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COMPOSITES THEORY AND PRACTICE

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ALUMINA/GRAPHENE COMPOSITE IN FRICTION AND WEAR TESTS AT ELEVATED TEMPERATURES

Alumina materials are of great interest due to their high wear resistance, resistance to corrosion, high strength and low friction. However, studies show that a graphene addition can significantly reduce wear and friction. Even a small addition of platelets results in noticeable changes in the material properties. The presented work describes the results of the ball-on-disc test conducted on two friction pairs: alumina-alumina and alumina/graphene composite - alumina. The tests were conducted at four temperatures: 20, 150, 300 and 500°C in air. Surface profile geometry measurements after the test were used to determine the wear rate. Observation of the surface microstructure after friction was carried out using a scanning electron microscope. The tests showed improvement in the wear resistance and a decrease in the friction coefficient of the composite tested at room temperature compared to pure alumina, while the use of elevated temperatures adversely affected the composite.

Keywords: alumina, alumina/graphene composite, wear, friction, ball-on-disc

KOMPOZYT TLENEK GLINU/GRAFEN W TESTACH TARCIA I ZUŻYCIA W PODWYŻSZONYCH TEMPERATURACH

Materiały z tlenku glinu cieszą się dużym zainteresowaniem ze względu na wysoką odporność na zużycie, odporność na korozję, wysoką wytrzymałość i niewielkie tarcie. Badania pokazują jednak, że dodatek grafenu może znacznie zmniejszyć zużycie i tarcie. Nawet niewielki dodatek płytek powoduje zauważalne zmiany właściwości. W pracy przedstawiono wyniki badań w teście Ball-on-Disc przeprowadzonych na dwóch parach ciernych: tlenek glinu-tlenek glinu oraz kompozycie tlenek glinu/grafen- tlenek glinu. Badania przeprowadzono w czterech temperaturach: 20, 150, 300 i 500°C w powietrzu. Pomiary geometrii profili powierzchni po próbach wykorzystano do wyznaczenia wskaźnika zużycia. Obserwację mikrostrukturalną zużytych powierzchni prowadzono za pomocą skaningowego mikroskopu elektronowego. Badania wykazały poprawę odporności na zużycie i zmniejszenie współczynnika tarcia kompozytu badanego w temperaturze pokojowej w porównaniu z czystym tlenkiem glinu, natomiast zastosowanie podwyższonych temperatur miało negatywny wpływ na kompozyt.

Słowa kluczowe: tlenek glinu, kompozyt tlenek glinu/grafen, zużycie, tarcie, kula-tarcza

INTRODUCTION

Ceramics are leading materials in applications where high wear resistance is required. Alumina products are in high demand because of their relatively high strength, abrasion resistance, chemical resistance and relatively low manufacturing costs [1-3]. However, researchers suggest that these favorable properties can be improved even more by adding a small amount of graphene platelets to alumina. Improvement in the electrical and thermal properties compared to the pure alumina phase seems obvious, but in the case of mechanical properties such as fracture toughness and wear resistance improvement can also be expected [4-8]. Improvement in fracture toughness is mainly due to elongation of the crack path, crack branching or crack bridging [6]. Enhancement of the wear resistance is caused primarily by the formation of a graphene tribofilm on the material surface during the wear process [7, 8]. The resulting layer provides so-called self-lubrication. It is, however, important to choose the appropriate amount of the additive since an excessive amount of graphene in the ceramic matrix may cause significant deterioration of the mechanical properties.

In our previous work, the tribological properties of pure alumina were tested at room and an elevated temperature (500°C) using the ball-on-disc testing standard [9]. The surface layer after friction on alumina was observed. Nevertheless, in this material the coefficient of friction and wear were relatively high. Improvement in the wear resistance was achieved by manufacturing alumina-zirconia composites. The aim of the study was to investigate the coefficient of friction and wear of alumina materials containing graphene nanoplatelets. The test was performed on sintered materials against an alumina ball at four temperatures: 20, 150, 300 and 500°C. While the alumina/graphene testing at room temperature was expected to give better results than pure alumina phase, higher temperature testing deserved more attention.

EXPERIMENTAL PROCEDURE

The samples tested in the research presented in this paper were manufactured from commercial α-alumina powder (TAIMEI Chemicals, TM-DAR) and 0.25 wt.% 12 nm graphene platelets, Graphene Supermarket (grade AO-2, particles of a 5 μm lateral size and average flake thickness of 8 nm). Pure alumina samples were obtained using the procedure described in [9]. The composite materials were homogenized in isopropanol using a mechanical stirrer and ultrasound simultaneously. The dried powder was hot-pressed at 1450°C for 1 hour under 25 MPa pressure in an argon atmosphere.

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 standard [10] using a Tribotester T-21, manufactured in The Institute for Sustainable Technologies in Radom. The tests were conducted at the speed of 120 rpm and with a 10 N load at 20, 150, 300 and 500°C. During the test, 30000 cycles were performed. The radius of the wear trace was 5 mm. Alumina balls 6 mm in diameter were used as the counterparts. Then, the samples were examined with a interferometric profilometer ProFilm3D to calculate the wear rates for the samples and the counterparts (W_v) according to the procedure described in [10] using the equation:

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

where: V - volume of material worn in the friction process, F_n - contact force between ball and sample surface, s - friction path.

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 the working time and set speed.

RESULTS AND DISCUSSION

The preparation procedures resulted in a density of 3.96 g/cm^3 for alumina and alumina/graphene composite density of 3.92 g/cm^3, representing 99.28% and 98.99% densification, respectively. The numerical data collected during the wear tests at the given temperatures are collected in Table 1 and then visualized in Figure 1. The results showed a reduction in wear in the composite compared to alumina at 20 and 150°C. In the case of the two higher temperatures, the addition of graphene did not show any improvement in the wear resistance. What is more, at 500°C the wear of the composite material was much higher.

TABLE 1. **Wear rate of alumina and alumina/graphene composite sample and counterpart at indicated test temperatures**

TABELA 1. **Zużycie ścierne próbki z tlenku glinu i kompozytu tlenek glinu/grafen oraz przeciwpróbki we wskazanych temperaturach testu**

Material	**Wear rate [10^{-6}mm^3/(N*m)]**	***W_V***			
	Temperature [°C]	20	150	300	500
Al_2O_3	sample	0.066	102.494	382.960	421.100
	alumina ball	0.044	48.171	87.005	141.200
Al_2O_3-graphene	sample	0.056	53.925	384.527	953.616
	alumina ball	0.009	0.124	8.575	25.449

The alumina counterparts were also subjected to wear which increased with the temperature, and friction against the pure alumina sample caused greater wear in every case. It is worth underlining that in the whole range of applied test temperatures, the counterpart wear rate was distinctly less than the sample wear rate. Detailed comparison of the wear rates in the pure alumina system showed that at room temperature the wear rates of the sample and counterpart were similar. With a temperature increase the wear rate of the samples was more than the counterpart wear rate. Introducing graphene platelets into the alumina matrix changed this behavior. At room temperature the wear rate of the sample with graphen is comparable to the pure alumina sample, but the wear rate of the counterpart sliding against the alumina/graphen sample was about 6 times lower. At 150°C this phenomenon is much more distinct, the difference between the counterpart wear rates, sliding against the pure alumina and composite, was a few hundred times. At higher temperatures the counterpart wear rate compared to the sample wear rate was a few times lower again. These results suggested the presence of a local minimum wear rate connected with moderate temperatures higher than room temperature. At 300°C and higher this beneficial phenomenon disappeared.

It is interesting that the mentioned behavior could not be explained by the coefficient of friction changes.

In the system with the composite sample, the CoF was stable at the level of 0.9 from 150 to 500°C (Fig. 2). Generally, the coefficient of friction measurement results corresponded well with the results of wear at room temperature and at the higher applied temperatures (300, 500°C). The CoF measurement results obtained at 150°C were a little bit different. The coefficient of friction in the system containing the composite sample was higher than in the pure alumina system. Similarly, the relative wear rates (sample/counterpart ratio) were the most favorable at this temperature. Moreover, the wear rate sample with graphene was the most favorable when compared with the pure alumina sample wear rate. This suggests that the CoF value was not the decisive factor influencing the wear rate.

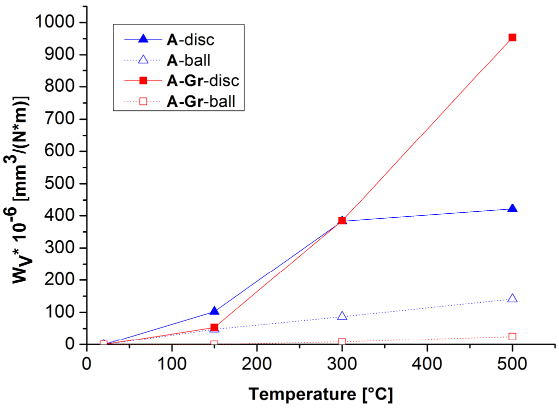

Fig. 1. Wear of examined materials vs. temperature

Rys. 1. Zależność zużycia badanych materiałów od temperatury

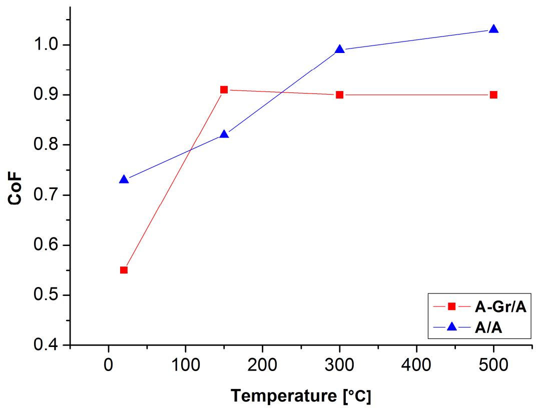

Fig. 2. Relation of coefficient of friction of tested tribological associations with temperatures in friction zone

Rys. 2. Zależność współczynnika tarcia badanych skojarzeń od temperatury w strefie tarcia

In order to illustrate the occurring phenomena, SEM micrographs for the two extreme temperatures of 20 (Figs. 3-6) and 500 °C (Figs. 7 and 8) are presented. At 20°C the main difference between the presented materials was that two processes occurred during the test on the pure alumina sample surface. The first was destructive crushing of the alumina grains and the second one was filling of the created surface irregularities with small alumina debris (Figs. 3 and 4).

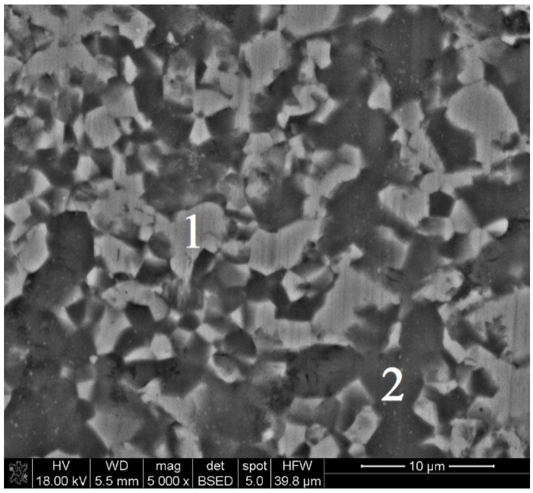

Fig. 3. SEM micrograph of wear trace in alumina sample at 20°C

Rys. 3. Obraz SEM śladu wytarcia próbki z tlenku glinu w 20°C

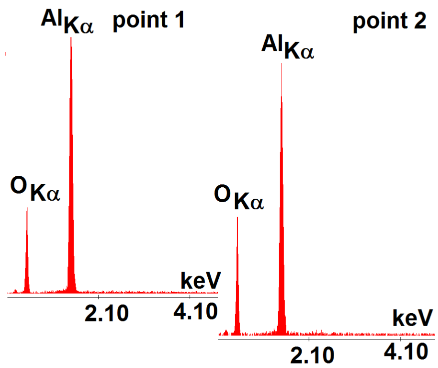

Fig. 4. Result of qualitative EDS analyses in areas indicated in Figure 3

Rys. 4. Wynik analiz jakościowych EDS w punktach zaznaczonych na rysunku 3

The sample containing graphene platelets was covered with a surface layer which was cracked and in many observed cracks graphene particles were present (Figs. 5 and 6). The resulting layer reduced the friction and thus the material wear rate. In the layer cracks, graphene platelets could be observed.

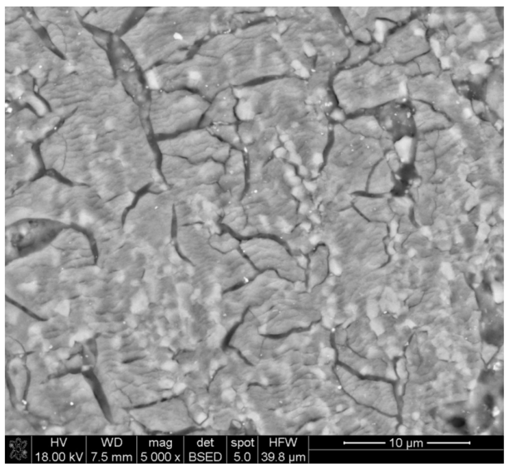

Fig. 5. SEM micrograph of wear trace in alumina/graphene composite sample at 20°C

Rys. 5. Obraz SEM śladu wytarcia próbki kompozytowej tlenek glinu/grafen w temperaturze 20°C

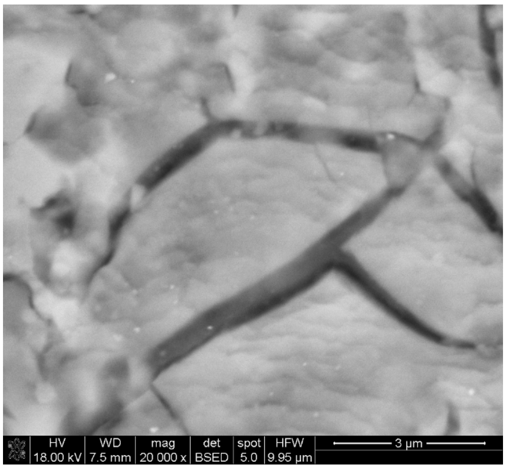

Fig. 6. SEM micrograph of wear trace in alumina/graphene composite sample at 20°C with distinctly visible graphene platelet

Rys. 6. Obraz SEM śladu wytarcia próbki kompozytowej tlenek glinu/grafen w temperaturze 20°C z wyraźnie widocznym płatkiem grafenowym

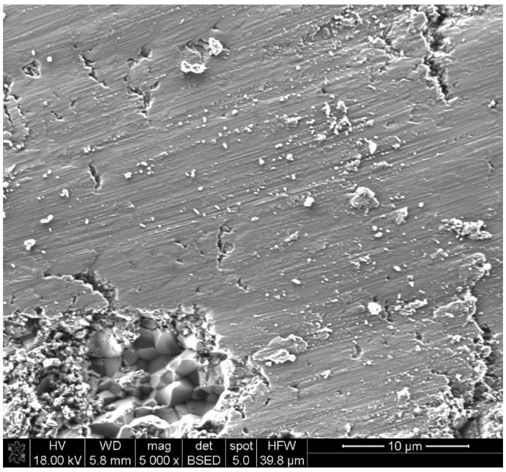

Fig. 7. SEM micrograph of wear trace in alumina sample at 500°C

Rys. 7. Obraz SEM śladu wytarcia próbki z tlenku glinu w temperaturze 500°C

At the highest temperatures graphene could no longer be observed in the voids, which significantly deteriorated the material tribological properties (Fig. 8). After graphene burnout, the surface of the material had many empty cracks, which resulted in a higher wear rate of the composite.

The wear rate results (Table 1) suggest that the applied temperature of 150°C is the optimal one (among the temperatures chosen for the tests) from the wear rate point of view. At this temperature, both the sample and counterpart wear rates showed the highest level of improvement when compared to the pure alumina system. The observed effect was the most probably a result of the synergic self-lubrication effect improved with graphene platelets and the plastic deformation of alumina (Fig. 9) which could act more effectively at slightly elevated temperatures (higher than 20 less than 300°C). At higher temperatures, degradation of the graphene particles limited their beneficial effect. Of course 150°C could not be considered as the optimal temperature for the tribological behaviour of the investigated system. The presented experiment was not an optimisation process. The precise statement of the most beneficial working temperature has to be selected in additional experiments.

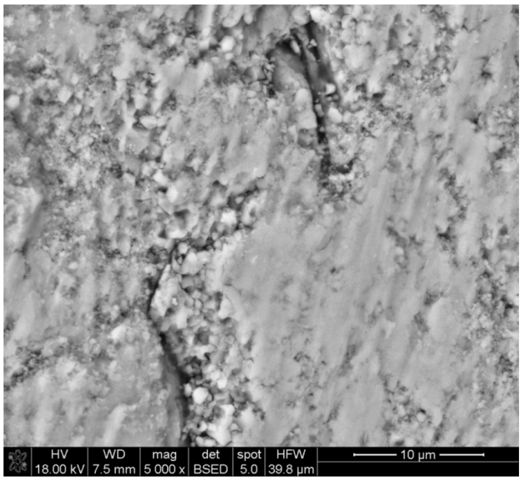

Fig. 8. SEM micrograph of wear trace in alumina/graphene composite sample at 500°C

Rys. 8. Obraz SEM śladu wytarcia próbki kompozytowej tlenek glinu/grafen w temperaturze 500°C

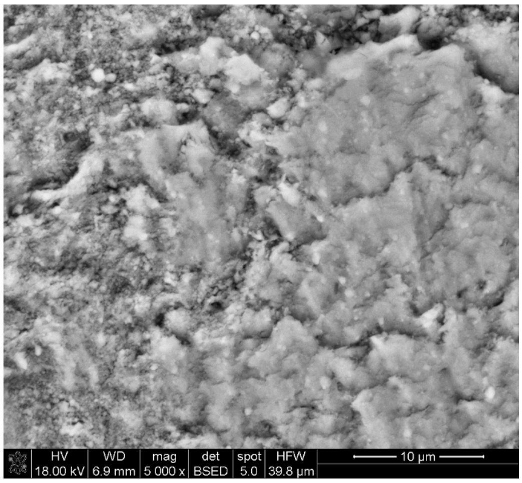

Fig. 9. SEM micrograph of wear trace in alumina/graphene composite sample at 150°C

Rys. 9. Obraz SEM śladu wytarcia próbki kompozytowej tlenek glinu/grafen w temperaturze 150°C

SUMMARY

The growing interest in graphene-enriched ceramic matrix composites is justified by the results of numerous studies carried out at room temperature. The presented work also confirmed that the addition of graphene to alumina material reduces both the friction coefficient and the material wear. At 20°C a layer, which could serve as a solid lubricant, was formed. For pure alumina the type of plastic deformation with layer creation occurred only at elevated temperatures. Nonetheless, the character of the layer was different than in the composite and it was rougher. However, the use of alumina/graphene materials at elevated temperatures in air does not lead to any improvement in the tribological properties in relation to the pure alumina phase. This is caused by burning graphene from the material and leaving voids that reduce the resistance of the material to abrasion. In this case, the use of a protective atmosphere would be reasonable. The performed experiments showed that at the temperature of 150°C, even in air, the synergic self-lubrication effect supported by graphene platelets and plastic deformation of alumina resulted in a very low wear rate of the alumina/graphene material sliding against the alumina counterpart.

Acknowledgements

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