MECHANICAL PROPERTIES AND WEAR OF HOT PRESSED SILICON NITRIDE

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ABSTRACT

The effects of hot pressing parameters and mechanical properties on wear of silicon nitride ceramics were analyzed. The weakest microstructure constituent was the brittle crystal boundary phase that broke and chipped off during the wear tests. The volume loss of separate ceramic materials during the wear tests depended mainly in inverse proportion to the hardness of the ceramics. Both microcutting and microcracking mechanisms played a part in the wearing of Si_3N_4 ceramics. Based on these experimental results the volume loss and also the wear resistance of the ceramics under study can be described by the model $V \sim H_V^{-1}$.

Keywords : Silicon nitride, mechanical properties, microstructure, wear resistance.

1. INTRODUCTION

Silicon nitride ceramics is a very promising wear resistant material. It has high strength and hardness with acceptable toughness, good chemical stability and excellent thermal shock resistance. Si_3N_4 has been successfully used in a wide variety of engineering applications involving contact with metallic surfaces such as drawing dies, roller bearings, cutting tools, and automotive or aerospace engine parts. Abrasive wear is the most common mechanism of ceramic material removal. Two basic mechanisms of surface damage are applied during the abrasive wear of ceramics: microcutting and microcracking [1-3].

The mechanisms of microcutting can be described according to a model that comes from Rabinowitz´s conception of abrasive wear mechanism description [4]. In this model the abrasive particle in the cone shape makes grooves on the surface of the solid body. The removed material volume V can be described by equation (1):

$$V = \frac{F.l}{\pi.tg\alpha.H_V} \qquad (1)$$

where F is the force necessary to get the abrasive particle into the abraded material, l is the length of a groove on the surface if the cone moves during relative motion in parallel with the worn material surface, H_V is the hardness of ceramics, and α is the angle of the cone which participates in the grooving of the surface. It follows from equation (1) that the removed material volume is dependent only on one material property, hardness H_V.

If we consider the mechanism of microcracking, the abrasive particle creates a crack in the plane of the load axis after the overrun of the specific limit value of the load. Cracks spread to the sample surface, where they can develop into a fracture [2, 5]. The volume of wear V can be expressed by means of equation (2).

$$V = \frac{F^{9/8}}{K_{IC}^{1/2}.H_V^{5/8}} \cdot \left(\frac{E}{H_V}\right)^{4/5} .l \tag{2}$$

In this equation (2) l is length of path of abrasive particle, F is load on the abrasive particle, K_{IC} is fracture toughness of the ceramics, H_V is the hardness of the ceramics and E is the modulus of elasticity of the worn ceramic material. According to equation (2) the removed material, volume V, is dependent on three material properties: the modulus of elasticity E, the fracture toughness K_{IC} and hardness H_V.

According to some works [4, 6] the ratio of K_{IC}/H_V values appears as the main characteristic determining dominant wear mechanisms of brittle materials during abrasive wear. This parameter defines the dominant wear mechanism at the point of contact. The mechanism of plastic microcutting is predominant at a high value of this ratio, i.e. wear volume will depend on hardness ($V \sim 1/H_V$). Brittle fractures is predominant at a low value of the ratio K_{IC}/H_V, i.e. wear will increase with decreasing fracture toughness ($V \sim K_{IC}/H_V$). This influences the growth of wear intensity. According to equations (1) the intensity of microcutting decreases with the hardness of ceramics, and according to equation (2) the intensity of microcracking decreases with fracture toughness of the worn surface. This can lead to a transition from plastic microcutting to brittle microcracking during abrasive wear [1]. Besides the mechanical properties of ceramics, the character of the structure like grain size and β-Si_3N_4 phase ratio plays an important role in determining how the ceramics will react to specific states of stress which arise under specific conditions of wear [2, 7].

MATERIALS AND EXPERIMENTS

The ceramic material chosen in this study was silicon nitride, hot-pressed with sintering additives of yttrium oxide and aluminum oxide. The amounts of sintering additives Y_2O_3 and Al_2O_3 were different in separate specimens, but the proportion of Y_2O_3 and Al_2O_3 was the same for all the prepared specimens. The Y_2O_3 and Al_2O_3 powders were added in concentrations that could set creating a $Y_3Al_5O_{12}$ phase in ceramic materials. This phase contributed to the sintering ability of the ceramics [7]. The designations and compositions of the studied materials are given in table 1.

Tab.1 Chemical composition of ceramic samples

Ceramic sample	Concentration (wt.%)		
	Si_3N_4	Y_2O_3	Al_2O_3
5 % YAG	bulk	7.75	2.14
7 % YAG	bulk	8.78	3.00
10 % YAG	bulk	10.34	4.30

Initial powder compositions were wet mixed in alcohol. After drying and sieving, the powder was compacted in steel dies. The final densification was accomplished using hot pressing techniques in a nitrogen atmosphere with the purity of 99.99 % and an overpressure of 75 kPa. All samples were hot pressed at a temperature of 1680 °C and a pressure of 34 MPa. Three various sintering times 5, 15 and 30 min were applied.

Densities of the hot pressed ceramics were measured by the Archimedes's method. Hardness and fracture toughness were determined by means of the Vickers indentation method. The wear resistance was evaluated by means of grinding the sample using a pin on disk method. 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 was 1.5 MPa. The wear resistance was determined based on the volume loss of the samples relative to the grinding trajectory. The microstructures of the hot pressed ceramics were observed using a scanning electron microscope. In order to identify the microstructure created, the hot pressed specimens were subjected to XRD analysis.

RESULTS

The effects of densification, and mechanical properties of the microstructure, and the wear resistance of the prepared ceramics samples were evaluated.

DENSIFICATION AND MICROSTRUCTURE

Average values from measured densities in particular ceramic samples are included in table 2. The densities of the ceramic samples were influenced by the amount of additives (Y_2O_3 and Al_2O_3) and by the sintering time. They increased with increasing the concentration of additives and sintering time from 3.21 to 3.27 $g.cm^{-3}$. These values corresponded to the relative density from 97.2 to 98.5 %, which indicate good densification of samples. The smallest density, 3.21 $g.cm^{-3}$, was measured in the 5 % YAG sample with the smallest concentration of the additives, sintered for 5 min. The highest value of 3.27 $g.cm^{-3}$ was achieved in the 7 % YAG sample, sintered for 15 min and in the 10 % YAG sample sintered for 5 and 15 min.

Tab.2 Density of ceramic samples

Ceramic sample	Density ($g.cm^{-3}$)		
	5 min	15 min	30 min
5 % YAG	3.21	3.24	3.25
7 % YAG	3.23	3.27	3.26
10 % YAG	3.27	3.27	3.26

Each of the samples with different compositions and pressing time were analyzed by the XRD method (Tab. 3). Only two phases were found in all specimens α-Si_3N_4 and β-Si_3N_4 phases. Phases with a concentration below 5 % couldn't be identified using the XRD method.

Tab.3 β-Si_3N_4 phase ratio of ceramic samples

Ceramic sample	β-Si_3N_4 phase ratio (wt.%)		
	5 min	15 min	30 min
5 % YAG	45	58	75
7 % YAG	42	69	81
10 % YAG	43	62	100

During hot-pressing, the initial α-Si_3N_4 powder was transformed to β-Si_3N_4 phase, which can be seen from table 3. The transformation stage increases with both the additive concentrations and the sintering time. The portion of β-Si_3N_4 phase from 42 to 45 % was measured by XRD at a shorter sintering time of 5 min for all prepared compositions. There were small differences between separate compositions, but these were within the measurement error, which was 5 %. The

complete 100 % transformation of α-Si$_3$N$_4$ powder into β-Si$_3$N$_4$ phase was achieved only in the 10 % YAG samples with the highest concentration of additives and sintered for 30 min.

The study of the microstructure confirmed the effect of the composition on the formed phases. The microstructure consisted of α- Si$_3$N$_4$ and β-Si$_3$N$_4$ phase. The amount of overextended β-Si$_3$N$_4$ grain increased with both the additive content and at the sintering time and reached the full β-Si$_3$N$_4$ microstructure at the highest additive content and longest time, which can be seen in Fig. 5 (ceramic sample 10 % YAG sintered for 30 min). This can be explained by the increase in the transformation velocity at higher additive contents and longer times. With the increase of the additive amount, both the grain size and the ratio of a binding phase at the grain boundary increased. This crystal boundary phase is relatively brittle and can by indicated as the weakest component of the microstructure [7, 8]. The grain size also grew with the sintering time.

The effect of the additional concentration and sintering time on the wear of ceramic samples can be seen in Fig. 1. From this figure it can be seen that the volume change during the wear test decreased with the increasing of the additions and with sintering time. The highest wear resistance was achieved in the 10 % YAG sample, which was pressed for only 5 min, and the least wear resistance was with the 5 % YAG specimen with the smallest additional content, pressed for the longest time of 30 min.

MECHANICAL PROPERTIES AND WEAR

The effects of the measured mechanical properties on the wear properties of the ceramic samples were studied in detail and are presented in the fig. 2, 3 and 4.
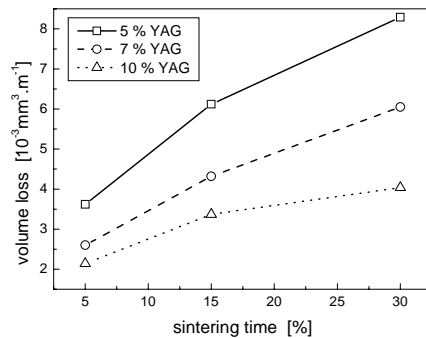

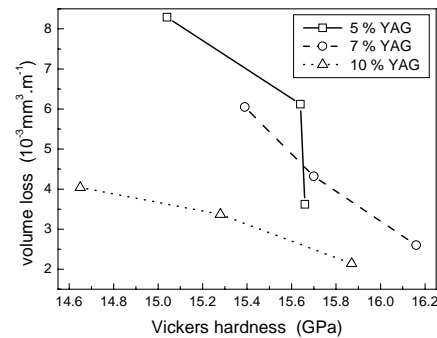Fig. 1 Effect of pressing time and additive concentration on wear of ceramics.

Fig. 2 Effect of hardness on wear of ceramics.

Hardness had a positive effect on wear resistance (Fig. 2). Higher hardness resulted in less wear. As the highest hardness was measured in the higher concentration of additives (ceramics 10 % YAG), these samples had the smallest volume of wear changes. The highest volume of wear changes were measured in the 5 % YAG samples with the least additives that was pressed for 5 min. The results in Fig. 2 correspond well with model ($V \sim H_V^{-1}$), where the volume losses V during the wear tests vary inversely in proportion to the hardness H_V of the ceramics.

A very interesting development was noted when the effect of fracture toughness on wear was measured (Fig. 3). All compositions showed the same progress. At first wear increased up to the maximum value. After reaching the maximum value wear decreased slightly. This can be explained by the relationship between the β-Si$_3$N$_4$ phase and grain size, but the differences between the separate samples within each composition were relatively small. The effect of grain

size was dominant. The highest grain size was always in the sample with a middle value of fracture toughness. That means the wear behavior can't be described only by the effect of fracture toughness on wear.

Wear behavior may be better described by the model which reflects the effect of ratio fracture toughness / hardness on wear rate. This can be seen in Fig. 4. These relations fit very well for each separate composition. The higher the value of the calculated ratio of fracture toughness to hardness is, the higher the wear rate is. The highest wear rate was noticed in the 5 % YAG specimen that contained the smallest concentration of additives that was pressed for 30 min. The ratio of fracture toughness to hardness accurately describes the relationship between the decrease of wear resistance in spite of the transformation progress α-Si_3N_4 phase to the β-Si_3N_4 phase, and the increase in the rate of wear in spite of grain growth.
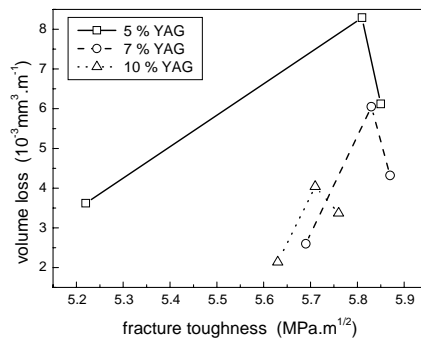
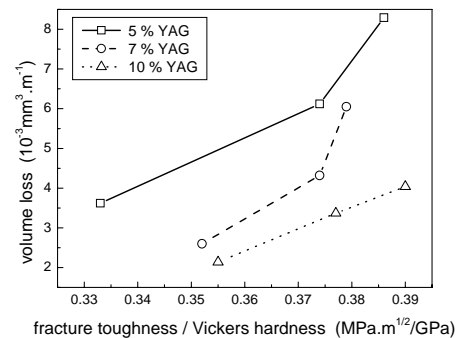Fig. 3 Effect of fracture toughness on wear of ceramics.

Fig. 4 Effect of ratio fracture toughness / Vickers hardness on wear of ceramics.

The ceramic surfaces after the wear tests confirmed the effect of additive concentration and sintering time on the wear resistance of specimens that were described above. The specimens which were pressed for a longer time showed greater damage than the specimens pressed for a shorter time. The specimens with higher contents of additives showed less damage than the specimen with smaller additive concentrations. These specimens have higher portions of binding phases, which is present on the grain boundary. The binding phase has a positive effect on the densification, which generally improves the mechanical properties of the ceramic materials. On the other hand, the binding phase is the weakest component of the microstructure. This brittle crystal boundary phase broke and chipped off during the wear tests, which can be seen in Fig 6. Prolongation of pressing time is connected to grain growth, which caused less wear resistance, thus the larger volume of the surface were enucleated.
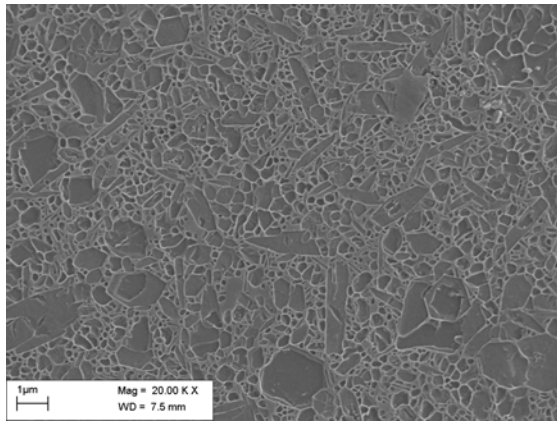
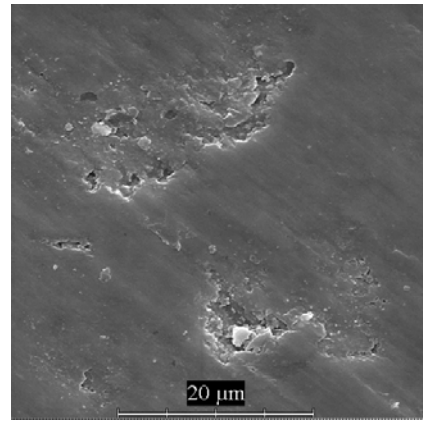Fig. 5. Microstructure of ceramic sample with 10% YAG sintered for 30 min-

Fig. 6 Surface of ceramic sample with 10% YAG sintered for 30 min after wear test-

Consistent with the theoretical background, the volume loss depends on hardness ($V \sim H_V^{-1}$) when the dominant mechanism of wear is microcutting. If the dominate mechanism is microcracking, the volume loss depends on the ratio hardness / fracture toughness ($V \sim K_{IC}/H_V$). All ceramic surfaces were damaged by microcutting and also by microcracking. This can be clearly seen from the observed surface after the wear tests (Fig 6). Several scratches and also damaged areas are visible. These areas are characteristic with dropped out material and many microcracks. These characteristic scratches and pits with microcracks were present on all specimens. Thus both procedures caused some wear on all specimens. However the extent of ceramic surface damage increased with pressing time.

In the case where both procedures occurred during the wearing process of the Si_3N_4 ceramic with alumina abrasive, both models could be used to interpret the experimental data. In our case, the experimental data corresponded better with the model, which assumes that microcutting is the dominant mechanism. Since there were only very small changes in the fracture toughness of separate specimens, the effect of ceramic hardness can be consider as dominant. Thus the volume loss during wear can be described by the first model ($V \sim H_V^{-1}$).

CONCLUSIONS

This paper concentrated on the analysis of the wear mechanism of hot pressed silicon nitride ceramics. The effect of preparation parameters chosen, such as chemical composition, sintering conditions in the microstructure, mechanical properties and wear resistance, was evaluated. Wear was mostly influenced by the hardness of ceramic materials. The specimen with the highest hardness achieved the highest wear resistance. Wear resistance of ceramics decreased with the grain growth and with the transformation progress of narrow α-Si_3N_4 phase to prismatic β-Si_3N_4 phase.

The weakest microstructure constituent was the brittle crystal boundary phase that broke and chipped off during the wear tests. Two damage mechanisms were taken into consideration on ceramic surfaces during the wear of Si_3N_4 ceramic pin samples with alumina grinding disks. Specimen surfaces were damaged by microcutting and also by microcracking mechanisms. The volume loss V of separate ceramic materials during the wear tests depended mainly inversely in proportion to the hardness H_V of the ceramics. Based on these experimental results the volume loss and also the wear resistance of the studied ceramics can be described by model $V \sim H_V^{-1}$.

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