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Effect of post heat treatment on the tribological behaviour of Si_3N_4 based ceramics

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Post heat treatment operation in oxidizing atmosphere were applied on Si_3N_4 based ceramics produced by sinter-HIP method. Mechanical and microstructural characteristics have been studied investigating the possible effect of the applied heat-treatment process on the tribological properties. Wear resistance and friction coefficient was determined by pin-on-disc method. The possible microstructural changes induced by the heat treatment process were investigated by scanning electron microscopy. Micro-hardness measurements were applied to study the possible correlation between the wear resistance and the hardness of the material. The microstructural and mechanical testing enabled to reveal some specific features of the wearing behaviour of the investigated material. Among the factors influencing the tribological performance of the certain Si_3N_4 based ceramic material, the toughness characteristics are considered to play more important role than hardness. The importance of understanding the wearing damage process at microscopic level and identification of the controlling mechanisms are emphasized.

1. INTRODUCTION

Wear resistant rotational parts are basic structural elements in mechanical engineering. Long-term space mission and operations require non-lubricated bearing parts. Technical ceramics especially Si_3N_4 based ceramics and SiAlONs are good candidates for this applications. Lifetime and surface performance of these materials could be increased by various surface and volume treatments. However, the exact micromechanical processes controlling the wearing damage process of Si_3N_4 ceramics are not still cleared up.

The present work aims at contributing to the better understanding of the wearing damage process of the Si_3N_4 based ceramic materials.

The first results put light on the possible effect of the post heat treatment operation on the wearing properties characterised by the worn volume and friction coefficient, as well as the geometry of the worn profile. These findings were completed by hardness and microstructural investigations in order to get a more complex picture on the wearing behaviour. The present study summarises the current state of the work.

2. EXPERIMENTAL PROCEDURES AND RESULTS

2.1 Material and heat-treatment conditions

The basic composition of the samples in weight percent is characterized by as follows: Si_3N_4: 90%, Al_2O_3: 4% and Y_2O_3: 6% [1]. Samples were heat treated in air at 800, 1000, 1200 and 1400°C for the same treatment time of 48 hours. After sintering the prismatic samples have dimensions of 5x4,5x50 mm. The Si_3N_4 based ceramic specimens were made by sinter-HIP method [1], produced by the MTA MFAKI (Research Institute for Technical Physics and Materials Science of the Hungarian Academy of Sciences).

2.2 Principle and conditions of the wearing test

The principle of the wear test and the scheme of the measuring equipment are illustrated in Fig. 1.

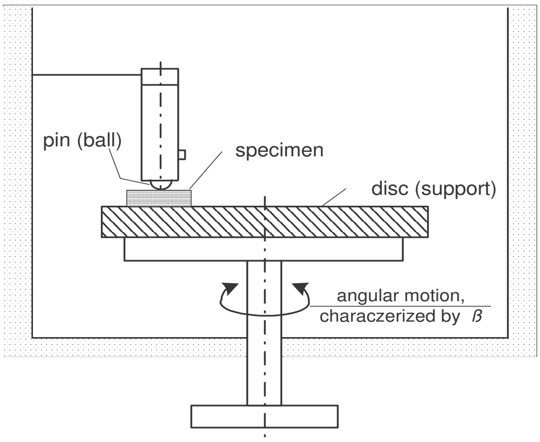


Fig. 1. Theoretical scheme of the pin-on disc measurement

The relative motion of the tool and the specimen is provided by the alternating circular motion of the specimen fastened on a disc rotating in the plane perpendicular to the tool axis.

The track length can be controlled by the angular displacement (β) of the support, which was kept constant during our investigations.

Pin-on-disc wear tests were accomplished using Al_2O_3 ball tools, having diameter of 4,6 mm. The specimens had a grinded surface quality. The testing conditions are characterized by the following circumstances:

- Loading force: F = 5N, 10N and 20N,
- Total running distance, L ~ 100m,
- Track length, l was 7,3 mm,
- Circumferential velocity of specimen (disc) = 0.05m/s.
- Environment: air;
- Temperature: 24°C;
- Relative humidity: 60%.

2.3 Tribological characterisation of the investigated material

Tribological behaviour of the ceramic materials were characterized by the following features:

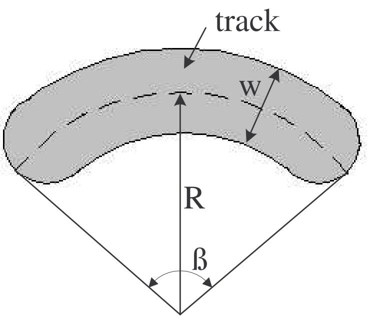
- geometrical characteristics of the worn surface;
- wear rate;
- friction coefficient.

The geometrical characteristics of the wearing track are shown in Fig. 2.

The wear rate, *r* is defined as the worn volume per unit loading force, per unit sliding distance, i.e.

$$r = \frac{V}{F \cdot L}\left[\frac{mm^3}{N \cdot m}\right], \tag{1}$$

where *F* is the loading force, [N]; *L* is total sliding distance, [m] and *V* is the worn volume, [mm^3].



R: radius of track
ß: angle of track
w: width of the track

Fig. 2. Principle of the tribological test

The worn volume is the sum of the track length *(l)* and the cross area *(A)* of the worn profile:

$$V = l \cdot A \; [mm^3], \tag{2}$$

where $l = (r \cdot \pi)\left(\frac{\beta}{180°}\right)$, and *A* is determined by profilometer measurement. The area of the worn profile was quantified using PC controlled Stylus profilometer (Rank Taylor Hobson Surtronic 3+).

According to Fig. 3. the worn profile is defined by the diameter of the worn surface of the ball (tool), *D*, furthermore the width of the track, *w*. Based on the geometrical model illustrated in Fig. 3. it was assumed that the intersection line of the matching surfaces, i.e. that of the ball and specimen is parallel with the rotational plane of the disc. The detailed measuring results relating to values of *w, D* and *A* can be found in work [2]. The wear rate, i.e. *r* values calculated by eq. 1. are summarised in Table 1. Detailed evaluation of the results will be given in the next section.

The friction coefficient, μ was recorded during the wear test by the computer program of the pin-on disc equipment. As an example, Fig. 4. illustrates a typical diagram indicating the minimum, mean and peak values of the friction coefficient. Table 1. presents the mean values which ranged from 0,3 to 0,5 for all test conditions (varying load). The friction coefficient represents a similar trend as the wearing rate, i.e. higher wear rate was associated by higher friction coefficient.

Table 1.
Results of tribological measurement

Sample	Heat-treatment	Friction coefficient	Wear rate [mm^3/Nm]
Am3509	No	0,36	6.7E-5
Am3510	No	0,35	5.8E-5
Am3511	800 °C	0,49	12E-5
Am3512	800 °C	0,5	9.3E-5
Am3513	1000 °C	0,36	n.d.*
Am3514	1000 °C	0,47	8.2E-5
Am3515	1200 °C	0,38	5.2E-5
Am3516	1200 °C	0,3	7.8E-5
Am3517	1400 °C	0,45	1.1E-3
Am3518	1400 °C	0,46	n.d.

*No available measuring data

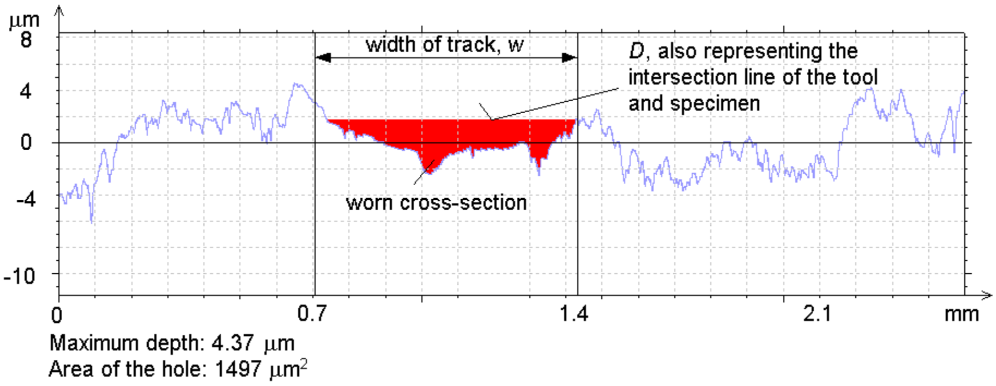


Fig. 3. Explanation of the worn cross sectional area on the specimen surface

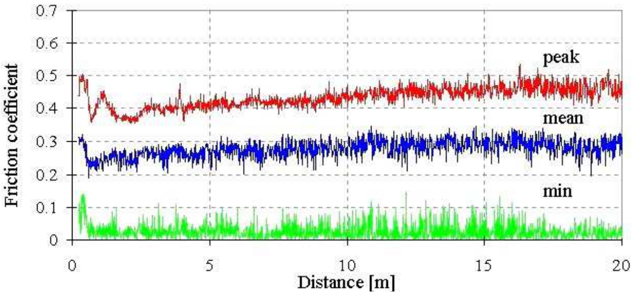


Fig. 4. Typical friction coefficient diagram registered during pin-on-disc wear test

3. EFFECT OF THE POST HEAT TREATMENT OPERATION

The post heat treatment operation is a useful mean of controlling the amount of the crystalline and the grain boundary glassy phase [3], whereby the mechanical performance of the ceramic material could be adjust to the application demands.

During post heat treatments, i.e. that is applied after the sintering process of the product, the material may experience significant microstructural changes. At certain temperatures characteristic reactions, phase transformations may occur: e.g. at 1350°C the reaction of the matrix and the grain boundary glassy phase can result in the formation of ß'Si_3N_4 of a slightly modified composition, as well as crystalline $Y_3Al_5O_{12}$ may be formed. At 1050°C another crystalline phase, i.e. the Y_2SiAlO_5N may be developed. In the temperature region of 1000-1800°C further, partly metastable phases can be produced. All of these changes may affect the wearing behaviour of the ceramic.

For investigating the effect of the applied post heat-treatment operation the wear rate of heat-treated samples were compared, as illustrated in Fig. 5.

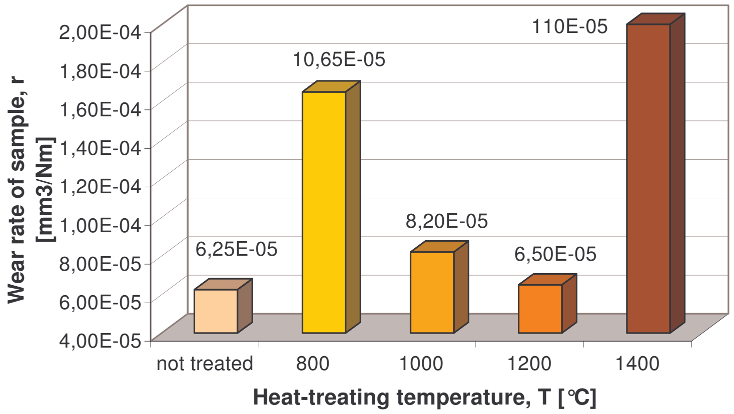


Fig. 5. Wear rate of heat-treated samples

Comparing to the untreated sample the wear rate (see Table 1.) was higher in case of heat-treatments at 800, 1000°C. Samples heat treated at 1200°C show the best wearing behaviour among the treated samples, however not better, than the untreated material. It is assumed that during heat treatment structural modification occurs. This will result in worse wear resistance in the case of 800 and 1000°C treatments.

For samples with heat treatment of 1400°C the wear rate show the highest value. Moreover, the samples in this case had very rough surface.

At higher temperatures new phases develop this has been proved by X-ray diffraction technique. These results are still under evaluation, for identification of the new phases.

The crystallisation of the intergranular glassy phase, as well as formation of complex oxides may be responsible for the experienced better results belonging to 1200°C.

Microstructural observations

The microstructure of the differently heat-treated Si_3N_4 ceramics was examined by scanning electron microscopy. The surface was automatically polished using ϕ9, 6, 3 and 1µm diamond paste. The polishing time was 10 minutes for each steps. The specimens surface were etched in 50% NaOH for 10 minutes [4]. The untreated sample contained predominantly ß-Si_3N_4 grains as it could be observed by electron microscopy, as illustrated in Fig. 6.

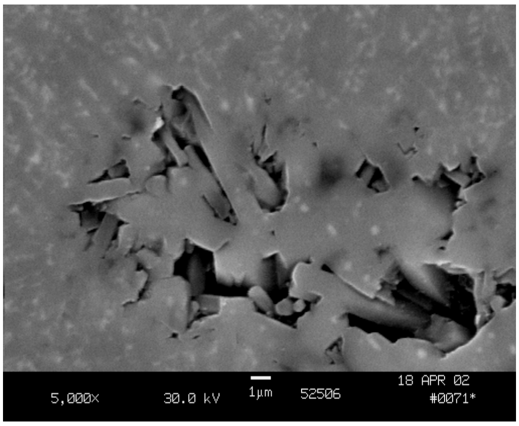


Fig. 6. SEM picture of the etched surface of untreated sample, (U_{acc} = 30.0 kV, M = 5.000×)

The post heat-treatment operation caused not significant microstructural changes at temperature 800°C. At 1000°C the initiation of structural modification could be observed, i.e. the ß-Si_3N_4 grains became thinner. The structure of the samples treated at 1200°C indicated further refinement of the ß-Si_3N_4 needles.

Fig. 7. shows a drastical structural transformation of the material under the influence of treatment at 1400°C.

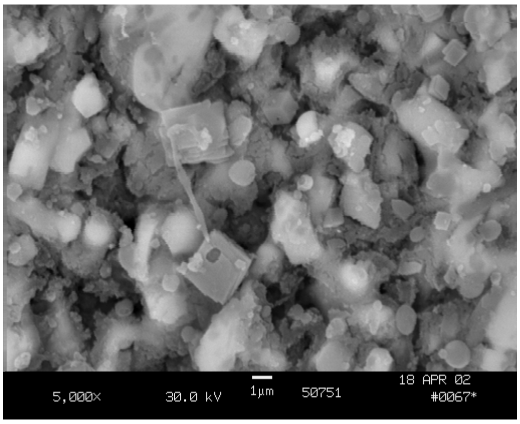


Fig. 7. SEM picture of sample treated at 1400°C (U_{acc} = 30.0 kV, M = 5.000×)

On the one hand, characteristic new grains of cubic morphology can be found in the structure. Furthermore the volume of the sample increased, as reported by the producer that was not experienced in case of the previous samples. The growing volume could be ascribed to oxigen built up into the Si_3N_4 in the form of complex oxides of Si and N, due to the oxidizing atmosphere.

4. HARDNESS TEST

Micro-Vickers hardness tests were carried out, using a MITUTOYO micro-hardness testing equipment. The applied load, *F* was 5N and the loading time, *t* was 10sec. The indentation diagonals were measured on polished samples by optical microscope, with magnification of M=400x.

In Fig. 8. the wear rates of the differently heat-treated samples and their hardness values are compared.

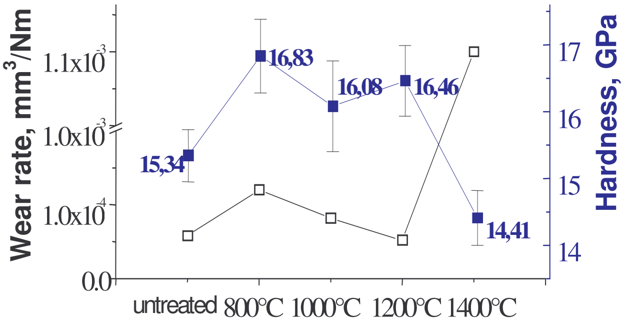


Fig. 8. Comparison of the wear rate and hardness values determined on Si_3N_4 ceramics, (□: wear rate; ■: hardness)

It can be seen, that the post heat-treatments at 800 and 1000°C increased the hardness of the silicon nitride ceramic comparing to the untreated state, while the measured wear rate values reflect higher material loss during the wearing. This behaviour is different comparing to the metals, where higher hardness is associated with better wearing performance. In the ceramic material the wearing damage is controlled by brittle fracture of grains, while in case of metals plastic deformation play a leading role during wearing.

In case of the 1200°C treatment higher hardness value was accompanied by a comparatively same wear rate as for the untreated state. It is assumed that the structure of the ceramic material started to undergo a transformation that could change the controlling mechanism of the wearing process.

The hardness-wear relationship can not be evaluated for the case of 1400°C treatment, due to the extremely rough surface of the probe caused by the observed (see Fig. 8.) structural changes.

5. DISCUSSION AND CONCLUSIONS

At the current stage of our research directed to getting a more detailed overview on the tribological behaviour of a sinter-HIP-ped Si_3N_4 ceramic material the most important observations and conclusions can be summarised as follows:

The applied post heat-treatment operation in oxidizing atmosphere influenced the wearing properties of the investigated Si_3N_4 material in a complex way.

In case of 1200°C operation the wear rate was comparable with that of the untreated state, while treatments at 800, 1000, and 1400°C caused significantly higher material loss.

The assumed structural changes above 1000°C, i.e. crystallization of the intergranular glassy phase or formation of new complex oxides should be verified by further investigations, e.g. X-ray diffraction technique.

Heat-treatment of 1400°C caused phase transformation leading to extremely rough surface of the material, deleterious to the wear resistance.

In contrast with metallic materials, unambiguous correlation between hardness and wear rate can not be established for the studied Si_3N_4 material.

Since the conventional approach on the direct relationship between hardness and wear resistance does not apply, correlation between fracture toughness — an essential property that may control the wearing behaviour — and wear resistance has been suggested to be investigated for the given material.

A direct practical benefit of this analysis can be the possible optimisation of the post heat-treatment operation in order to provide improved tribological performance of the studied Si_3N_4.

As a future goal, this research programme is continued for more detailed understanding of the tribological behaviour of Si_3N_4 based ceramics, revealing the complex interaction of the structural and mechanical characteristics, as well as investigating the possible advantageous effect of applying advanced volume and surface treatment technologies.

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