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THE EFFECT OF MODERN PROPERTY MODIFICATION TECHNOLOGIES ON THE TRIBOLOGICAL BEHAVIOR OF Si_3N_4-BASED TECHNICAL CERAMICS

Synopsis of PhD theses by

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1. INTRODUCTION

1.1 PRELIMINARIES

In recent decades, ceramic materials have been widely used in numerous engineering applications due to their excellent material properties. Thanks to the development of the material science, material technology and the methods of material testing they can be tools of the diverse user needs. In many areas, they are not only suitable for replacing metals, but also for providing superior properties [1].

Ceramics are non-metallic inorganic solids. Compared to conventional ceramics, modern technical ceramics (e.g. Al_2O_3, Si_3N_4, SiAlON, SiC, ZrO_2, TiC, TiN, B_4C, etc.) show more advantageous properties in terms of hardness, density, abrasion resistance, strength, etc. and their toughness characteristics are more favourable. These are artificially produced ceramics with a well-defined composition and microstructural characteristics formed in a strictly controlled manufacturing process.

Their application is extremely wide-ranging: on the one hand, motor and precision instrument parts, abrasive discs, cutting inserts, pull rings, furnace liners, reactor jackets, bioprostheses, reinforcing fibers, etc. as bulk materials, on the other hand, they are used in the form of a coating to improve the surface properties of the products [2].

Institute of Technical Physics and Materials Science deals with the production and material development of Si_3N_4-based, high-tech quality ceramics in Hungary. The products manufactured here - during their use so far - have been subjected primarily to stress and heat resistance.

1.2 CHARACTERISTICS OF DEVELOPMENT AND RESEARCH OF ION-IMPLANTED Si_3N_4 CERAMICS

In addition to technical adequacy, cost is always a decisive argument in selecting the most suitable material for a given application. The production of silicon nitride ceramics is a very expensive process due to the high cost of foreign raw materials, special manufacturing processes that ensure suitable mechanical properties for technical application, and the high material and equipment costs of qualification tests. Therefore, it is very important to make the production of these products as economical as possible by optimizing the user properties and increasing the reproducibility and reliability of the tests.

On the other hand, it is inevitable to develop new property improvement processes that increase the performance of the material while not or only slightly increasing the cost. Improving the mechanical and tribological properties of Si_3N_4-based ceramics can be achieved in two ways: one is to modify the structure of the material through the parameters of the manufacturing process (additives, sintering temperature, sintering medium, etc.) and thereby achieve the desired changes in properties. The other option is to submit the finished product to some structural or property-modifying treatment. According to the latter the tests presented in the dissertation were performed on samples modified by heat treatment and ion-implantation.

Heat-treatment

One way to improve the high temperature properties is to post-heat treat the ceramics, thereby promoting crystallization of the intergranular glass phase [3, 4, 5, 6, 7, 8]. The phase transitions that occur during the oxidation of Si_2N_4-based ceramics containing Y_2O_3 and Al_2O_3 as sintering aids are highly dependent on the concentration of components and heat treatment conditions. The presence of the intergranular phase, and even more so the content of nitrogen, oxide cations, and impurities, has a large effect on the behaviour of silicon nitride in ceramic oxidation. By heat treatment of HIP sintered silicon nitride ceramics in an oxidative atmosphere, surface oxidation mostly occurs, but some degree of internal oxidation may also occur. This phenomenon is most characteristic of Y_2O_3-containing ceramics and contains an intergranular fourth-order Si-Y-O-N phase. Oxidation of oxynitrides involves a large increase in volume, which causes material structural changes at higher temperatures and, consequently, a possible decrease in strength. [9, 10]. By selecting the appropriate amount of excipients, a stable Si_3N_4-$Y_2Si_2O_7$-Si_2ON_2 phase triple can be formed, which is resistant to oxidation at high temperatures, avoiding a decrease in strength [11]. The temperature ranges where post-heat treatment inevitably increasing the strength are the glass transition temperature (800 °C) and the eutectic temperature of the intergranular phase of the Si3N4-based ceramic (typically the range of 900-1000 °C). Typically, the above-mentioned Si-Y oxynitrides are formed below 1050 °C, at which time crystallization of $Y_2Si_2O_7$ also begins [12]. Oxygen diffusion occurs between 1100 ° C and 1350 ° C, the components diffuse to the surface at the grain boundaries, reacting with the SiO_2 film here [13] and above 1350 ° C a liquid silicate phase is formed and the SiO_2 film dissolves in this phase [14]. Above 1400 ° C, a significant surface layer thickening and a significant decrease in density are to be expected [15].

Thus, the amount of the crystalline phase or the grain boundary glass phase can be controlled by targeted heat treatment, while the mechanical properties can be customized for the desired use [16, 17, 18, 19].

Ion-implantation

Ion implantation can be used both to change the properties of the surface layer and to make a coating [20]. Ion implantation is a surface treatment process in which a solid is bombarded with accelerated ions to get inside the material. These accelerated ions are collectively referred to as ion beams.

Only few research groups deal with the property-improving surface treatment of silicon nitride-based ceramics, possibly due to the fact that the use of ion implantation is more common method for semiconductors rather than ceramics.

BREINSCHEID [21] et al. found that the wear of silicon nitride ceramics implanted with Ti + ion (0.5 MeV, 1 MeV, 1.5 MeV, and 2 MeV) was decreased in all cases due to the formation of a near-surface amorphous layer during implantation. This is explained by the fact that amorphization results in a strong increase in volume, which creates a compressive residual stress in the material, which in turn leads to the closure of surface microcracks. The formation of nano-crystals in the amorphous layer is also mentioned, their formation depends largely on the applied implantation energy, the TiN nano-crystal formed in Si_3N_4-based ceramics mentioned in the literature was formed at 70 keV energy [22].

NAKAMURA et al. [23, 24, 25] also performed microstructural and wear studies on ion-implanted silicon nitride-based ceramics. Implantation was performed with 200 keV energy and different ions (B^+, N^+, Si^+ and Ti^+). Their experimental results also support the formation of a subsurface amorphous layer.

GYULAI [26, 27] and his group performed implant surface treatment on C^+ and N^+ ions with energies of 190 keV and 350 keV on silicon nitride ceramics. The series of measurements presented in this dissertation is intended to supplement these measurement results, among other things.

BOLSE [28] points out that for all materials the structural freedom of the crystallographic arrangement of the associated atoms determines the amorphizability and recrystallization, so in the case of tetrahedral Si_3N_4 polycrystalline ceramics the amorphization occurs in most cases, recrystallization is performed at high temperature [29].

1.3 Objectives

In my dissertation, I present the tribological properties of Si_3N_4-based ceramics, which is a key issue among the technical ceramics, looking for correlations between the mechanical and wear properties and the microstructural properties of the material. My basic goal is to present the different surface and volume modification methods and to investigate how the surface and volume treatments applied in my research influence the wear behavior of the material, are they suitable, and if so, to what extent to improve tribological performance?

Abrasion testing is a widely known and effective method for characterizing pairs of materials in contact and friction, but is not suitable for the quantitative characterization of wear damage to a given material. Among other things, I would like to prove this within the framework of the dissertation. The test conditions used and the material modification methods of the material highlight that the concept of tribological characteristic should be understood as the abrasion behavior under conditions and under a given material pairing, and this approach will help in material selection rather than estimating wear damage value retrieved from a table for the purpose. When compiling the series of measurements, I carefully considered and optimized the number of samples available to me - in some cases very expensive - and expediently determined the test parameters needed to study and prove the assumed correlations between material structure and wear behavior.

Based on the questions to be answered, the measurements performed can be divided into two major groups:

- Measurements to analyze the effect of test conditions and to select measurement parameters (Preliminary experiments)
  - Effect of humidity on wear behavior;
  - Effect of loading force on wear behavior;
  - Effect of cycle number on wear behavior.
- measurements to investigate the effect of property modification procedures on wear behavior (Target experiments)
  - Investigation of the effect of volume management procedure;
  - Investigation of the effect of surface treatment procedure.

In order to understand these effects and find relationships between mechanical and wear properties, it is also important to explore the microstructural changes that take place in the material. I tried to formulate my objectives in such a way that the findings of my research could reveal gap-filling relationships between the property-modifying treatments of Si_3N_4-based ceramics, including oxidative heat treatment and ion implantation, both in terms of material structure, mechanical and tribological properties.

2. INVESTIGATIONS PERFORMED

During the literature review, my goal was to explore the theoretical background and existing knowledge required for the research work included in the objective. To this end, I analysed the material structure properties of the examined Si_3N_4-based ceramics and the effect of the applied heat treatment and ion implantation on the composition. After understanding the wear properties of ceramics, I explored the steps and computational background of the tribological study.

Surface and volume treatment of Si_3N_4-based ceramics was performed with several parameters to determine the relationship between material structure properties and wear behaviour. Based on the literature data and the manufacturer's recommendation, the post-sintering heat treatment was performed on the ceramics at 800, 1000, 1200 and 1400 ° C in an air atmosphere. During the heat treatment in an oxygen atmosphere, the heating rate was 24 °C/min and the cooling rate was 10 °C/min. The holding time was 50 h in each case. The Si_3N_4-based ceramic samples with polished surface were implanted with two types of C^+ and N^+ ions with energies of 0.5 MeV, 1 MeV and 2 MeV. The energy density was $9.9 \cdot 10^{17}$ ions/cm^2 in each case.

The abrasion tests were performed at the Federal Institute for Materials Research and Testing in Berlin, applying ball-on-disc type abrasion tribometer for the tests, using an oscillating fretting technique. Using several test techniques - load force, number of cycles - the following tribological parameters were determined:

- coefficient of friction;

- wear coefficient;
- wear mechanisms.

During the investigations, the wear coefficient was interpreted as the combined wear of the tested sample (disc) and the friction pair (ball) as follows:

$$k = \frac{W_v}{\Delta x \cdot N \cdot F_n}, \quad (1)$$

where

k: wear coefficient [mm^3/Nm];

W_v: the total volumetric wear [mm^3];

F_n: normal load [N];

Δx: length of wear track [mm];

n: number of cycles.

Thus, the degree of wear damage is basically characterized by the amount of material that occurs during wear, i.e., the amount worn out and the various specific amounts that can be derived therefrom.

Besides the tribological studies, several additional investigations were performed to determine the tissue structure, hardness, Vickers imprint fracture toughness, modulus of elasticity, and phase composition of the heat-treated ceramics and the material structure of the surface layer of ion-implanted specimens.

3. SUMMARY

During my research work, in order to increase the tribological performance of domestically produced Si_3N_4-based ceramics, I performed ion-implantation with N^+ and C^+ ions and volume post-heat treatment in order to modify the surface layer of the sintered ceramic.

In order to evaluate the changes in tribological properties, I performed fretting-type wear tests according to the typical technical use of the given type of ceramic.

Experiences of the tribological behaviour of Si_3N_4-based ceramics **heat-treated in oxygen atmosphere** with 24 °C/min heating, 10 °C/min cooling rate and 50 h holding time at T = 800, 1000, 1200 and 1400 °C in full volume can be summarized below:

- The changes in the structure of the heat-treated material were detected by scanning electron microscopy, X-ray diffraction and transmission electron microscopy.
- I found that the heat-treatment at 800 °C (glass transition temperature of the Y_2O_3 and Al_2O_3 based ceramics) had a beneficial effect on the wear resistance of the investigated tribo-systems. The lower wear coefficient was caused by the combined decrease of wear of both the specimen and the ball.
- By the structural transformation of the specimen heat-treated at 1000 °C the formation of a new phase, $Y_2Si_2O_7$ has started, but its effect on tribological properties is not yet clear. The wear of the whole system was decreased (although only to a small extent, due to a reduction in tool wear, i.e., an indirect effect related to sample handling).
- Due to the significant structural transformation of Si_3N_4-based ceramics heat-treated at 1200 °C, new phases can be already clearly detected, which represent a drastic structural change in both the samples heat-treated at 1200 and 1400 °C. All of these brought some improvement in sample wear compared to the 1000 °C treatment, however, even at these temperatures, the porosity of the surface layer already greatly increased, promoting the dominance of abrasive wear, which increased tool wear in particular. The wear coefficient decreased slightly compared to the previous condition (1000 °C heat-treatment), approaching the wear value measured on the untreated sample.
- It can be stated that the tribological properties of the tested Si_3N_4-based ceramics can be improved by heat-treatment after sintering, and there is an optimal temperature (T = 800 °C) in the experimentally studied temperature range where the structural transformations are favourable in terms of wear resistance [30, 31].
- The value of the friction coefficient showed a general decrease in tribo-systems containing specimens treated at different temperatures, compared to the untreated

material, which was clearly caused by changes in the material structure due to heat-treatment. A novel observation is that while in the case of the material heat-treated at 800 °C this structural change was accompanied by a decrease in the wear coefficient due to the initial transformation (crystallization) of the glass phase, the wear properties of the heat-treated samples deteriorated at 1400 °C caused by the structural degradation (porosity increase, loss of intergranular phase integrity) associated with glass phase transformation.

Ion-implantation of C^+ and N^+ ions, 0.5, 1 and 2 MeV energies and $9.9 \cdot 10^{17}$ ion/cm^2 energy densities is modified the tribological behaviour of the investigated Si_3N_4-based ceramics, the main findings of the relevant experimental work are as follows:

- During microstructural studies, I confirmed by transmission electron microscopy that an amorphous layer was formed in the surface layer of the ion-implanted Si_3N_4-based ceramic. Between the surface and the amorphous layer, due to the inhibition of ions in this range, a partial structural transformation took place, “nano-bubbles” were formed at the boundaries of the crystalline particles and the intergranular glassy phase.
- Avoiding costly studies, I estimated the thickness of the near-surface layer formed by ion-implantation with different parameters and the depth of the amorphous layer typically formed in with SRIM (Stopping Range of Ions in Matter) simulation program. The values estimated by the simulation showed a very good agreement with the results of the TEM experiments performed on the samples actually treated with ion-implantation. Based on this, the expected value of the relevant properties (layer thickness, amorphous layer depth) can be reliably predicted even in cases when no actual surface treatment is performed, so this estimation method is acceptable for arbitrary implantation parameters. Further validated studies are required for the range of validity of the estimate.
- As a result of the microstructural changes, the mechanical and tribological properties changed. To verify this, I performed flexural strength and wear tests.
  - The results of the abrasion tests showed that in the case of both 0.5 MeV energy carbon and nitrogen implantation, the surface treatment improved the abrasion resistance of the Si_3N_4-based ceramic samples and untreated Si_3N_4 ball friction pair and reduced the value of total volumetric wear.
  - Implantation with different ions and energies in the most cases reduced the flexural strength of Si_3N_4-based ceramics.

Examining the effect of implant energy, I found that the wear of the surface-treated Si_3N_4-based ceramic samples increased with the use of both ions compared to the untreated samples. The wear even more increased with increasing implantation energy, so the observed decrease in wear of tribo-systems containing superimposed specimens can be clearly explained by a decrease in friction pair in contact with the specimen, i.e., tool wear.

The tool wear is clear and proportional to the increase of implant energy, systematic decrease of it was characteristic in case of implantation with C^+ ions, in case of N^+ ion implantation no such clear relationship was found between implant energy and tool wear.

Analyzing the effect of the type of ion used, I showed that the degree of wear is influenced only slightly by the type of ion, even less influenced when N^+ ions were used 0.6 ÷ 14.7% lower depending on the energy.

Overall, in terms of surface treatment parameters, the best results in terms of wear coefficient were provided by N^+ ion implantations, including the lowest energy (0.5 MeV) ion implantation, which improved wear by about 32% compared to the untreated condition and resulted in an improvement of more than 40% in tool wear.

The decrease in the coefficient of friction was generally specific in tribo-systems containing implanted samples, decreased by 7.7 ÷ 10.7% for 1MeV energy N^+ ion implantation, while using C^+ ions it decreased by 12.3% for implantations with 0.5 and and by 10.8% when 1 MeV energy used.

4. NEW SCIENTIFIC RESULTS - THESES

*Based on the results of Si_3N_4-based ceramics made by a given production technology which were **heat-treated in oxygen atmosphere** with a heating rate of 24 °C/min, with a cooling rate of 10 °C/min and with a holding time of 50 h at T = 800, 1000, 1200 and 1400 °C on which with loading force of F = 2, 5 N, with n=30 ÷ 50.000 cyclenumber, with RH = 50% relative humidity, with commercially avaiable Si_3N_4 ball (d = 10 mm), on Δx = 0,2 mm wear track, with ν = 20 Hz frequency and on T = 24 °C temperature fretting type tribological test were made I formulate the following new scientific results:*

T1. Tribological properties (wear coefficient, friction coefficient) of heat-treated Si_3N_4-based ceramics treated in oxygen atmosphere, with a heating rate of 24 °C/min, with a cooling rate of 10 °C/min and with a holding time of 50 h at a temperature of T = 800 °C can be improved (7), (15).

T2. The improvement of tribological behavior of Si_3N_4-based ceramics can be explained by microstructural and related strength changes due to a given volumetric heat-treatment. Effects responsible for change:

- the appearance of the $Y_2Si_2O_7$ phase through the crystalline transformation of the intergranular glass phase,
- and increase in four-point flexural strength (R_{h4}) and Vickers hardness (HV) (8), (16), (17).

T3. The wear coefficient of the tribo-system given by the Si_3N_4 tool and the Si_3N_4-based ceramic specimen during the fretting test under the given conditions:

- in low (RH=3%) humidity medium is the highest,
- in normal (RH=50%) humidity medium is the lowest,
- in high (RH=99.5%) humidity medium is larger compared to the lowest.

The results can be explained by the predominant wear mechanism, which is clearly abrasive wear in dry media, tribochemical wear controlled wear in normal media, abrasive wear combined with tribofilm formation due to limited tribochemical mechanism in high humidity media (18).

Based on the results of Si_3N_4-based ceramics made by a given production technology which were ***ion-implanted*** *with C^+ and N^+ ions, energy of 0.5 MeV, 1 MeV and 2 MeV, energy density of $9.9 \cdot 10^{17}$ ions/cm^2 on which with F = 2, 5 N loading force, with n=30 ÷ 50.000 cyclenumber, with RH=50% relative humidity, with commercially avaiable Si_3N_4 ball (d = 10 mm), on Δx = 0,2 mm wear track, with ν = 20 Hz frequency and on T = 24 °C temperature fretting type tribological tests were made I formulate the following new scientific results:*

T4. As a result of ion-implantation with N^+ ion with an energy of E=0.5 MeV, nano-sized bubbles were formed at the interface of the crystalline particles and an amorphous layer was formed under the surface in the tested Si_3N_4-based ceramics (11), (12), (13).

T5. As a result of ion-implantation with C^+ ion (E=2 MeV energy) nano-crystals were formed with building C^+ ions in the region between the amorphous layer and below the surface (at a depth of h≈2 μm) (9), (10), (17).

T6. The tested Si_3N_4-based ceramics modified by ion-implantation with the parameters detailed above, the tribological properties can be effectively improved as follows:

- Both N^+ and C^+ ion-implantation can be effective in reducing the coefficient of friction, the efficiency of implantation depends on the irradiation energy used;
- The decrease of the wearing rate of the tested Si_3N_4 (Y_2O_3, Al_2O_3) / Si_3N_4 (MgO) tribo-system can be explained by the decrease of the wear of the tool which was in contact with the implanted surface;
- The most favorable results are provided by ion-implantations with 0.5 MeV N^+ ions, which result in an improvement of the wear coefficient of about 32% and the wear of the tools by more than 40% compared to the untreated sample. (12), (14)

5. POSSIBLE APPLICATIONS AND FUTURE RESEARCH

According to the existing results and supplementing them with other material structure studies, volumetric- and surface treatment technologies can be developed, which can be used to optimize commercially available Si_3N_4-based technical ceramics for a given field of application.

Oxidative heat-treatment with appropriate parameters (heating and cooling rate, heat-treatment time, heat-treatment temperature) can improve the strength and wear properties of Si_3N_4-based technical ceramics produced under standard conditions, which is a very cost-effective, fast and simple process for developing these materials. One of the development directions of the research topic may be the heat-treatment of technical ceramics in a nitrogen atmosphere, which modifies the changes in the material structure that take place, thus we can change the mechanical properties of the material.

Ion-implantation which is especially used in semiconductors is a well-known but relatively expensive method for modifying the surface layer of ceramics. It is not a common method to improve the tribological properties of parts subjected to wear, which is currently at the basic research level developed method. Surface treatment of a special, custom-made part is feasible with this technology, thanks to the promising results so far.

A number of modern examination techniques are now available to detect changes in material structure, such as focused ion beam scanning electron microscopy (SEM / FIB), X-ray diffraction (XRD), and small-angle X-ray scattering (SAXS). With the help of these, the structural transformations can be explored, even at the atomic level, that occur as a result of volume and surface treatments.

In practical applications, where the material of the friction elements of the tribo system is similar to the Si_3N_4-based ceramic disk and silicon nitride ball material used in the presented model experiments, the abrasion resistance of the system can be advantageously influenced by the studied volume and surface treatments. As a result of the experimental work, those the technological parameters can be formulated, which can be modified to reduce the total volumetric wear of the investigated types of the tribo systems. Based on the results of the extensive material structure, mechanical and tribological studies presented in the dissertation, the values of the technological parameters promising the most favorable effect in the given application can be estimated in advance.

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