

The Influence of the Phase Arrangement of ATZ Composites on Their Wear Rate Under Ball-on-Disc Tests

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ABSTRACT

An alumina-toughened zirconia (ATZ) material, fabricated using a procedure consisting of the common sintering of two different zirconia powders, was tested using the ball-on-disc method in a temperature range between 20 and 400 °C. Tetragonal zirconia balls were used as a counterpart. Three different types of microstructure were designed, one consisting in separated alumina inclusions in zirconia matrix, the second one containing alumina inclusion in the amount close to the percolation point and another one which was a combination of two continuous phases, penetrating the whole volume of the composite. It was detected that at elevated temperatures all materials showed distinct decrease of measured wear rate. Composite with a low alumina content showed minimal wear rate at 300 °C and composites with higher amount of alumina were the most wear resistant at 400 °C. There are some evidences that this minimal wear rate is connected with a pseudoplastic behavior of a layer formed between co-operating elements of tribological pair.

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1. INTRODUCTION

Permanent development of ceramics technologies is a necessary condition for the continuous improvement of many branches of industry [1]. Each type of ceramic product demand a specific technology and has an optimal field of application. Alumina-toughened zirconia (ATZ) materials are relatively well recognized and commercialized due to their low manufacturing costs and good properties, which, in some applications, are much better than the properties

of monophase tetragonal zirconia or alumina products. A very good example are knee or hip-joint ceramics endoprosthesis [2,3], but the mentioned ATZ materials have a significantly wider field of application in the machinery industry. Mentioned composites are very often used as an efficient material for parts of machinery subjected to sliding, rolling, or any other movement usually correlated with mechanical loading and the potential abrasive acting of the environmental elements. The applications of ATZ materials are not only limited

to room temperature, as ATZ materials can withstand elevated temperatures (a few hundred Celsius degrees). Many previous studies [4–10] have elaborated the different aspects of ATZ composite processing, microstructures, and correlations with their final properties. Usually, attention has been focused on the zirconia/alumina ratio, phase composition, and sintering conditions (or methods). The important issue is also residual stress state caused by coefficients of thermal expansion mismatch of both alumina and zirconia phases [11,12]. In alumina/zirconia materials, the zirconia phase is always under tension and alumina under compression. Values of these stresses depends on individual phase content and grains size and shape. They also could be introduced to the composite system by additional processes, e.g., ion exchange [13]. However, composite powder processing and, consequently, sintering procedure could also significantly influence the final phase composition, microstructure and residual stress state. The aim of the presented paper was to use a zirconia matrix in the ATZ composite as a specific material with a fine microstructure and high tendency to the tetragonal to monoclinic phase transformation, which could assure a high level of mechanical and tribological properties [14–18].

2. MATERIALS AND METHODS

Materials investigated during sliding wear tests were three composites from alumina toughened zirconia (ATZ) material's family. Investigated materials were manufactured utilizing a specific idea of zirconia matrix preparation described more detail in [19, 20]. The general idea was to prepare composite mixing two zirconia powders: pure nanometric ZrO_2 one and a solid solution of 4 mol.% Y_2O_3 in ZrO_2 powder. The raw materials used for the preparation of the powders were zirconyl chloride, yttrium chloride, and ammonia (all delivered by Polskie Odczynniki Chemiczne S.A. Gliwice, Poland), which were also used for the precipitation process. Both zirconia powders (the pure one and the 4 mol. % of yttria solid solution) were obtained separately and homogenized by milling in a rotary-vibratory mill for 2 h in a propyl alcohol environment. The weight ratios of both powders were established on a level which assured 3 mol.% nominal content of yttrium oxide in the fabricated material. The final

composition of the material was supplemented with the addition of 2.3 vol.% (1.5 wt.%) of nanometric alumina powder (TM-DAR, Taimicron, Taimei Chemicals Co. Ltd., Tokyo, Japan). The mixing process was performed in a rotary-vibratory mill for 30 min. in a propyl alcohol environment. The final material was a composite with a zirconia matrix, with a small addition of nanometric alumina grains. The material was prepared as described and is herein designated as BC. An others investigated materials, designated as BC10A and BC20A, were a mixture of BC and 10 or 20 vol. % of alumina (TM-DAR, Taimicron), respectively. The BC material was designed as composite with a small amount of isolated alumina particles dispersed in the zirconia matrix. The BC10A material has microstructure with minority phase content close to the percolation point. The BC20A material was designed as a duplex microstructure containing two continuous phases.

Samples in the form of 60×60×6 mm plates were first pressed uniaxially (50 MPa) and consequently isostatically re-pressed at 200 MPa. Then, the samples were sintered at 1450 °C for 2 h.

The densities of the sintered samples were determined using the Archimedes method at 21 °C and related to their theoretical values (assuming that $d_{Al_2O_3} = 3.99 \text{ g/cm}^3$ and $d_{ZrO_2} = 6.10 \text{ g/cm}^3$). Using the rule of mixtures assuming predicted zirconia and alumina content in the composite theoretical densities were calculated as $d_{BC} = 6.01 \text{ g/cm}^3$, $d_{BC10A} = 5.81 \text{ g/cm}^3$ and $d_{BC20A} = 5.61 \text{ g/cm}^3$.

The friction coefficient (CoF) and wear rate values were obtained based on the proper standard [21] using a Tribotester T-21, manufactured in the Institute for Sustainable Technologies (Radom, Poland). Wear rates for flat samples and counterparts (balls) were designated as W_V and W_{VB} , respectively. Normal load F was established on 10 N, the sliding speed was 120 rpm, and number of cycles was 30,000. The applied temperatures ranged from 20 (RT) to 500 °C. The radius of the wear trace was 5 mm. Zirconia balls (5 mm in diameter) were used as the counterparts. In this role, we used a commercially available grinding media manufactured by Tosoh Comp. usually used in attritor-type mills.

After the sliding wear tests, the worn surfaces were examined with an interferometric profilometer ProFilm3D (Milpitas, CA, USA) to estimate the wear rates for the samples and counterparts (W_V , W_{VB}) according to the procedure described by the authors of [22].

The volume of the worn material was determined based on the averaged measurement of the cross-sectional area of the examined wear trace. The sliding distance was calculated based on the working time and set speed. Three individual measurements were performed for each investigated material. The differences in the results were less than 10% for all tested materials.

Coefficient of friction calculation (CoF) values were elaborated by tribometer software as a mean value measured in the determined range. Images of the materials' microstructures were obtained with a scanning microscope Nova NanoSEM 200 (FEI Company).

3. RESULTS AND DISCUSSION

Images in Figure 1 illustrate a typical microstructures of BC (top), BC10A (middle) and BC20A (down) materials. Lighter grains are zirconia one and darker once are alumina. Micrographs confirm very fine microstructures.

Densities of samples measured by Archimedian method showed a similar densification level for all materials. Measures density of BC samples was $5.97 \pm 0.01 \text{ g/cm}^3$ what mean $99.3 \pm 0.02 \%$ of theoretical density. Densities of BC10A and BC20A samples were $5.78 \pm 0.03 \text{ g/cm}^3$ ($99.5 \pm 0.05 \%$) and $5.58 \pm 0.05 \text{ g/cm}^3$ ($99.4 \pm 0.09 \%$), respectively. These data confirm very good level of densification what is very profitable for tribological properties testing.

Figure 2 shows the aggregated wear test results for all samples. It can be observed that each of the tested materials has a temperature at which sliding wear is minimal. For BC material it was $300 \text{ }^\circ\text{C}$; for BC10A and BC20A it was $400 \text{ }^\circ\text{C}$. This means that the most favorable conditions for cooperation with a zirconia counter-sample were slightly different for a material with a minimum content of inclusions (the lower temperature) and for a materials enriched with alumina inclusions (the higher temperature).

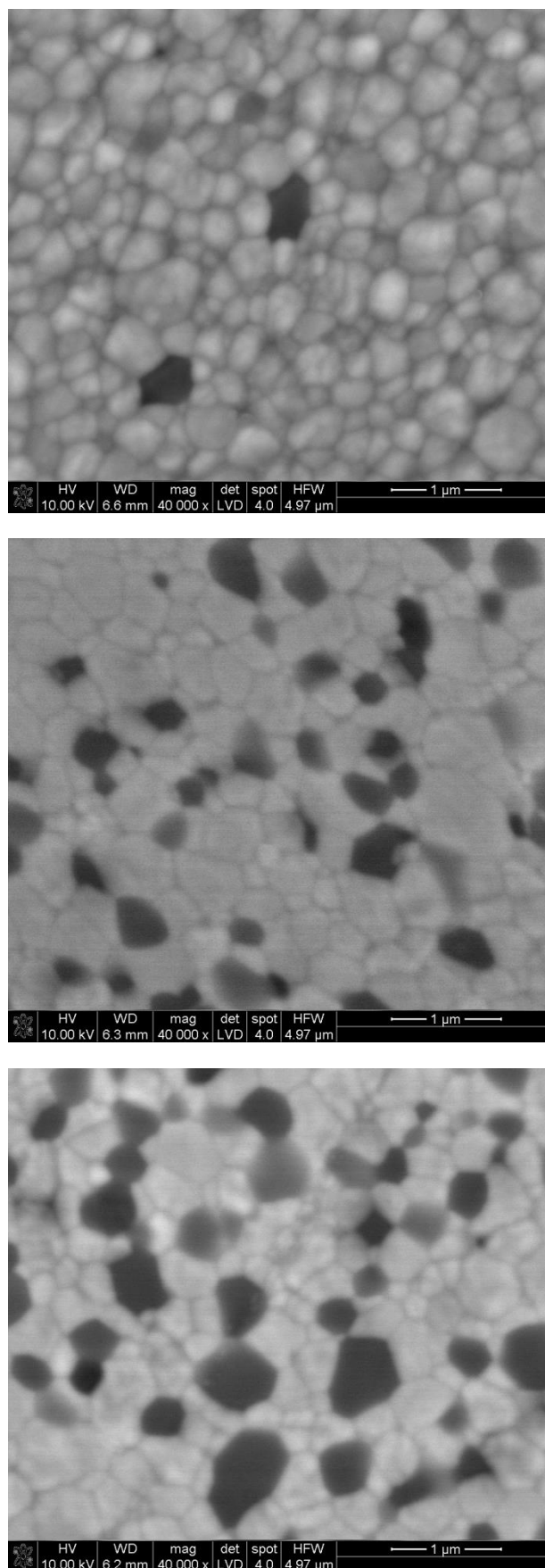


Fig. 1. Microstructures of polished and thermally etched surfaces of investigated composites (top – BC; middle – BC10A; down – BC20A).

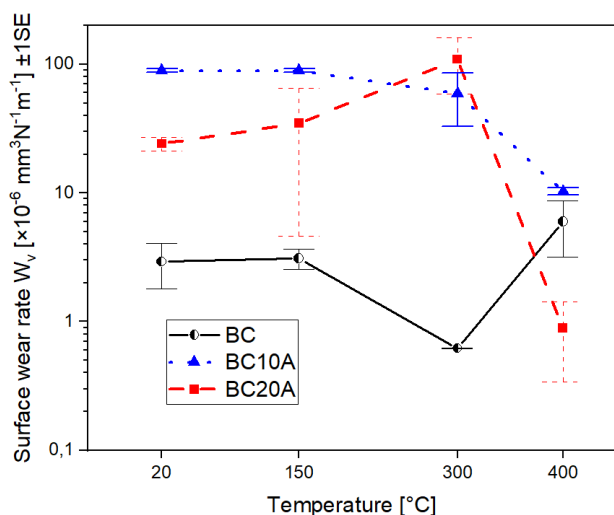


Fig. 2. Sliding wear rates W_V at different temperatures for all investigated materials.

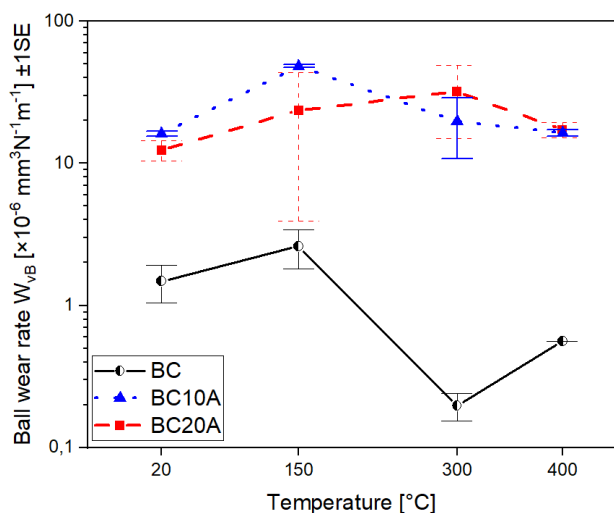


Fig. 3. Sliding wear rates W_{VB} for zirconia counterparts at different temperatures working against all investigated materials.

Figure 3 shows the collected results of wear tests for zirconia counter-samples working with investigated samples. Distinctly the lowest wear rate of counterpart was measured in cooperation with BC material.

The wear rate in this case decreases significantly at higher temperatures (300 and 400 °C). Counter-sample working with BC10A and BC20A were more stable at the whole range of applied temperatures, however their wear rate was distinctly higher than the rate detected for BC containing tribological pair.

Figure 4 illustrate changes of CoF parameter for all investigated cases in the whole temperature range. For all materials tendency

for CoF decrease with work temperature increase was observed. Generally, values of CoF did not differ significantly for individual materials at the same temperature.

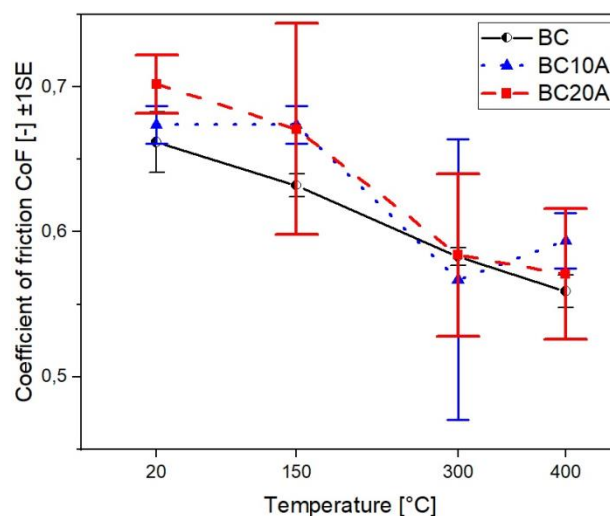


Fig. 4. Coefficients of friction (CoF) at different temperatures of tribological pairs defined by ATZ materials and zirconia ball.

Figures 5 – 8 illustrate details of selected accumulated wear traces of tested materials. These profilometric data were used to calculate the wear rates of the samples.

Figures 5 and 6 illustrate wear trace of BC material after test in the whole temperature range. It is worth to notice that character of the surface is distinctly different for samples tested at lower (room temperature and 150 °C – Fig. 5) and higher (300 and 400 °C - Fig. 6) temperatures. Surfaces after test at higher temperatures were much rough. It is correlated with lower wear rates. Figure 7 illustrates wear traces after test for BC10A material tested at 300 and 400 °C. Also in this case one can observed co-relation between the surface roughness and the wear rate.

Wear trace collected at 300 °C was much smoother than that observed after test at 400 °C. It is also well co-related with the lower wear rate at 400 °C.

Figure 8 illustrates practically identical behavior of the sample BC20A tested at 300 and 400 °C. In all cases, when we observe local material growth on the surface, it takes place for samples operating at temperatures where the lowest wear rates were recorded.

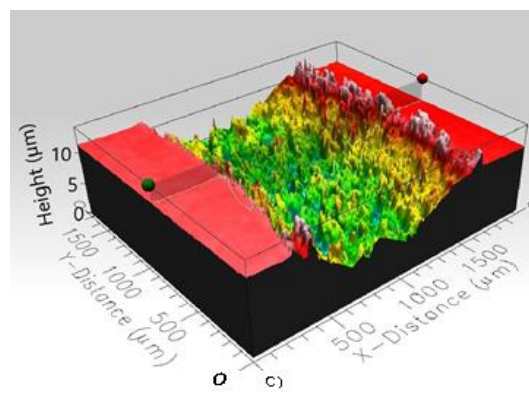
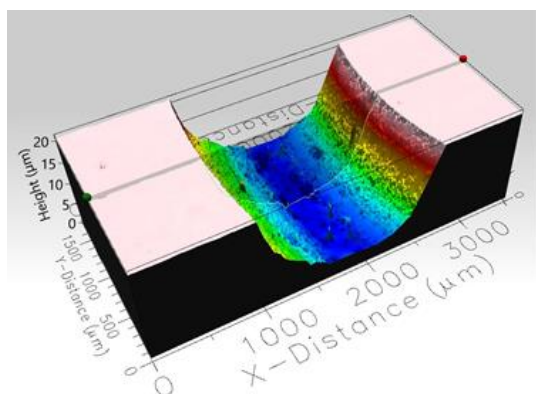
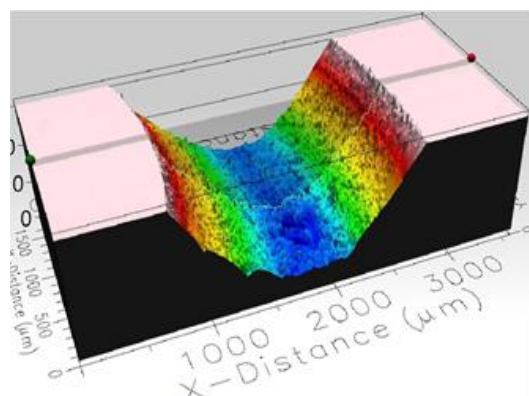
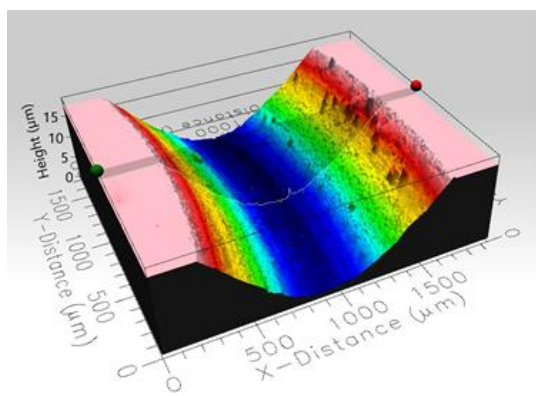


Fig. 5. Three dimensional images of worn surfaces of BC sample after tests performed at room temperature (top) and 150 °C (down).

Fig. 7. Three dimensional images of worn surfaces of BC10A sample after tests performed at 300 °C (top) and 400 °C (down).

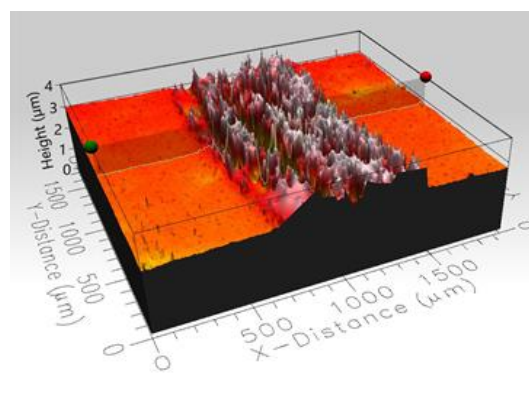
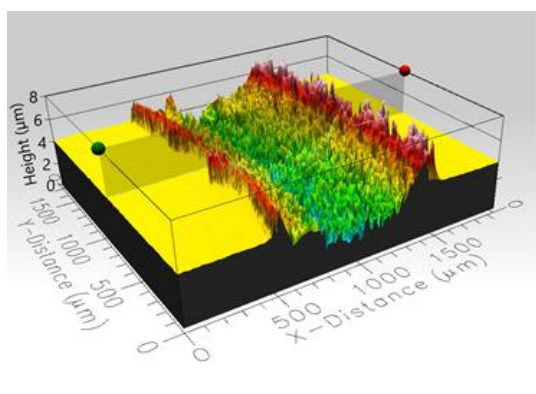
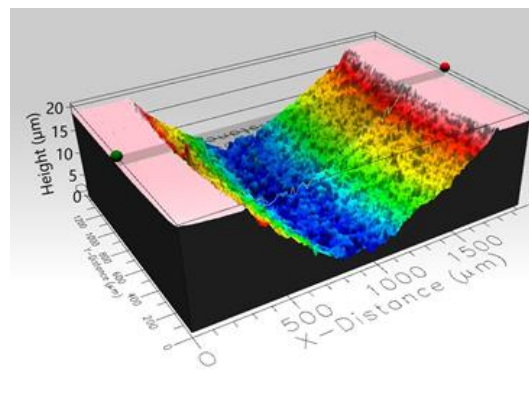
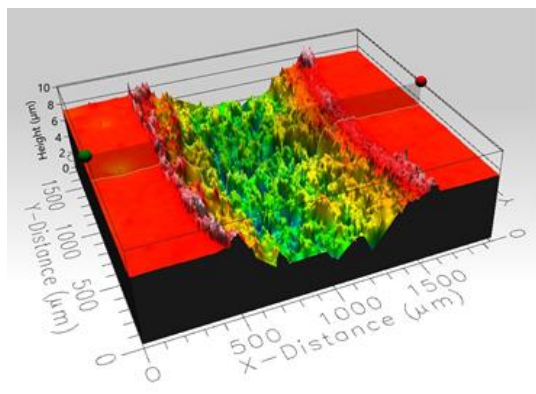


Fig. 6. Three dimensional images of worn surfaces of BC sample after tests performed at 300 °C (top) and 400 °C (down).

Fig. 8. Three dimensional images of worn surfaces of BC20A sample after tests performed at 300 °C (top) and 400 °C (down).

Wear trace collected at 300 °C was much smoother than that observed after test at 400 °C. It is also well co-related with the lower wear rate at 400 °C.

Figure 8 illustrates practically identical behavior of the sample BC20A tested at 300 and 400 °C. In all cases, when we observe local material growth on the surface, it takes place for samples operating at temperatures where the lowest wear rates were recorded.

SEM observations of worn surfaces presented in Figures 9 – 11 confirmed that during tests performed at higher temperatures very fine debris stayed at the surface and act as pseudoplastic layer which may determine the cooperation in the friction pair.

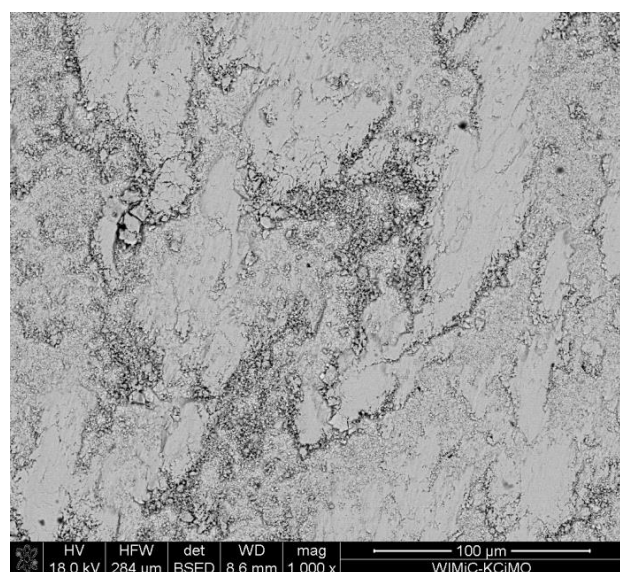
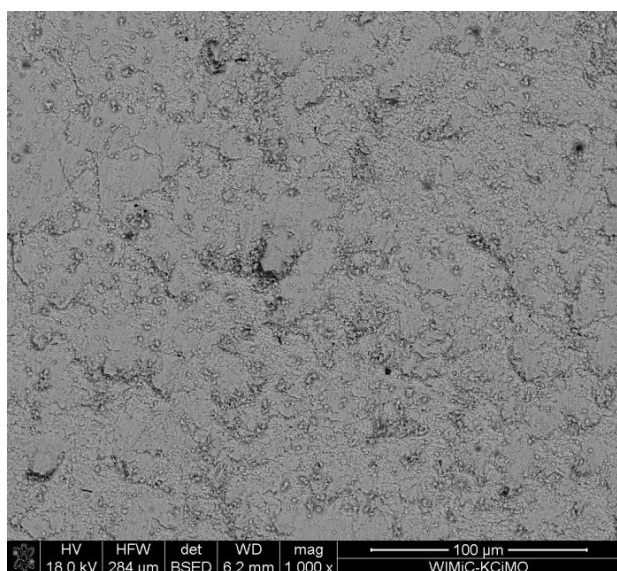


Fig. 9. SEM images of worn surface of BC material tested at 150 °C (top) and 300 °C (down).

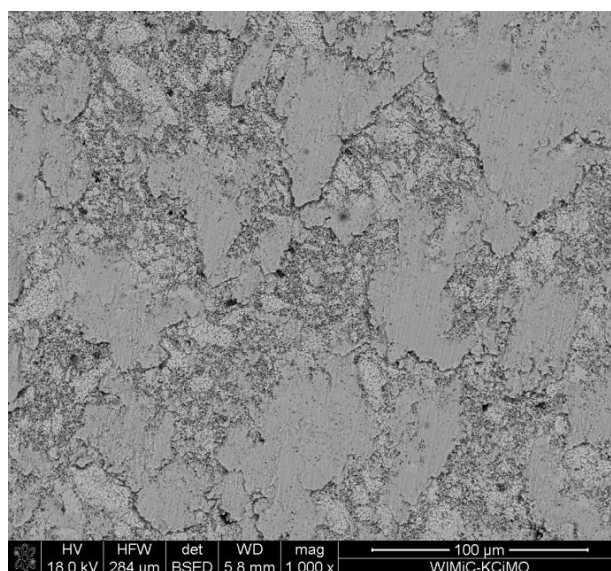
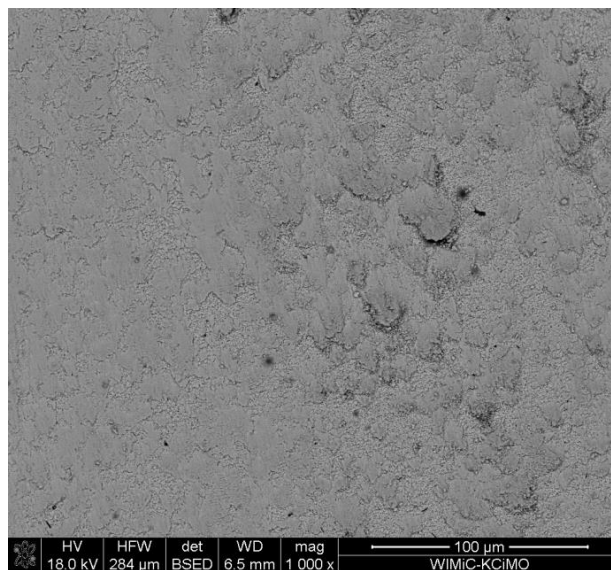


Fig. 10. SEM images of worn surface of BC10A material tested at 150 °C (top) and 400 °C (down).

Figure 9 showed SEM images of worn surface of BC material tested at 150 and 300 °C. It is easy to detect that layer formed on the worn surface at higher temperature has continuous microstructure what influenced sliding process and decreased the wear rate.

Figures 10 and 11 confirmed similar behavior of samples BC10A and BC20A with one restriction that effective continuous layer was formed during test at higher temperature (400 °C). Such conditions were detected as optimal for minimal wear rate of investigated samples. The results obtained during performed sliding wear tests showed that optimal wear rates for all investigated samples were observed at different temperatures of test. Profilometric

and SEM analyses confirmed the presence of distinct surface layer composed of loose grains and grains deformed by intensive local stresses induced by friction forces at optimal temperature condition. This layer composed of very fine elements (due to the small zirconia grains of AZT) had the possibility of a plastic-like deformation, which made co-operation of both ceramic elements of the sliding pair much smoother. Similar effect of low-temperature pseudo-plastic behavior in system containing ceria were reported by Chevalier et al. [22].

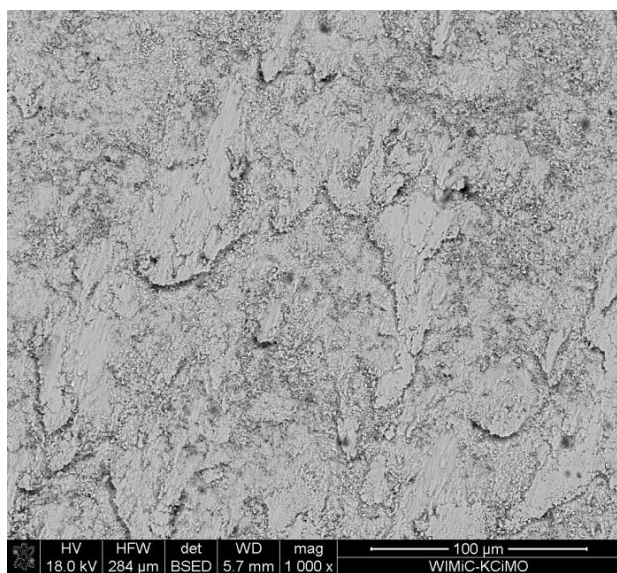
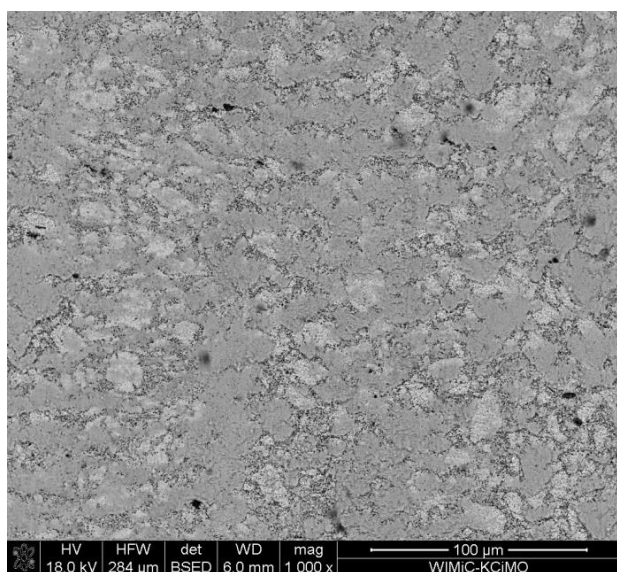


Fig. 11. SEM images of worn surface of BC20A material tested at 150 °C (top) and 400 °C (down).

Our results confirm suggestions that a similar pseudo-plastic effect could be induced locally in yttria-stabilized materials during sliding.

4. CONCLUSIONS

Generally, results of conducted experiments confirm that the ATZ composite materials manufactured by the proposed technique from powders with different chemical composition had a strong potential to be used for reliable machinery parts working in the sliding regime at elevated temperatures. Tribological pairs composed of ATZ composites and tetragonal zirconia counterpart behave in similar way independently of ATZ composite phase arrangement. The particulate composite with isolated alumina particles or composite with duplex microstructure showed wear rate and coefficient of friction decrease with temperature increase. Observed differences concern a temperature value at which minimal wear rate is detected. In a typical particulate composite (BC) temperature of forming of stable layer which supporting sliding co-operation is lower than in materials with higher alumina content (BC10A and BC20A).

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