

Structures Formation on the Y–TZP–Al₂O₃ Ceramic Composites Surface

Sergei Kulkov^{1,2,3,a}, Irina Sevostyanova¹, Tatiana Sablina¹, Svetlana Buyakova^{1,2,3}, Artem Pshenichnyy³ and Nickolai Savchenko^{1,2,3}

¹Institute of Strength Physics and Materials Science SB RAS, 2/4, pr. Akademicheskii, Tomsk, 634055, Russia

² Tomsk Polytechnic University, 30, Lenin Avenue, Tomsk, 634050, Russia

³ Tomsk State University, Tomsk, 36, Lenin Avenue, Tomsk, 634050, Russia

^a Corresponding author: kulkov@ms.tsc.ru

Abstract. The paper discusses the structure of Y–TZP–Al₂O₃ ceramics produced from nanopowders and friction surface, wear resistance, friction coefficient of Y–TZP–Al₂O₃ composites rubbed against a steel disk counterface at a pressure of 5 MPa in a range of sliding speeds from 0.2 to 47 m/s. Analysis by X-ray diffraction, scanning electron microscopy showed that the high wear resistance of Y–TZP–Al₂O₃ composites at high sliding speeds is due to high-temperature phase transitions and protective film formation on the friction surface.

1. Introduction

Additive technologies, or technologies of layer-by-layer synthesis is one of the most dynamically developing directions of “digital” production. Zirconia-based ceramics produced from nanopowders is considered one of the most promising materials for additive technologies thanks to its ability to widely change the mechanical properties as a result of structural changes [1]. Particularly promising is the production by means additive technologies of zirconia high speed bearings.

The ZrO₂ tetragonal phase transformation into the monocline phase under the effect of applied stresses is the main toughening mechanism of the Y-TZP-based ceramics [1]. One specific feature of the above materials is that the temperature rise reduces the contribution of the transformation toughening hardening mechanism to their mechanical properties. Especially strongly reduced strength properties in Y-TZP ceramics without additives of other oxides.

The Y-TZP–Al₂O₃ composites are free of this drawback because the Al₂O₃ particles act as a hardening additive. The Y-TZP–Al₂O₃ composites are widely popular because of their relatively high thermal stability [1]. As follows from the data presented in the literature, usually by friction tests in a pair of Y-TZP/ceramic end even at speeds above 1 m/s due to the catastrophic destruction of the friction surface [2,3].

The upper limit of the sliding speeds can be raised to 10–40 m/s if the steel serves as the counterbody [4, 5]. This is caused by the transfer processes of the metal and its oxides to the ceramic surface, producing a so-called transfer layer fulfilling the protective function and promoting the wear resistance of the ceramics at fast sliding speeds [5]. However, the published data on the friction of zirconium-dioxide ceramics are not sufficient to draw conclusions on the processes evolving on the friction surfaces or the wear mechanisms within a broad range of speeds and loads in order to choose the optimum structure of the material and expand the potential of its practical application.



The aim of the current work is to study the structure of friction surface and tribological characteristics of the Y-TZP–Al₂O₃ ceramics in dry friction steel interfaces within a broad range of sliding speeds (between 0.1 and 47 m/s).

2. Materials and experimental procedure

The Zr, Y, and Al aqueous salt solutions served as the original materials for obtaining transformation hardened ceramic composites; the salts were turned into powders in the high-frequency plasma discharge. They had the following composition: 80 wt % ZrO₂ (3 mol % Y₂O₃) + 20 wt % Al₂O₃. X-ray phase analysis revealed that powder contained a mixture of three phases: the ZrO₂ tetragonal and Al₂O₃ γ - and ϵ - modifications. The Y-TZP–Al₂O₃ powders consisted of hollow spheres and their fragments (Figure 1a). The walls of the hollow spheres were polycrystals with a grain size no greater than 20 nm.

The ceramic specimens were compacted in a metallic press mould and then sintered in vacuum at 1700°C for 3 hours. The microstructure of the sintered ceramic shown in Figure 1b.

X-ray phase analysis revealed that after sintering produced tetragonal (T) and cubic (K) zirconium-dioxide phases and α -phases Al₂O₃ in the ceramics. Mechanical properties of sintered composites are shown in Table 1. The sintered ceramics had high hardness, high bending strength and fracture toughness. After sintering, metallographic analysis revealed the residual porosity of the ceramics equal to 5 vol %.

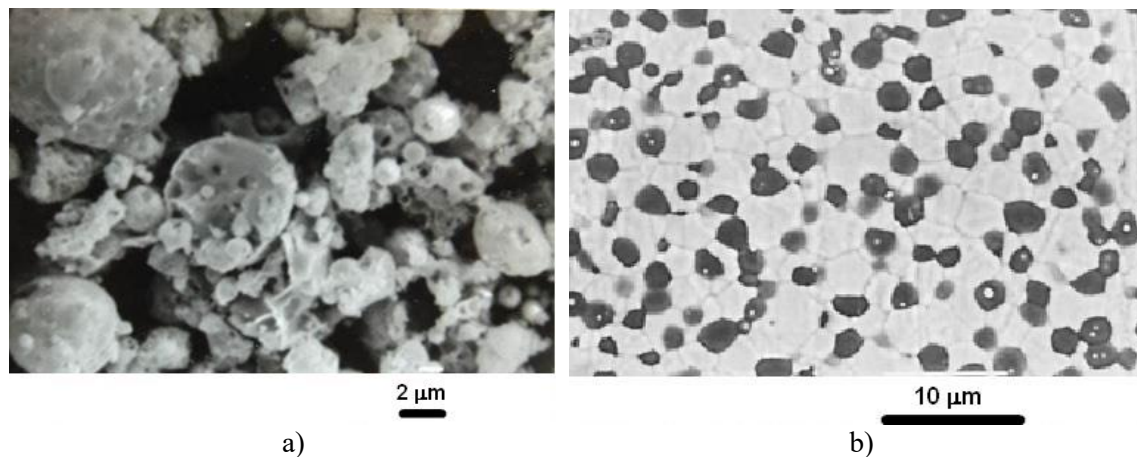


Figure 1. Particle morphology of Y-TZP–Al₂O₃ powders obtained by the decomposition of salts in the plasma of a high frequency discharge (a). Microstructure of Y-TZP–Al₂O₃ specimens after sintering. The surface of the sintered Y-TZP–Al₂O₃ specimen was thermally etched at 1500°C for 10 min (b).

Table 1. - Mechanical properties of the composites

Relative density	Hv, GPa	Bending Strength, MPa	K _{1c} , MPa·m ^{1/2}
0.95	11.4	900	9

The pin-on-disk pairs were tested in dry friction on a UMT-1 unit with the sliding speed increased in steps. The counterbody was a disk from cast steel with the martensite structure (HRC 63–65), M₁₂C carbides, and minimal residual austenite. The specimens of the ceramics in question had rectangular sectional area 60 mm². The test pressure was 5 MPa and the sliding speeds ranged between 0.1 to 47 m/s; the test duration was selected so that the friction path would be 2 km at any speed.

3. Results and discussion

The tribotests carried out in the range of sliding speeds from 0.2 to 47 m/s at a pressure of 5 MPa showed that with increasing the sliding speed wear rate of Y-TZP- Al_2O_3 ceramics first increases and then decreases (Figure 2a). The friction coefficient of the ceramics decreases from 0.5–0.8 at low speeds to 0.15–0.20 at speeds of 25–47 m/s (Figure 2b).

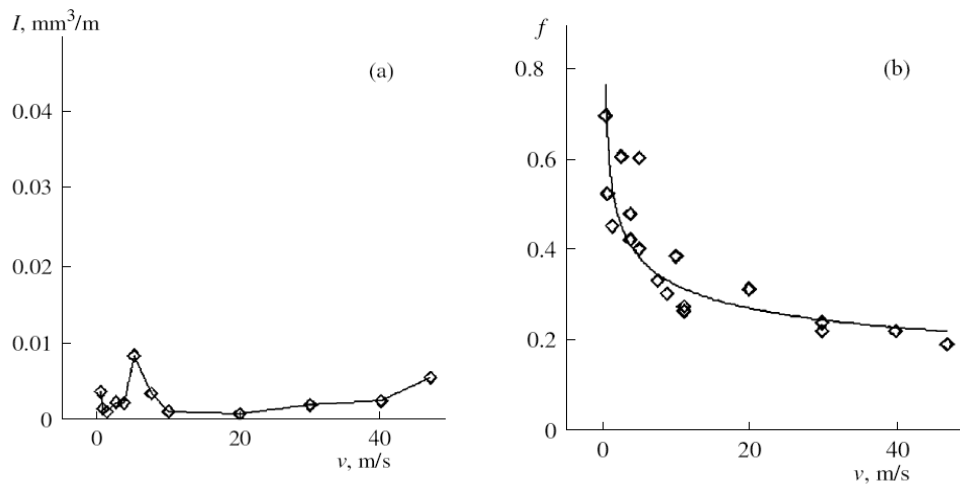


Figure 2. Wear rate (a) and friction coefficient (b) of Y-TZP- Al_2O_3 ceramics as a function of sliding speed on steel.

Scanning-electron and optical microscopy demonstrated that the ceramic surfaces acquired a grooved relief typical of the abrasive wear mechanism after friction within the speed range up to 1 m/s (Figure 3a). The worn surfaces manifested extensive areas with traces of delamination and spalling of the material at speeds >3 m/s (Figure 3b), while the ceramic surfaces had a rather smooth relief at speeds >6 m/s (Figure 3c). It is particularly noteworthy that the Y-TZP- Al_2O_3 friction surfaces contain a cracking mesh that divides them into individual fragments (Figure 3b,c). Measurement of the spacing between the cracks in the sliding direction has revealed that their size distribution has the pattern close to a normal distribution with a distinct maximum.

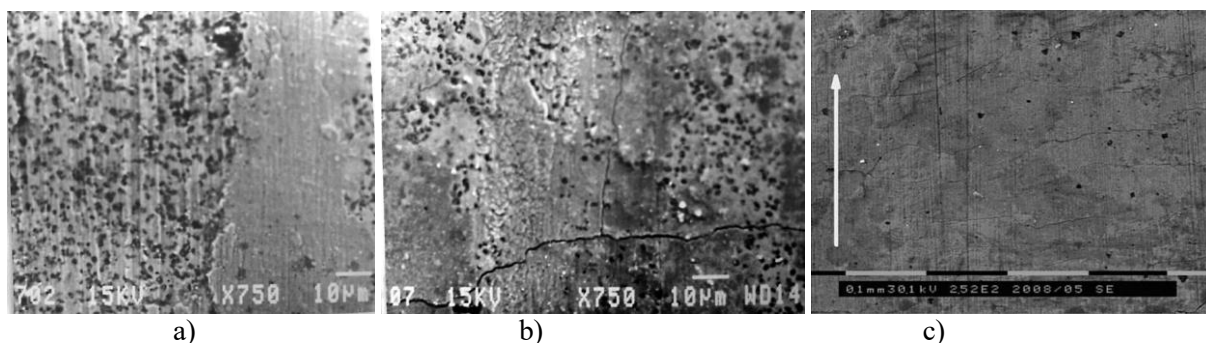


Figure 3. SEM images of friction surface of Y-TZP- Al_2O_3 ceramics after tests at: $v = 0.9$ m/s (a); $v = 4.7$ m/s (b); $v = 47$ m/s (c). Arrow indicate sliding direction.

According to the X-ray phase analysis of the friction surfaces of the Y-TZP- Al_2O_3 ceramics, there are three (monocline, cubic, and tetragonal) crystalline modifications, depending on the sliding speed. It should be noted that the monocline phase does not exist in the original ceramics, while it exists only in the amount of 7–15% by volume after tests at slow sliding speeds, for example up to 11.1 m/s of the Y-TZP- Al_2O_3 ceramics.

The appearance of a monoclinic phase on the friction surface of Y-TZP- Al_2O_3 evidence of martensitic transformation of the metastable tetragonal phase to the stable monoclinic phase. Reduction of the monocline zirconium-dioxide phase as the sliding speed accelerates can be attributed to growth in the contact-zone temperature and reduction of the thermodynamic stimulus for the tetragonal monocline transformation [1]. The concentration of the K-phase typical of the original condition of the ceramics (10 vol. %) remains unchanged in the two materials up to the speed 20 m/s. If the sliding continues to accelerate, its concentration in the specimens grows after the tests to ≈ 15 vol %. The emergence of the cubic phase on the surfaces of the specimens at the maximum sliding speeds is likely due to the fact that a part of the tetragonal phase undergoes diffusion and transforms into the cubic phase. This phase is favored by the elevated temperatures in the contact zone, assessed according to [6] to be on the order of 2000°C at the speed of 10 m/s. Optical and scanning-electron microscopy reveals that the Y-TZP- Al_2O_3 friction surfaces at sliding speed above 6 m/s become relatively evenly covered with a transfer layer. The transfer layer at slow and moderate speeds is distributed extremely irregularly over the surface, leaving large areas completely uncovered.

It has previously been shown [5] that of the subsurface regions of the Y-TZP ceramics reveals a region of material after the tests within the speed range from 0.1 to 4 m/s that underlies the fine layer ($1\text{--}2\ \mu\text{m}$) and has a subsurface layer with the submicrocrystalline structure in which the shapes of the grains are considerably changed in the sliding direction. This zone is thickest (about $10\ \mu\text{m}$) at moderate sliding speeds (≈ 4 m/s), i.e., when the wear rate of the ceramics matters most. The differently shaped grains disappear in the Y-TZP ceramic specimens as soon as the speed exceeds 6 m/s; the grains remain the uniaxial, as they originally were. The differently shaped grains are not observed in the Y-TZP- Al_2O_3 ceramics throughout the whole range of speeds (Figure 4), including at the moderate sliding speeds when the material undergoes intensive wear (Figure 4a).

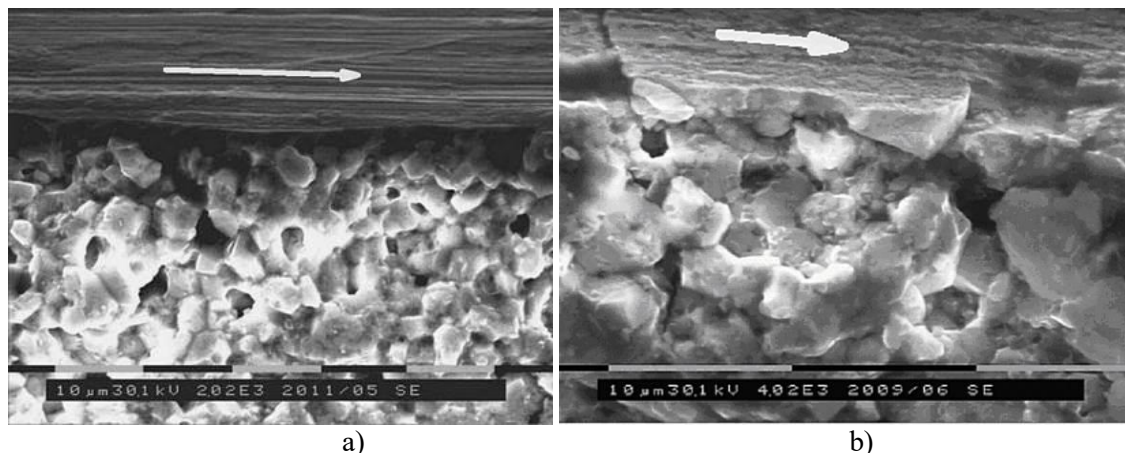


Figure 4. SEM images of structure of subsurface regions of Y-TZP- Al_2O_3 ceramics after friction at $v = 4.3$ m/s (a) and $v = 11.1$ m/s (b).

Arrows indicate sliding direction.

The wear processes evolving at speeds 2–4 m/s correspond to high-temperature adhesive - notwithstanding the common tribological behavior of the Y-TZP [5] and Y-TZP- Al_2O_3 ceramics, the latter material containing the Al_2O_3 particles manifests stronger wear resistance at moderate (3–4 m/s) sliding speeds. In all probability, the structure of this ceramic composite with hardening aluminum-oxide particles has a stronger surface load-bearing capacity during friction when the continuous transfer layer has not yet formed on the ceramic surface.

4. Summary

The accomplished experiments have demonstrated that the Y-TZP- Al_2O_3 ceramic specimens sintered from nanopowders possess high wear resistance at high sliding speeds close to 47 m/s, irrespective of

the elevated temperature in the contact zone resulting in high-temperature phase transitions. A broad range of sliding speeds has been identified (6–47 m/s) within which the ceramics undergo virtually no wear because a quasiliquid film appears and it coats regularly their surface.

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