Polish Society of
Composite Materials
Polskie Towarzystwo
Materiałów Kompozytowych

18: 1 (2018) 51-56

COMPOSITES
THEORY AND
PRACTICE

Agnieszka Wojteczko[1]*, Grzegorz Wiązania[2], Marcin Kot[2], Zbigniew Pędzich[1]

[1] AGH - University of Science and Technology, Faculty of Materials Science and Ceramics, Department of Ceramics and Refractory Materials
al. A. Mickiewicza 30, 30-059 Krakow, Poland
[2] AGH - University of Science and Technology, Faculty of Mechanical Engineering and Robotics, Department of Machine Design and Technology
al. A. Mickiewicza 30, 30-059 Krakow, Poland
**Corresponding author. E-mail: agdudek@agh.edu.pl*

Received (Otrzymano) 8.03.2018

FRICTION AND WEAR OF COMPOSITES IN ALUMINA/ZIRCONIA SYSTEM

The paper presents the results of friction and abrasive wear measurements of composites in the α-alumina/tetragonal zirconia system. Two types of materials with mutually interpenetrating phases in a continuous manner were investigated. Pure alumina and zirconia samples were used as the reference materials. Ball-on-disc tests were conducted at 20 and 500°C. An alumina ball was applied as the counterpart. The tests showed that the composites have a significantly reduced wear rate and friction coefficient when compared to monophasic materials. Improvement of the properties was noticeable especially at the elevated temperature.

Keywords: sliding friction, alumina, zirconia, composites, ball-on-disc

ZUŻYCIE ORAZ TARCIE W KOMPOZYTACH Z UKŁADU TLENEK GLINU/DWUTLENEK CYRKONU

Przedstawiono wyniki pomiarów tarcia oraz zużycia ściernego kompozytów z układu tlenek glinu/tetragonalny dwutlenek cyrkonu. Badano dwa typy materiałów z fazami wzajemnie przenikającymi się w sposób ciągły. Próbki z czystego tlenku glinu oraz tlenku cyrkonu zostały użyte w badaniu jako materiały odniesienia. Badanie metodą Ball-on-Disc przeprowadzono w temperaturach 20 oraz 500°C. Kula z tlenku glinu została użyta jako przeciwpróbka. Testy wykazały, że kompozyty mają wyraźnie obniżony współczynnik tarcia oraz podatność na ścieranie w porównaniu do materiałów jednofazowych. Poprawa właściwości była zauważalna szczególnie w podwyższonych temperaturach.

Słowa kluczowe: tarcie ślizgowe, tlenek glinu, dwutlenek cyrkonu, kompozyty, kula-tarcza

INTRODUCTION

In recent years research on the optimal materials for high-temperature applications has inclined towards oxide ceramic materials. There are many particular applications when ceramic components are used as moving or rotating parts as various types of pistons, rotors, pushers or tappets constantly or incidentally working at elevated temperatures (a few hundred degrees Celsius). Ceramic parts are utilized in such applications due to their very good mechanical properties, including wear resistance, considering work at elevated temperatures [1]. Particular attention is paid to α-alumina and tetragonal zirconia materials due to their low manufacturing cost and possibility of easy shaping. What is more, creating their composites is a popular way of improving properties [2-4]. Materials containing tetragonal zirconia have enhanced mechanical properties due to martensitic transformation toughening. However, combining phases with different thermal expansion coefficients results in the distribution of residual stresses [5-8]. In the case of alumina inclusions, the zirconia matrix is under tensile stresses and the reverse in the alumina-zirconia composite. Residual stresses significantly affect the mechanical properties of a material, therefore it is important to optimize the amount of inclusions in matrices. Previous studies have suggested that the most promising composites for wear resistant applications are alumina-zirconia and zirconia-alumina composites with a 35 vol.% minor phase content. This was noticed when the materials were subjected to abrasion under both, dry or wet conditions [9]. Nevertheless, these tests were conducted using SiC powder as an abrasion medium. In order to investigate the sliding friction phenomenon, applying the ball-on-disc method is advisable.

Surface preparation and work conditions have a major influence on friction coefficients. If the load is high and the effective pressure exceeds the tensile strength of the material, it may cause surface grains to fracture or pull out, which may lead to additional abrasion of the material due to these grains or debris [10].

The presented paper shows the results of friction and wear measurements of composites in the α-alumina/ tetragonal zirconia system, where an alumina ball was used as the counterpart. The authors compare the results of friction and wear tests performed at room and elevated temperatures (500°C).

EXPERIMENTAL PROCEDURE

The samples investigated in the research described in this work were prepared using commercially available 3Y-TZP (TOSOH, TZ-3Y) and α-Al_2O_3 powders (TAIMEI Chemicals, TM-DAR). Composite powders were made by wet mechanical mixing of the zirconia and alumina powders (in isopropanol suspension) in an attritor mill using 2 mm zirconia grinding media. Two composite materials were prepared - the first was alumina with a 35 vol.% zirconia content and the second, zirconia with a 35 vol.% alumina content, respectively designated as **AZ35** and **ZA35**. Pure monophasic materials (designated as **Al_2O_3** and **ZrO_2**) were also manufactured using the mentioned commercial powders. Disc-shaped 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 for the alumina and 1500°C for the zirconia and the composites. The soaking time was 1 hour in each case. The densities of the sintered samples were determined using the Archimedes method at 21°C and related to their theoretical values (assuming that $d_{Al2O3} = 3.99$ g/cm^3 and $d_{ZrO2} = 6.10$ g/cm^3). Microstructure and wear trace observations were performed using a Nova Nano SEM 200 scanning electron microscope. The friction coefficient (*CoF*) and wear rate (W_v) values were obtained on the basis of the proper standard [11] using a Tribotester T-21, manufactured in The Institute for Sustainable Technologies in Radom. The tests were conducted at a 120 rpm speed and 10 N load at 20 and 500°C. Room temperature was used as the reference point and the second test temperature was chosen as a relatively high one in order to check the behaviour of the materials in the most extreme conditions possible. During the test 30000 cycles were performed. The radius of the wear trace was 5 mm. Alumina balls (6 mm diameter) were used as the counterparts. Subsequently, the samples were examined with a interferometric profilometer ProFilm3D to estimate the wear rates for the samples and counterparts (W_v) according to the procedure described in [11]. The volume of worn material was determined on the basis of the averaged measurement of the cross-sectional area of the examined wear trace. The sliding distance was calculated on the basis of working time and the set speed. The number of performed measurements for individual material samples was changed from 1 to 3 times. When the measurements were repeated, the differences in the results were less than 10%. The values of residual thermal stresses in the composites caused by the mismatch of coefficients of thermal expansion (*CTE*) of zirconia and alumina were calculated using the FEM procedure described in [5]. The data used for the calculations and results are collected in Table 1. The mean grain size values and their standard deviation for the investigated sintered materials were calculated using the commercial ImageJ program.

TABLE 1. **Data utilized for FEM calculations (on the basis of authors' own measurements) and calculations results**

TABELA 1. **Dane wykorzystane w obliczeniach MES (wg własnych badań) i wyniki tych obliczeń**

Material	*CTE*, α [10^{-6} C^{-1}]	Young's modulus *E* [GPa]	Poisson's ratio ν	Mean value of compressive stresses in alumina phase [MPa]	Mean value of tensile stresses in zirconia phase [MPa]
Al_2O_3	7.9	385	0.250	-	-
ZrO_2	11.0	210	0.210	-	-
AZ35	-	-	-	340	630
ZA35	-	-	-	446	240

RESULTS AND DISCUSSION

The applied production conditions lead to high densification of the obtained specimens (Table 2). In every case, the densification was higher than 99%, as a result, the effect of the porosity on the test outcome could be omitted.

TABLE 2. **Relative densities and mean grain sizes of tested materials**

TABELA 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

The results of wear tests conducted at room temperature are shown in Figures 1 and 2. Generally, the wear rate of composites is distinctly lower than the wear rates of monophasic materials. It is also correlated with a lower coefficient of friction of both composite materials sliding against the alumina ball as the counterpart.

The wear rate of the counterpart was lower than that noticed for the samples in each case when the alumina phase was present in the material (pure alumina or composites). The measured values of the sample/ counterpart wear ratio varied in the range of 0.50÷0.75. When the sliding couple was composed of alumina and zirconia, the wear rate of the alumina counterpart was distinctly higher than the wear of the zirconia sample.

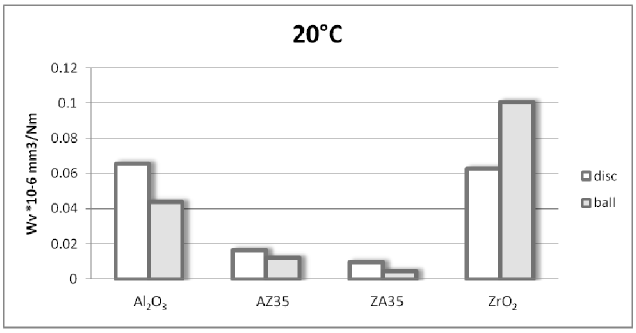

Fig. 1. Wear rate of materials and counterparts tested at room temperature

Rys. 1. Zużycie materiałów i przeciwpróbek badanych w temperaturze pokojowej

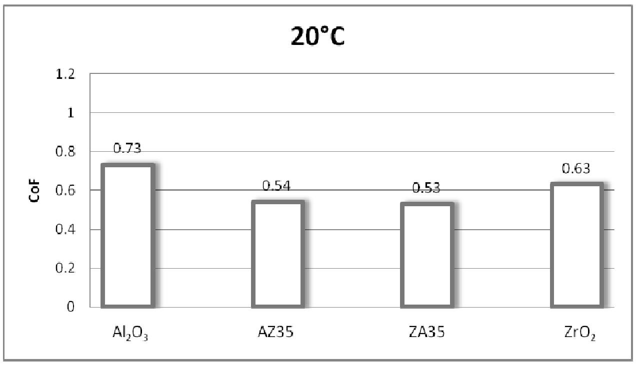

Fig. 2. Coefficient of friction of materials tested at room temperature

Rys. 2. Współczynniki tarcia materiałów badanych w temperaturze pokojowej

The results of the tests conducted at 500°C (Figures 3 and 4) show wear rates higher of a few orders of magnitude when compared to the results achieved at room temperature. Such severe test conditions revealed a distinct difference in the behavior of alumina and zirconia properties. At the elevated temperature the pure alumina material showed a much higher *CoF* value than the pure zirconia material. It is correlated with the much higher wear rate of alumina. Moreover, the **AZ35** composite with the dominant alumina content exhibited a high *CoF* value.

The wear rate of the counterpart was lower than the wear rates of the pure alumina sample and the composite with the higher alumina content (**AZ35**). The measured sample/counterpart wear ratios were 0.30 and 0.70, respectively. A distinct difference in the counterpart wear rate was observed for samples of pure zirconia and the composite with the higher zirconia amount (**ZA35**). When the sliding couple was composed of alumina and zirconia, the wear rate of the alumina counterpart is distinctly lower than the wear of the zirconia sample. Contrary to that, the sliding of **ZA35** and alumina showed the lowest *CoF* and a very low wear rate of the composite sample and relatively high wear rate of the counterpart.

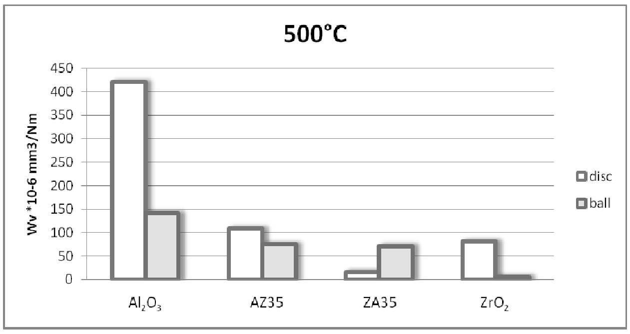

Fig. 3. Wear rate of materials and counterparts tested at 500°C

Rys. 3. Zużycie materiałów i przeciwpróbek badanych w 500°C

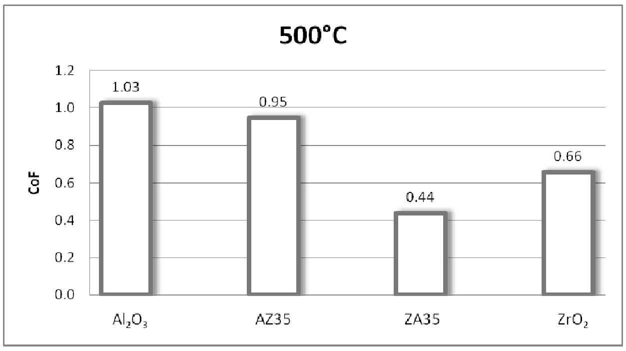

Fig. 4. Coefficient of friction of tested materials at 500°C

Rys. 4. Współczynniki tarcia materiałów badanych w 500°C

The *CoF* values for alumina and zirconia at room temperature did not differ much. The *CoF* values for both composites were practically identical. In such conditions the wear rates of both the composite materials were comparable. At the elevated temperature the *CoF* of alumina increased significantly (almost 50%) and the *CoF* of zirconia stayed at the same level as for room temperature. The wear rates for the monophasic materials at 500°C were significantly different (over 500% difference). This phenomenon was also visible for the composites. The materials with the higher alumina content (**AZ35**) had a higher *CoF* and much higher W_v than the **ZA35** composite. It is worth noting that the **ZA35** material showed the lowest wear rate and corresponding to this fact, the lowest coefficient of friction. The materials with the dominant alumina content (**Al_2O_3** and **AZ35**) showed a relatively stable sample/counterpart wear ratio and the composite (AZ35) has a lower wear rate in both the applied temperature conditions than the alumina. The pure zirconia material and composite with the dominant zirconia content (ZA35) exhibited different behaviors. The wear rate of ZA35 at room and the elevated temperature was the lowest among the investigated materials. However, its cooperation with the alumina counterpart changed significantly depending on the temperature. Similarly, the increase in test temperature caused significant changes in the behavior of the zirconia sample rubbing against alumina.

The comparison of the microstructures of the investigated materials described by the mean grain size (see

Table 2) showed the main difference between the **Al_2O_3** material and the remaining ones containing the zirconia phase. The sintered alumina has a mean grain size on the level of single micrometers and the remaining samples showed a much finer microstructure with submicrometric grains. Such a phenomenon was an effect of using the inexpensive and simple method of sintered bodies manufacturing (pressureless sintering). In such conditions assuring the highest possible density demanded such conditions which caused serious grain growth in the alumina. The **ZrO_2** after applying the above mentioned sintering conditions was very well densified as the mean grain size was not larger than 0.4 micrometers. Sintering of the composites resulted in very small grain sizes due to the very well known phenomenon of stopping the grain boundary movement by the presence of inert particles [12]. The authors confirmed such behavior in the alumina/zirconia system in previous investigations [13]. The microstructure of the alumina ball used as the counterpart was not investigated but most probably it was similar to the microstructure of **Al_2O_3** because it was a commercial product manufactured by means of similar technology.

The large grain size of the **Al_2O_3** sample was a serious cause of the relatively high wear rate. An important wear mechanism which could be observed in Figure 5a was the removal of whole grains or their broken parts. These elements were relatively large and each individual act of material destruction occurred in larger volumes when compared with **ZrO_2** or the composites. The darker material present on the alumina surface and located in the hollows in the **Al_2O_3** sample surface is also alumina removed from the counterpart.

Observations of the wear traces after the test performed at 20°C (Fig. 5) explain the difference between the wear rate of the monophasic materials and composites. The most interesting observed fact is the distinct alumina film detected on the **ZrO_2** sample (Fig. 5d). It confirms the high wear rate of the counterpart measured in this sliding pair. Observations of the **AZ35** and **ZA35** composite surfaces (Fig. 5b and 5c) reveal a complete lack of alumina layer on their surfaces. The *CoF* values registered for these materials were also the lowest (Fig. 2).

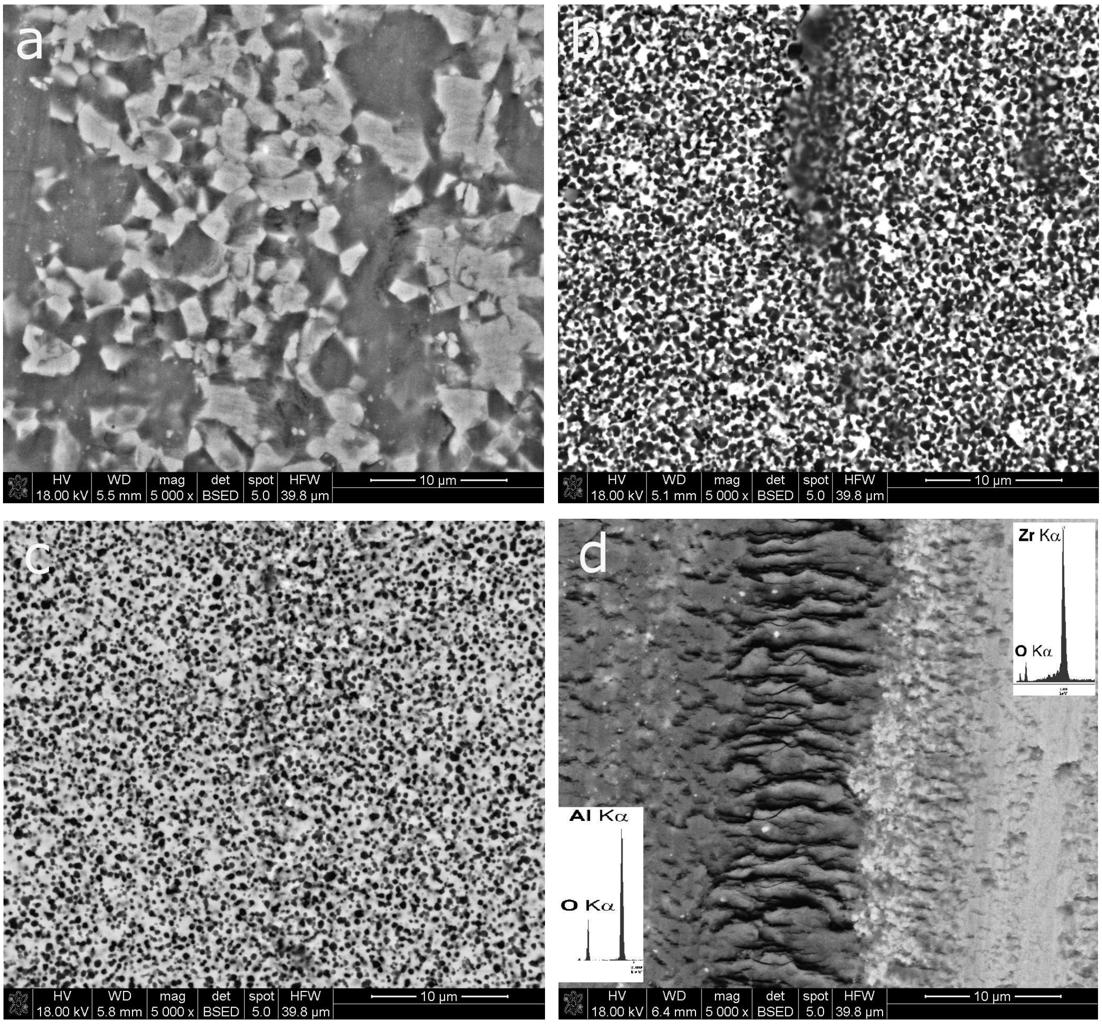

Fig. 5. SEM images of wear trace at 20°C for: a) **Al_2O_3**, b) **AZ35**, c) **ZA35** and d) **ZrO_2**. In Fig. 5d results of elementary EDS analysis is included

Rys. 5. Obrazy SEM śladów wytarcia w 20°C dla: a) **Al_2O_3**, b) **AZ35**, c) **ZA35** oraz d) **ZrO_2**. Na rys. 5d załączono wyniki analizy pierwiastkowej ED

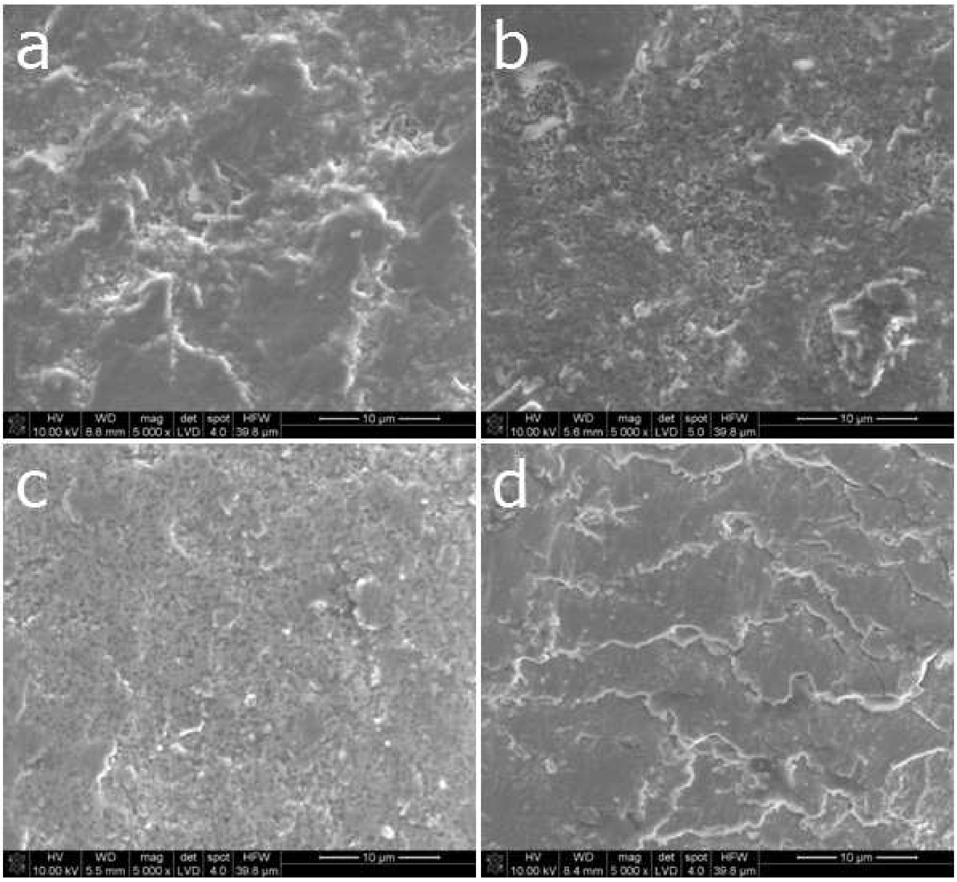

Fig. 6. SEM images of wear trace at 500°C for: a) **Al_2O_3**, b) **AZ35**, c) **ZA35** and d) **ZrO_2**

Rys. 6. Obrazy SEM śladów wytarcia w 500°C dla: a) **Al_2O_3**, b) **AZ35**, c) **ZA35** oraz d) **ZrO_2**

Observations of the wear traces after the test performed at 500°C (Fig. 6) showed a tendency to plastic deformation of the worn surface in each investigated material. This tendency is the strongest for the alumina material but it also occurred in the zirconia one, which is clearly visible in Figure 6. In comparison, the observations of the composite (**AZ35** and **ZA35**) wear traces confirm the much lower intensity of this process, especially in the case of the **ZA35** material, which had the smoothest worn surface, as well as the lowest *CoF* and W_v. Such behavior of the composites is most probably an effect of their finer microstructures and complicated residual stress state which had to influence their mechanical properties. At the elevated temperature the measured **ZrO_2**/counterpart wear ratio was minimal (about 0.07). Such a result indicates that the wear mechanisms had changed significantly. The possibility of the plastic deformation of both oxide phases limited the phenomenon of alumina layer deposition, lowered the *CoF* value and finally limited the wear rate of the counterparts. The observed effects of the wear tests indicated that the wear rate and of *CoF* value of the composites is connected with the mean value of residual stress state in the materials (Table 1). The composite with the better properties (**ZA35**) has a much lower mean value of tensile stresses in the zirconia phase than this provided for the **AZ35** material. This fact is much more important than the difference in the compressive residual stresses value for the alumina phase because degradation of the composite during the wear process is more strongly conditioned by the action of the tensile stresses than compressive ones.

SUMMARY

The results achieved at the elevated temperature clearly confirm the limited possibility of applying materials with a high amount of alumina when they rub against pure alumina counterparts. Oxide ceramics are known for their good mechanical properties and resistance to moderately elevated temperatures, which could be achieved in a relatively easy and inexpensive manufacturing process. The conducted investigations showed that manufacturing composites in the alumina/zirconia system lead to significant improvement of the wear behavior of alumina and zirconia materials at both room and elevated temperatures. However, the level of improvement depends on the constituent phases ratio in the composite material. Utilizing the composite materials with the dominant zirconia content was much more effective in improving the wear properties at the elevated temperature. The investigated **ZA35** composite had the lowest *CoF* and a minimal wear rate. It is worth emphasizing that it was the only case among the investigated materials when the *CoF* measured at 500°C was lower than that measured at 20°C. On the other hand, in this sliding system (**ZA35**/alumina) the wear rate of the counterpart was the highest. This limits the potential application of such a system due to the non-uniform wear of the cooperating elements. The results of the wear test suggest that the composite with the dominant alumina content (**AZ35**) could be the best solution for substituting pure alumina parts. Its wear rate at the elevated temperature is significantly lower than that measured for **Al_2O_3,** and it cooperates with the alumina counterpart in a more stable manner than the composite with the dominant zirconia content.

Acknowledgements

The work was supported by the Polish State Ministry of Science and Higher Education under AGH University work No. 11.11.160.617.

REFERENCES

[1] Liang Y., Dutta S.P., Application trend in advanced ceramic technologies, Technovation 2001, 21, 61-65.

[2] Zhou Z., Wang Z., Yi Y., Lan J., Tribological characteristics in dry friction environment of zirconia-alumina composites with or without layered structure, Ceramics International 2014, 40, 13139-13144.

[3] Nevarez-Rascon A., Aguilar-Elguezabal A., Orrantia E., Bocanegra-Bernal M.H., On the wide range of mechanical properties of ZTA and ATZ based dental ceramic composites by varying the Al_2O_3 and ZrO_2 content, International Journal of Refractory Metals & Hard Materials 2009, 27, 6, 962-970.

[4] Tuan W.H., Chen R.Z., Wang T.C., Cheng C.H., Kuo P.S., Mechanical properties of Al_2O_3/ZrO_2 composites, Journal of the European Ceramic Society 2002, 22, 16, 2827-2833.

[5] Pędzich Z., Grabowski G., Naprężenia resztkowe w kompozytach ziarnistych na osnowach tlenku glinu i tlenku cyrkonu, Kompozyty (Composites) 2006, 6, 2, 76-80.

[6] Grabowski G., Pędzich Z., Residual stresses in particulate composites with alumina and zirconia matrices, Journal of the European Ceramic Society 2007, 27, 2-3, 287-1292.

[7] Pędzich Z., Grabowski G., Wpływ rodzaju wtrąceń na stan naprężeń, wytrzymałość i niezawodność kompozytów ziarnistych na osnowie tlenku glinu, Kompozyty (Composites) 2009, 9, 2, 149-153.

[8] Grabowski G., Lach R., Pędzich Z., Świerczek K., Wojteczko A., Anisotropy of thermal expansion of 3Y-TZP, α-Al_2O_3 and composites from 3Y-TZP/α-Al_2O_3 system, Archives of Civil and Mechanical Engineering 2018, 18, 1, 188-197.

[9] Dudek A., Grabowski G., Lach R., Kot M., Ziąbka M., Wojteczko K., Pędzich Z., The influence of thermal residual stress state on the abrasive wear rates of oxide matrices particulate composites in different work environments, Brittle Matrix Composites 11: Proceedings of the 11th International Symposium on Brittle Matrix Composites (BMC-11), 83-90.

[10] Ortiz-Merino J.L., Todd R.I., Relationship between wear rate, surface pullout and microstructure during abrasive wear of alumina and alumina/SiC nanocomposites, Acta Materialia 2005, 53, 12, 3345-3357.

[11] ISO 20808:2016 Standard, Determination of friction and wear characteristics of monolithic ceramics by ball-on-disc method.

[12] Kang S.-J.L., Sintering - Densification, Grain Growth & Microstructure, Elsevier Butterworth-Heinemann, Oxford-Burlington 2005, 95-96.

[13] Wojteczko A., Lach R., Wojteczko K., Pędzich Z., Investigation of subcritical crack growth phenomenon and estimation of life time of alumina and alumina-zirconia composites with different phase arrangement, Ceramics International 2016, 42, 8, 9438-9442.