sintered samples

	SN	SN10SC	SN20SC
HV (GPa)	14.48 ± 0.69	16.36 ± 0.55	16.99 ± 0.32
K_{IC} (MPa·m$^{1/2}$)	$8.30 \pm 0,57$	8.18 ± 0.47	7.09 ± 0.69
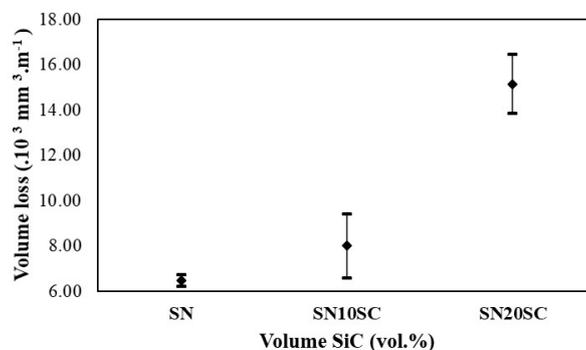
Fig. 11. Effect of SiC portion on volume loss of worn ceramic materials
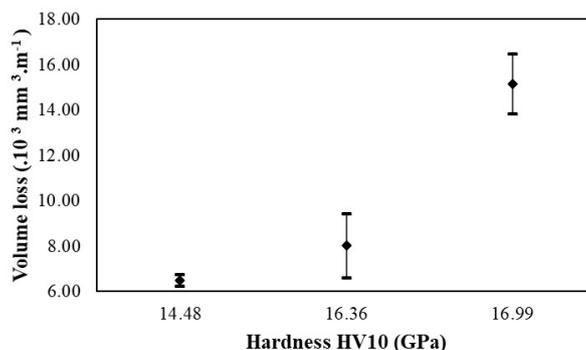
Fig. 12. Effect of hardness on volume loss of worn ceramic materials
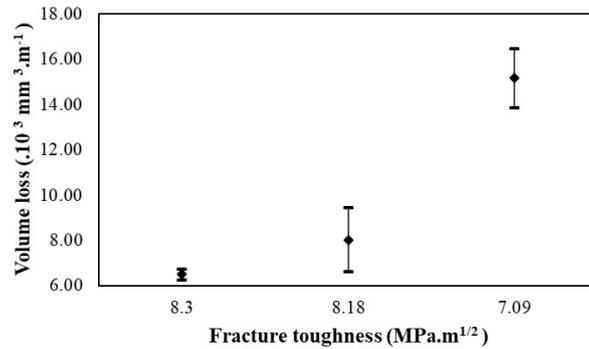
Fig. 13. Effect of fracture toughness on volume loss of worn ceramic material

composite material with 20 vol.% SiC (SN20SC). The lowest wear was measured for Si_3N_4 monolithic ceramics (SN). The hardness did not positively influence wear of ceramic samples (see Fig. 12). The highest volume loss was measured for material SN20SC with the highest hardness value. The lowest volume loss (the highest wear resistance) was achieved for material SN with the lowest hardness value. The effect of fracture toughness on volume loss of experimental material is documented in Figure 13. In this figure the lowest volume loss (the highest wear resistance) was observed for material SN with the highest fracture toughness value. The highest volume loss value was measured for material SN20SC with the lowest fracture toughness.

Figure 14 depicts the effects of β-Si_3N_4 phase portion on mechanical properties of experimental ceramic materials. The increase in β-Si_3N_4 portion had a positive effect on fracture toughness with the highest value of 8.30 MPa.m$^{1/2}$ reached at monolithic Si_3N_4 which had the highest β-Si_3N_4 portion. On the other side, higher hardness values were measured at lower portion of β-Si_3N_4 phase. The lowest wear damage was reached at the highest β-Si_3N_4 portion. The wear extent of ceramic

composites was significantly influenced by the presence of SiC particles which were braking out from the surface of composite materials during the wear test.

The extent of abrasive wear of ceramic materials can be expressed by the parameter ratio K_{IC} / HV. Based on the value of the K_{IC} / HV ratio, the predominant wear mechanism of ceramics can be determined. The effect of K_{IC} /HV ratio on volume loss of worn materials is presented in Figure 15. From the progress in Figure 15 it is clear, that the lowest volume loss (highest wear resistance) was measured at the highest K_{IC} /HV ratio for monolithic SN ceramics. The highest wear damage was recorded at the lowest K_{IC} /HV ratio for SN20SC ceramic composite. SN20SC ceramic composite achieved the lowest fracture toughness. From these facts it can be stated, that microcracking was the predominant wear mechanism during the wear test of experimental materials and fracture toughness has dominant effect on wear resistance of tested ceramic materials.

The worn surfaces of experimental ceramic materials after wear tests are documented from Figure 16 to 18. Both wear mechanisms microcutting and microcracking damage can be
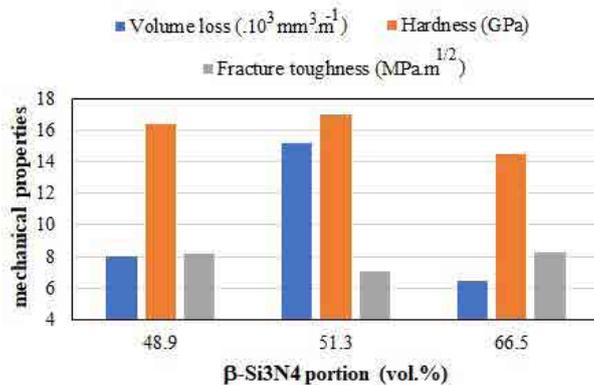
Fig. 14. Effects of β-Si_3N_4 portion on mechanical properties of ceramic materials
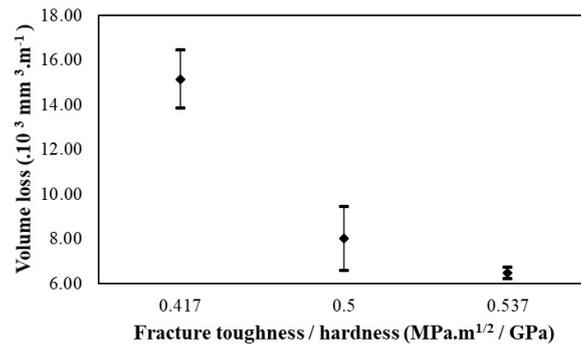
Fig. 15. Effect of K_{IC}/HV ratio on volume loss of worn ceramic materials
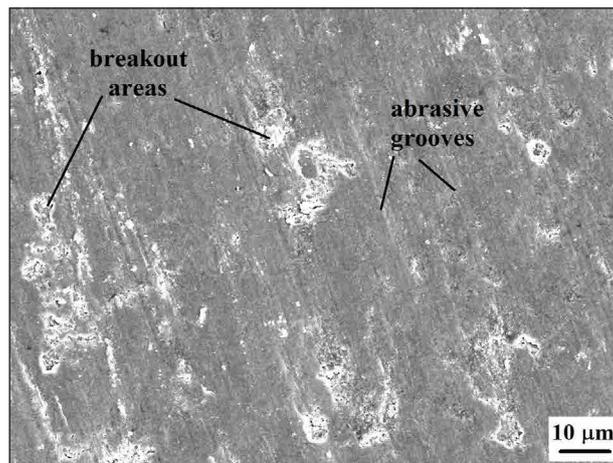
Fig.16. Surface of ceramic sample SN after wear test
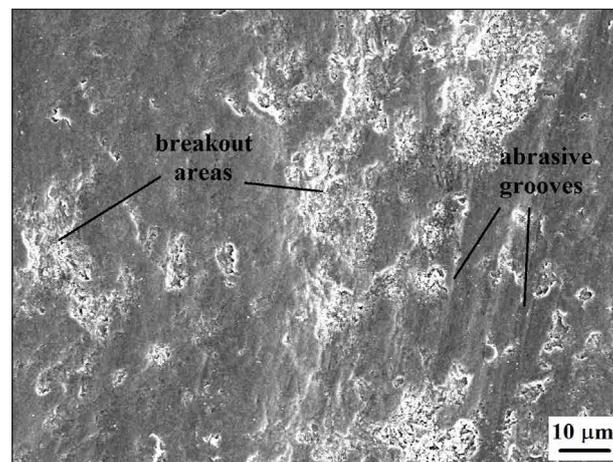
Fig.17. Surface of ceramic sample SN10SC after wear test

observed on all worn surfaces. Abrasive grooves and breakout areas can be seen in these figures. Some rips created by breakout matrix and microcracks (see Fig. 19) can be seen in damaged areas. The damage extent of SN surface is lower compared to SN10SC and SN20SC which corresponds with measured volume loss values of tested ceramic samples.

The detail of damaged area on the worn surface of SN10SC ceramic composite is documented in Figure 19. Several cracks can be seen at the borders of damaged areas. These cracks created mainly perpendicular to the grinding direction. Less cracks developed at monolithic SN ceramic sample. This fact can be attributed to higher fracture toughness of
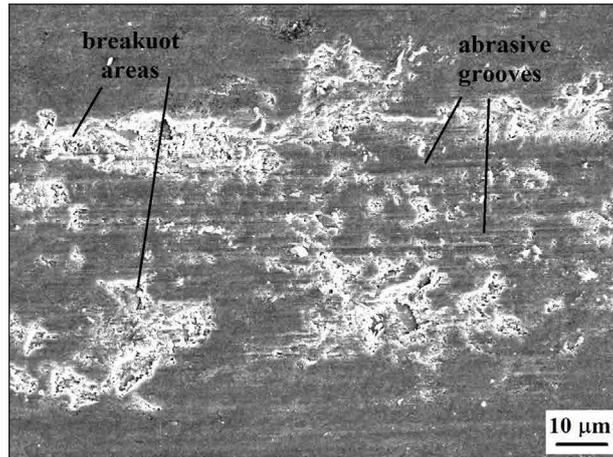
Fig. 18. Surface of ceramic sample SN20SC after wear test
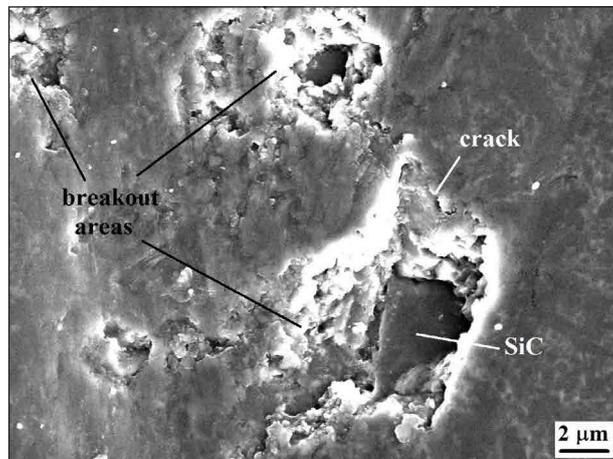
Fig. 19. Detail worn surface

monolithic ceramic compared to ceramic composites. Lower fracture toughness of ceramic composites contributed to easier breakout of matrix material or secondary phase from the tested composite surfaces.

Breakout areas created mainly in vicinity of SiC secondary phase particles which can be observed on the surface of composite materials. Both large grain size and irregular morphology of SiC contributed to the breakout of materials from surfaces of SN10SC and SN20SC ceramic composites. Based on results of work [25], high comprehensive stresses develop in Si_3N_4 matrix and tensile stresses develop in SiC particles because of different thermal coefficient of these phases, and these stresses support the breakout of materials from tested surfaces during the wear tests. The amount of such damaged areas increased with increased portion of SiC secondary phase which was proved by the highest volume loss for SN20SC ceramic composite.

The $Y_3Al_5O_{12}$ binding phase has a significant effect on the damage of tested ceramic surfaces. The thermal expansion coefficient of binding phase ($7 \cdot 10^{-6}$ °C^{-1}) is higher compared to Si_3N_4 ($2.2 \cdot 10^{-6}$ °C^{-1}), so high residual stresses develop at interface Si_3N_4 grains – $Y_3Al_5O_{12}$ binding phase. The fracture toughness of binding phase (3 MPa·m$^{1/2}$) is significantly lower than Si_3N_4 (8.30 MPa·m$^{1/2}$). High created stresses and low fracture toughness contributed to easier breakout of grains from tested surface.

CONCLUSIONS

The effect of SiC secondary phase portion on microstructure, density, hardness, fracture toughness of SiC-Si_3N_4 ceramic composite materials was investigated in the paper. Subsequently the effect of mechanical properties of SiC-Si_3N_4 composites on their abrasive wear resistance was

studied. From achieved results followed conclusions were derived:

- The matrix of all experimental materials consists of equiaxed α-Si$_3$N$_4$ grains, prismatic β-Si$_3$N$_4$ grains, and binding phase. Irregularly distributed SiC particles are in the matrix of experimental ceramic composite materials (SN10SC and SN20SC). At adding of SiC particles into Si$_3$N$_4$ matrix, the β-Si$_3$N$_4$ grains maintain their prismatic shape but thy are fine grained.

- The increase of SiC strengthening phase portion slightly increases relative density of Si$_3$N$_4$ – SiC ceramic composite materials.

- The SiC strengthening phase has a positive effect on the hardness of Si$_3$N$_4$ – SiC ceramic composite materials. The hardness of ceramic materials increased from 14.48 GPa at monolithic Si$_3$N$_4$ ceramics to 16.99 GPa at ceramic composite with 20 vol.% SiC.

- The SiC strengthening phase portion does not have a positive effect on the fracture toughness of Si$_3$N$_4$ – SiC ceramic composite materials. The highest fracture toughness value of 8.30 MPa.m$^{1/2}$ was achieved for monolithic Si$_3$N$_4$ ceramics, the lowest value of 7.09 MPa.m$^{1/2}$ was achieved for ceramic composite with 20 vol.% SiC.

- Wear resistance of tested ceramic materials was mainly influenced by their fracture toughness. The highest wear resistance value was achieved for material SN with the highest fracture toughness value. The lowest wear resistance was measured for material SN20SC with the lowest fracture toughness.

- Worn surfaces of all experimental ceramic materials were damaged by both microcutting and microcracking mechanism. Microcracking was the predominant wear mechanism mainly at ceramic composites. Cracks created mainly perpendicular to friction direction at the borders of damaged areas.

- Based on the achieved results, the wear and wear resistance of SiC-Si$_3$N$_4$ ceramic composites can be described by model W ~ HV/K$_{IC.}$

Acknowledgements

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