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TRIBOLOGICAL PROPERTIES OF Al_2O_3/ZrO_2 SINTERED CERAMICS

WŁAŚCIWOŚCI TRIBOLOGICZNE SPIEKÓW CERAMICZNYCH Al_2O_3/ZrO_2

Key words: sliding friction, alumina, zirconia, composites, ball-on-disc, high temperature.

Abstract The paper presents mechanical and tribological properties of Al_2O_3/ZrO_2 composite sinters with different proportions of Al_2O_3 and ZrO_2 phases. These materials are commonly used in dry friction contact due to relatively low manufacturing costs of even complex shapes of products and the possibility of working at elevated temperatures. The tests were carried out by the ball-on-disc method at temperatures of 20, 150, 300, and 500°C. A ball made of Al_2O_3 was used as a counterpart. The results were compared with the following sintered mono-phase materials: Al_2O_3(alumina) and tetragonal yttria-stabilized ZrO_2 polycrystallines (Y-TZP). The tests showed the significantly better properties of composite materials.

Słowa kluczowe: tarcie, zużycie, tlenek glinu, dwutlenek cyrkonu, kompozyty, wysoka temperatura.

Streszczenie W pracy przedstawiono właściwości mechaniczne i tribologiczne spieków kompozytowych Al_2O_3/ZrO_2 o różnej proporcji faz Al_2O_3 i ZrO_2. Są to materiały chętnie stosowane w węzłach tarcia suchego ze względu na relatywnie niskie koszty wytwarzania nawet skomplikowanych kształtów wyrobów i możliwość pracy w podwyższonych temperaturach. Testy przeprowadzono na stanowisku z węzłem tarcia ball-on-disc w temperaturach 20, 150, 300 i 500°C, jako przeciwpróbkę zastosowano kulkę wykonaną z Al_2O_3. Wyniki badań porównywano z wynikami badań spieków wytwarzanych z materiałów jednofazowych: α-Al_2O_3 (korund) i tetragonalnych polikryształów ZrO_2 stabilizowanych tlenkiem itru (tzw. Y-TZP). Badania wykazały znacząco lepsze własności materiałów kompozytowych.

INTRODUCTION

Ceramic materials have a wide range of applications in technical fields, especially when high wear resistance is needed. They can be used in elevated temperatures without any required lubricants **[L. 1]**, e.g., in cutting tool elements **[L. 2]**. Oxide based materials are often used due their relative low costs and simple manufacturing process. In this material group, the most popular are α-alumina (α-Al2O3) and tetragonal zirconia (ZrO2). They exhibit good mechanical properties (hardness, tensile strength, fracture toughness). Their important advantage is that they can be easily obtained in polycrystalline form in any volume ratio of phases **[L. 1]**. Composite materials based on alumina and zirconia could have better mechanical properties than monophasic materials, e.g., hardness and fracture toughness (Tab. 1). Moreover, their wear resistance under various conditions was also presented **[L. 3–7]**. It is considered that the most important factors in improving mechanical properties of the discussed composite materials are the following: fine-grained microstructure developed due to the limitation of grain growth caused by presence of a second phase and residual stresses generated during sintering, caused by different thermal expansion of combined phases **[L. 8–11]**.

The main aim of this work is the analysis of tribological properties of two composite materials based on the ZrO_2–Al_2O_3 system. The first was prepared as zirconia reinforced with alumina particles, and the second was prepared as alumina reinforced with zirconia particles. Additional tests have been done for monophasic alumina and zirconia materials.

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Table 1. Typical values of hardness and fracture toughness K_{Ic} for tested materials
Tabela 1. Typowe wartości twardości i odporności na kruche pękanie K_{Ic} dla badanych materiałów

Material	Hardness [HV]	K_{Ic} [MPa * $m^{0.5}$]
Al_2O_3	16.71	4.34
AZ35	17.26	4.84
ZA35	14.33	4.32
ZrO_2	12.46	6.10

MATERIALS AND METHODS

Four materials were tested:
- Pure α-Al_2O_3 powder (TAIMEI Chemicals, TM-DAR), designated as A;
- Pure yttria-stabilized zirconia 3Y-TZP (TOSOH, TZ-3Y), designated as Z;
- Alumina matrix composite with 35 vol.% zirconia content, designated as AZ35; and,
- Zirconia matrix composite with 35 vol.% alumina content, designated as ZA35.

Tested samples were formed by uniaxial pressing under 50 MPa and then isostatically re-pressed under 300 MPa. The sintering process was conducted at 1400°C and 1500°C for alumina and zirconia composites, respectively. The soaking time was 1 hour in each case. Dimensions of all cubic samples were 20 x 20 x 4 mm. A 6 mm diameter alumina ball was used as a tribological partner. The microstructure and properties of tested materials were described in previous papers **[L. 11, 12]**. Relative densities and mean grain sizes of each phase are summarized in **Table 2**.

Table 2. Relative densities and mean grain sizes of tested materials
Tabrla 2. Gęstości względne i średnia wielkość ziaren badanych materiałów

Material	Relative density [%]	Mean grain size [µm]	
		Al_2O_3	ZrO_2
Al_2O_3	99.28 ±0.05	5.2 ±2.9	-
AZ35	99.04 ±0.30	0.45 ±0.16	0.25 ±0.08
ZA35	99.04 ±0.09	0.36 ±0.18	0.28 ±0.11
ZrO_2	99.96 ±0.01	-	0.34 ±0.12

ISO 20808:2016 standard **[L. 13]** was used to determine both the coefficient of friction and wear rate. Tests were carried out on T-21 High Temperature Testing Machine manufactured by ITE – PIB, Radom, and the scheme of the device is shown in **Figure 1**. It allows one to determine the tribological properties of materials exposed to high temperatures in ball-on-disc geometry and dry sliding contact. The ball mounted in tribometer presses on the sample, fixed in the rotating holder, loaded by normal force (*F*). The 1.5 kW heater inside the thermally isolated chamber provides the possibility of testing at temperatures up to 750ºC. During tests, the friction force between ball and sample, rotating speed and number of revolutions of the sample (*n*), as well as the temperature near sliding contact can be measured.

After each test, the surface topography of the sample and ball was measured using the optical profilometer Profilm 3D manufactured by Filmetrics, USA. The results of these measurements were used to determine the wear rate using Formula 1.

$$w_v = \frac{V}{F_n \cdot s}\left[\frac{mm^3}{N \cdot m}\right] \qquad (1)$$

where V – worn volume of the material,
F_n – normal force,
s – length of wear track.

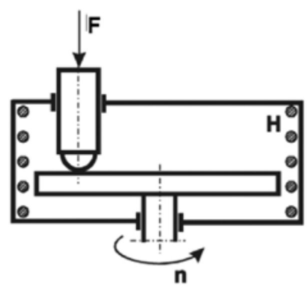

Fig. 1. Scheme of the friction pair in T-21 tribometer
Rys. 1. Schemat węzła tarcia testera T-21

The test parameters were as follows:
- Applied load F_n = 10 N,
- Wear radius 5 mm,
- Revolution speed 120 rpm,
- Total number of revolutions 30000.

For each specimen, tests were performed in temperatures of 20, 150, 300, and 500°C.

The example results of the surface profiles of the ball and the sample measured by optical profilometer are shown in **Figures 2a** and **2b**.

a)

b)

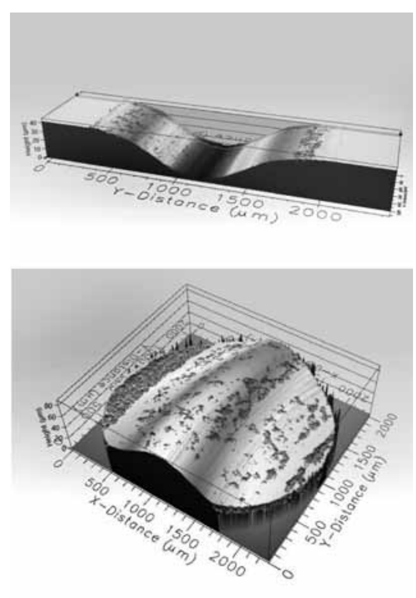

Fig. 2. The surface profiles of: a) sample b) ball
Rys. 2. Profil powierzchni: a) zużycia próbki, b) zużytej części kulki

RESULTS AND DISCUSSION

The results of the tests for all samples conducted in all applied temperatures are shown in **Figure 3**. The AZ35 and ZA35 materials have significantly higher wear resistance at 20°C than the monophasic materials (A and Z), which can be seen in **Figure 3a**. At this temperature, the wear rate of every countersample was almost the same as the corresponding sample (differences in the values at the two temperatures were significant). Results of the tested samples at 150°C are shown in Figure 3b. One can see a significant increase in the wear rate for all samples and countersamples, especially for the pair of alumina-alumina. An increasing tendency of wear rate is also visible in the next temperature of 300°C (**Fig. 3c**). The ZA35 composite had the lowest wear among all tested samples at this temperature. Compared to results obtained at lower temperatures, countersample wear rates were several times higher than the composites AZ35 and ZA35 wear rate. Tests performed at 500°C shown similar results (**Fig. 3d**); however, it is important that the wear rate of the AZ35 and ZA35 composites and corresponding countersamples decreased significantly compared to the results obtained at 300°C. The wear rates of the monophasic zirconia at both 300 and 500°C were similar.

Average values of the friction coefficient obtained during tests are shown in **Figure 4**. For the alumina-alumina pair, the friction coefficient is higher than the other tested materials and increases with temperature. The friction coefficient for the sample made of pure zirconia phase is relatively low and more or less constant over the applied temperatures.

For both of the composite materials, the friction coefficient values changed in a similar manner during the tests. The highest values were obtained at 300°C. At elevated temperatures, the ZA35 composite exhibited lower values of the friction coefficient compared to the AZ35. Especially at the highest temperature, the friction coefficient value for pair alumina-ZA35 was significantly lower (over two times) and the lowest within the tested group of materials.

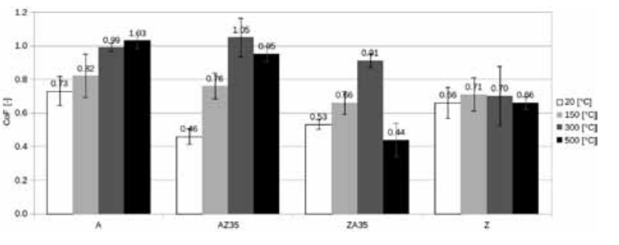

Fig. 4. Mean values of the friction coefficient obtained during the tests

Rys. 4. Średnie wartości współczynników tarcia

Comparisons of the wear rate values obtained during the tests at the four temperatures, separately for all tested sample materials, are shown in **Figure 5**. For the A and Z materials, as expected, the wear rate increases with increasing temperature. Up to 300°C, the composite materials behaved similarly; however, at 500°C, the wear rate values started to decrease significantly.

The wear rate values of countersamples for all applied temperatures, separately for the tested sample materials, are shown in **Figure 6**. For each sample-countersample, a high correlation between changes in value of the friction coefficient and the wear rate of the sample and countersample pair exist, which can be seen in **Figures 4, 5**, and **6**.

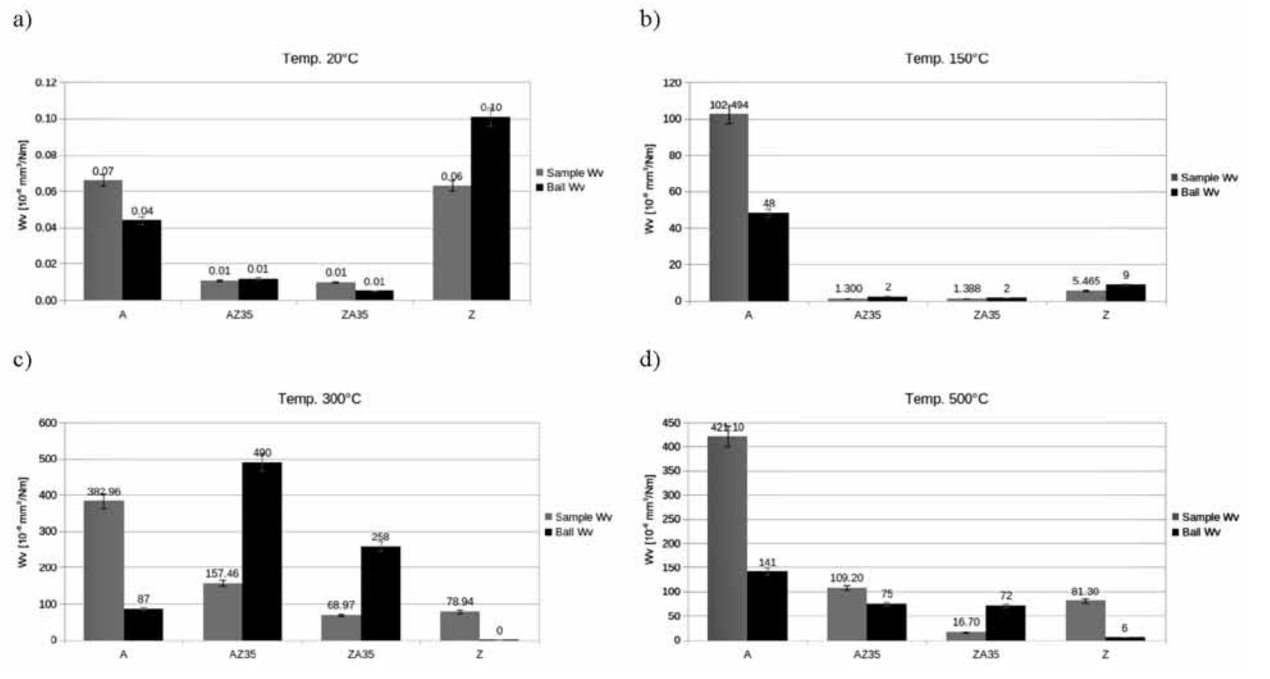

Fig. 3. Wear rate of the sample and countersample tested at temperatures: a) 20°C, b) 150°C, c) 300°C, d) 500°C

Rys. 3. Wartości wskaźników zużycia próbki i przeciwpróbki po badaniu w temperaturach: a) 20°C, b) 150°C, c) 300°C, d) 500°C

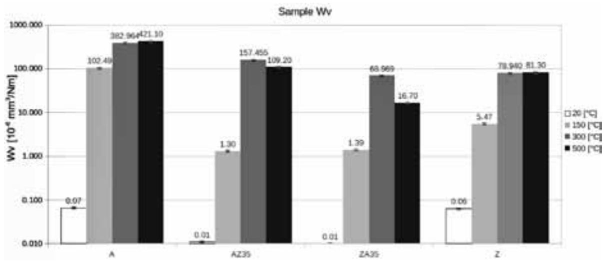

Fig. 5. Values of the wear rate after tests in applied temperatures separately for all tested sample materials (logarithmic scale)

Rys. 5. Zależność wartości wskaźnika zużycia od temperatury dla badanych próbek materiałów (w skali logarytmicznej)

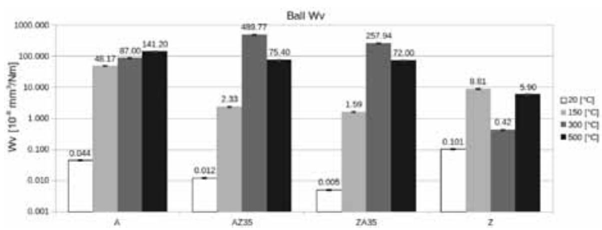

Fig. 6. Wear rate values of countersamples after tests in applied temperatures separately for all tested sample materials (logarithmic scale)

Rys. 6. Zależność wartości wskaźnika zużycia od temperatury dla kulki w połączeniu z badanymi materiałami (w skali logarytmicznej)

The lowest value of the friction coefficient at the elevated temperatures was obtained for the sample made of ZA35 composite. The wear rate of the corresponding ball and the friction coefficient values did not have the highest value. Values of analysed properties for AZ35 composite were worse than for corresponding values for monophasic zirconia at temperatures over 150°C. The worst properties, wear rate of the sample and countersample and the friction coefficient, were found for monophasic alumina.

The improvement of tribological properties of AZ35 and ZA35 composites in contact with the alumina ball is probably caused by the reduction of residual stresses in composite materials caused by the difference in coefficients of thermal expansion **[L. 8]**. Residual stresses in these materials at room temperature could reach even several hundred megapascals, mainly in the area close to grain interfaces **[L. 12]**. At 500°C, this value, due to a thermal expansion, decreases by up to 40%. Additionally, at higher temperatures, the susceptibility of fine-grained ceramic microstructures to pseudoplastic strain increases. This phenomenon is based on the appearance of permanent strains under lower than permissible loads in small areas subjected to loading. These "subcritical deformations," combined with the stable accumulation of micro-deformations, cause a significant increase in the critical deformation value and macroscopically can be seen as the effect of some kind of plastic strain of the ceramic polycrystals **[L. 14, 15]**. The appearance of this phenomenon depends on the temperature of the whole

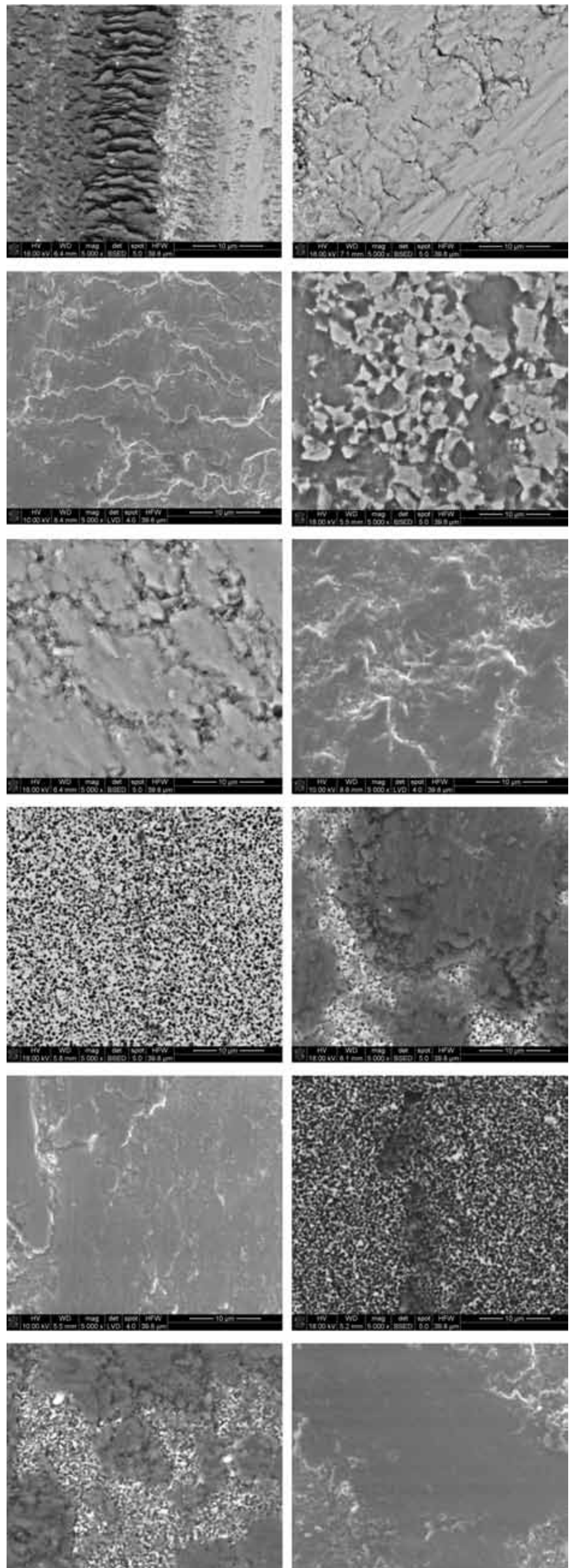

Fig. 7. SEM images of wear tracks: in the sequence form the left top Z, A, AZ35, and ZA35; in rows from the left for 20, 300, and 500°C

Rys. 7. Obrazy SEM śladów tarcia – w wierszach od góry: Z, A, AZ35 i ZA35; w kolumnach od lewej 20, 300 i 500°C sample but also is a result of contact stresses during a friction process that may lead to very high temperatures in micro/nano areas on the surfaces of samples. **Figure 7** shows micrographs of wear tracks of tested samples at 20, 300, and 500°C. The intensification of pseudoplastic deformation in the contact area of contacting elements is clearly visible and is more and more intensive with the increase of temperature. The surface of the samples of all materials is highly deformed after testing at 500°C. However, tests at ambient temperature do not practically cause significant deformations of composites (AZ35 and ZA35), contrary to single-phase materials A and Z. Observations of surfaces after testing at 300°C confirm similar differences – deformations on A and Z surfaces and only local deformations in composites. This can affect the variability of the friction coefficient of composites that is lower at 500°C compared to 300°C. Furthermore, this also corresponds to a lower wear index at 500°C for composites.

SUMMARY

The conducted research program showed the significant rise of the wear index with temperature for all tested ceramic materials. Particularly susceptible to this deterioration was Al_2O_3 aluminium oxide, which also exhibited the highest values of friction coefficients. However, the second single-phase material ZrO_2, maintained a relatively low wear index within tested temperature range, with relatively small changes in the friction coefficient. Compared to single-phase materials, the composites turned out to be materials with significantly lower wear indexes and friction coefficients at room and elevated temperatures. Probably, the reason for the improvement of the tribological properties of AZ35 and ZA35 at elevated temperatures compared to alumina is the reduction of residual stress and pseudoplastic deformations of the sub-micron microstructure of sintered composite. This phenomenon is not effective at 300°C; hence, the coefficient of friction and wear index are higher than at 500°C. An important conclusion arising from the obtained results is the higher wear resistance of AZ35 and ZA35 composites at higher temperatures (300 and 500°C). Whereas, the wear of counter-sample at 300°C is particularly high, i.e. under conditions in which the highest coefficient of friction was measured.

This behaviour can be an important recommendation for the application of such a combination of materials – ZrO_2/Al_2O_3 in which the high temperature resistance and equal wear of both elements of the friction pair are required. At room temperature, a combination of composite/Al_2O_3 is a much better solution.

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