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COMPOSITES THEORY AND PRACTICE

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CORRELATIONS BETWEEN STEREOLOGICAL PARAMETERS OF CARBON COMPONENT AND TRIBOLOGICAL PROPERTIES OF HETEROPHASE COMPOSITES Al-Al_2O_3+C

This paper is an attempt to describe the influence of the stereological properties of the reinforcement on the final material properties. The research was carried out on Al based composites with a heterogeneous reinforcement of Al_2O_3 and C. Various sizes of the carbon particle component (< 40, 80÷120, 160÷200 and < 200 μm) were applied for material manufacturing. The materials were obtained by high energy ball milling and subsequent hot pressing processes. As a result of the milling stage, a reduction in the compound size was observed. First, the real size of the carbon component, the real amount of carbon on the material surface and other properties were measured by quantitative metallography methods. Then, the correlations between the obtained stereological parameters and the tribological properties were checked. The analysis revealed that the average size of a single carbon particle and the distance between adjacent particles are the most important factors for the tribological properties of Al-Al_2O_3+C composite design. The larger the size of particles and the greater the distance between the particles resulted in increasing the friction coefficient value. It is related to the homogeneous distribution of the reinforcing component. However, the most surprising effect was discovered during analysis of the areal fraction of C particles. There were no clear correlations between the amount of C particles and the tribological properties. The conducted research revealed which of the analysed parameters are the most valuable for material design and predicting the final properties.

Keywords: **stereological parameters, tribological properties, aluminum matrix composite, carbon component, high energy milling**

WPŁYW PARAMETRÓW STEREOLOGICZNYCH KOMPONENTU WĘGLOWEGO NA WŁAŚCIWOŚCI TRIBOLOGICZNE HETEROFAZOWYCH KOMPOZYTÓW Al-Al_2O_3+C

W pracy dokonano oceny wpływu wielkości cząstek umacniających na właściwości tribologiczne. Badania zostały przeprowadzone na kompozytach o osnowie Al umacnianych heterofazowymi cząstkami Al_2O_3 oraz C. Materiały zostały wytworzone metodami wysokoenergetycznego mielenia w młynach planetarnych oraz prasowania na gorąco, stosując różne frakcje komponentu węglowego (< 40, 80÷120, 160÷200 oraz < 200 μm). W wyniku mielenia wysokoenergetycznego stwierdzono redukcję wielkości cząstek węglowych. W pierwszym etapie badań dokonano oceny podstawowych parametrów stereologicznych wytworzonych materiałów, takich jak udział C na powierzchni materiału czy średnia wielkość cząstki węgla. Następnie dokonano oceny zależności pomiędzy rzeczywistymi wartościami parametrów stereologicznych a właściwościami tribologicznymi badanych materiałów. Przeprowadzona analiza wykazała, że spośród najważniejszych czynników mających wpływ na właściwości tribologiczne kompozytów Al-Al_2O_3+C należy wymienić średnią wielkość pojedynczej cząstki C oraz odległość między sąsiednimi cząstkami węgla. Zwiększenie średniej wielkości cząstki, jak również jej odległości pomiędzy cząstkami skutkuje wzrostem wartości współczynnika tarcia. Jest to związane z homogenicznym rozmieszczeniem fazy węglowej w objętości kompozytu. Jednakże, najbardziej zaskakujące rezultaty zostały zaobserwowane podczas analizy udziału węgla na powierzchni kompozytu. Przeprowadzona ocena stereologiczna mikrostruktury kompozytu wykazała, które z parametrów mogą być uwzględnione i wykorzystane do określenia parametrów technologii wytwarzania oraz właściwości tribologicznych kompozytu.

Słowa kluczowe: **parametry stereologiczne, właściwości tribologiczne, kompozyty z osnową aluminiową, komponent węglowy, mielenie wysokoenergetyczne**

INTRODUCTION

Metal matrix composites with a ceramic reinforcement are commonly used in many fields of industry. Due to the high thermal conductivity of the matrix and high mechanical properties of the ceramics, those materials can be applied in demanding conditions. Moreover, the synergic action between the components has led to fulfilling the requirements of highly developed industries like the automotive or aerospace industries [1]. According to literature data analysis, the final properties of composite materials depend on many factors.

Among the most important points, the presence of reinforcement particles [2], proper reinforcement distribution [3], good bonding between the matrix and reinforcing agent [4], but also the production technology, its parameters [5] or final machining of the surface, should be noted. Moreover, the compound parameters can also affect the final properties of a composite. Proper phase composition, mass fraction [6], as well as shape or homogeneity [7] are related to the material properties. Thus, designing and manufacturing processes are complex and require detailed analysis of all the factors. Anticipating the influence of each factor has resulted in the opportunity to achieve established properties, facilitate the manufacturing process and curtail expenses at the same time. Thus, it appears advisable to search for the general trends in changing material properties. Moreover, having wide knowledge about technological property variations has resulted in the possibility to avoid problems at the design stage.

The general tendency of changing tribological properties with variations in the stereological parameters of the reinforcement was discovered during research. It should be noted that some disagreement between the established and real stereological properties can be observed, especially for techniques involving component size reduction, like mechanical milling. A similar analysis of the influence of compound size has been carried out by the researchers, however, there is no unequivocal description of the tendency of parameter changes [8-10]. This proves the complexity of the composite manufacturing process. Thus, further research in order to verify general assumptions are necessary, which was the subject of the authors' interest. The correlation between the stereological parameters of the carbon compound and the tribological properties of Al-Al_2O_3+C composites was checked. The presented results are the initial stage of manufacturing composite materials for potential applications in highly loaded friction points.

MATERIAL AND METHODS

The materials for research were heterophase composites, obtained via the powder metallurgy method. Aluminium powder was applied as the matrix material. This high purity powder revealed a spheroidal shape of particles 45 μm in diameter. As the reinforcement, 10.9 vol.% aluminum oxide powder and 9.6 vol.% glassy carbon particles were used. The first component of the reinforcement (Al_2O_3) was a commercial product of the Albemarle company (Martoxid MR 70). These particles ranged between 0.1 and 5 μm in diameter. The second component - glassy carbon (C) was obtained by means of the pyrolysis method from phenol-formaldehyde resin at 1000°C. As part of the preparation, the glassy carbon particles were crushed in a mill, and then sieved according to the desired fraction: 40, 80, 120, 160, 200 μm. Due to the specific mechanical properties (high hardness and low shear modulus), those particles revealed a characteristic spheroidal shape with sharp edges. The current analysis focused on selected fractions (< 40 μm, 80÷120 μm and 160÷200 μm), which were compared to the reference material reinforced by unsieved C particles (called < 200 μm).

The manufacturing process of the composites consisted of high energy ball milling with subsequent hot pressing techniques. The first step was conducted in a Planetary Mono Mill PULVERISETTE 6 classic line. High energy milling was ensured by 650 rpm, a ball to powder ratio 16:1 and 2 hours of milling time (in cycles 5 minutes of milling, then 30-minute break). The milling stage was performed in stainless steel jars using stainless steel balls 10 mm in diameter. Stearin acid was used as the control agent, while argon was applied as the ambient atmosphere. Consolidation was conducted in a semi liquid state. This stage was carried out in a Degussa press under 10 MPa pressure and 0.1 Pa vacuum. In the first step of sintering, the composite powders were preheated up to 400°C and kept at this temperature for 20 minutes under low pressure (2 MPa), then were heated up to 640°C for 15 minutes under the predetermined pressure. As a result of the described technology, four different composite materials with various sizes of carbon component were obtained.

The microstructures of the manufactured materials were observed using a Scanning Electron Microscope, with the secondary electron technique, 15 kV applied voltage and x1000 magnification (Fig. 1). For all the composites, 50 microstructure images were recorded. In the following part of research, those images were analysed using quantitative metallography techniques (Metilo program by Professor Dr hab. Eng. Janusz Szala from Silesian University of Technology). From among the described quantitative parameters, the areal fraction of the glassy carbon particles, average surface area of a single C particle and average size of a single particle were taken into account as the most valuable for thorough analyses.

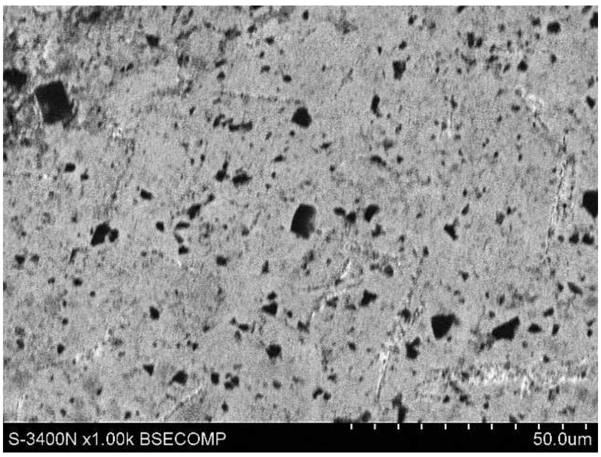

Fig. 1. Example of composite microstructure reinforced by glassy carbon particles

Rys. 1. Przykładowa mikrostruktura kompozytu zbrojonego cząstkami węgla szklistego

As the main part of the conducted research, tribological evaluations were carried out. These tests were made by the ball-on-disc method, under technical dry friction conditions, using a THT tribometer made by CSM Instruments. The sliding distance was 250 m, 10 N load and 0.1 m/s speed (rotary movement). The discs were made from the analysed composite, while steel 100Cr6 was used as the counter specimen (ball 5 mm in diameter). The coefficient of friction (COF), mass loss and predominant wear mechanisms were analysed and correlated to the quantitative parameters.

RESULTS AND DISCUSSION

The processing of the composite powder resulted in changes in the real stereological parameters of each element, however, the microstructure examinations focused on the carbon component. During the research, the basic stereological parameters were measured and described as a function of initial particle size (Table 1). As a result of the milling stage, a significant reduction in the initial size of the carbon particles was observed. On average, the particles were about 40 times finer (in comparison between the initial and final size of C particles). It led to obtaining a submicrometer size of particles, which was favourable for reinforcing composites. However, it should be noted that for the 80÷120 and 160÷200 materials, the highest scatter of single C particle surface area was observed. The level of scatter for those composites, was two times greater than for the < 40 or < 200 materials. Supposedly, for the material with the bigger C particle size, a higher energy of particle crushing is needed. It can be obtained increasing the milling time, rotation speed or ball to powder ratio. However, this analysis showed that increasing the initial C particle size resulted in a higher carbon areal fraction, and increased the average size of a single particle. The variations were not significant, but a clear, linear tendency of changes can be observed (Table 1). Nevertheless, the most interesting results were noticed for the reference material, which was reinforced by unsieved particles. This composite revealed the highest participation of C particles on the surface, and a very high reduction in particle size at the same time. The average size and surface area of single C particles of the reference material were comparable with the composite reinforced by the smallest initial size of C particles (< 40 μm). It was surprising because of the large difference in areal fraction of C particles. The described results are evidence for fine homogenisation of C particles in the volume of the < 200 material. Moreover, it is supposed that for the < 200 and < 40 composites, the utmost fineness was achieved. Nonetheless, this thesis should be explored by lengthening the milling time for all the analysed materials.

TABLE 1. **Stereological parameters of manufactured composites**

TABELA 1. **Parametry stereologiczne wytworzonych kompozytów**

Material	Initial size of C particle [μm]	C particle size after processing [μm] (standard deviation)	Areal fraction of C particles [%] (standard deviation)	Average surface area of single C particle [μm^2] (standard deviation)	Average distance between adjacent C particles [μm] (standard deviation)
< 200 (reference)	0÷200	1.31 (1.19)	4.53 (1.57)	1.65 (6.72)	3.35 (1.66)
< 40	1÷40	1.3 (1.09)	3.09 (0.63)	1.59 (4.15)	3.91 (2.04)
80÷120	80÷120	1.4 (1.30)	3.28 (1.76)	2.00 (13.78)	4.26 (2.22)
160÷200	160÷200	1.46 (1.46)	3.58 (3.13)	2.40 (29.62)	4.50 (2.25)

The tribological properties of the manufactured materials were measured with particular emphasis on the influence of the real stereological parameters of the glassy carbon particles. As the main parameters, the coefficient of friction (COF) and mass loss were taken into account. The performed research revealed a clear influence of the average particle size on the COF value. The bigger the area of a single C surface resulted in a higher friction coefficient (Fig. 2a). Moreover, the distance between the C particles influenced the COF as well. The composites with the smallest distance between adjoining particles (< 200 and < 40) resulted in a lower COF value and mass loss (Fig. 3). Both the described factors are connected with the homogeneity of the materials. The smaller particle size and smaller distance between the particles are evidence of better homogeneity of the materials. Proper distribution of the reinforcing agent provided an opportunity to transfer load stresses to the reinforcing compounds. It is especially important for C particles, because the lubrication effect of those particles can be limited due to the presence of large areas without reinforcement. This phenomena was strictly connected with the wear level, because limitation of the lubricating effect resulted in a higher mass loss of the analysed materials (Fig. 2b, 3b).

The most surprising effect was the fact that the larger amount of C particles on the surface of composite did not have an influence on the tribological properties (Fig. 4). Composites < 40 and < 200 revealed the biggest difference between the C particles present on the composite surface, while the average COF values for these materials were almost the same. The remaining materials revealed a higher COF value. It should be noted, that between 3 and 4% the dependency is almost linear. It suggests that a C presence limit value exists, and when it is exceeded, the COF value decreases. However, further research is necessary for precise description of the limit value. On the other hand, during the analyses of mass loss, no correlations with C particle presence were noticed (Fig. 4b).

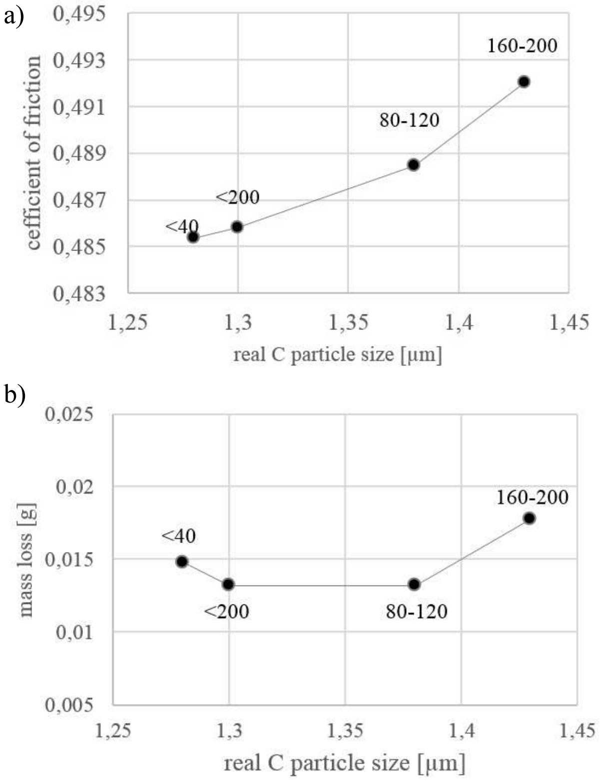

Fig. 2. Influence of real C particle size on friction coefficient (a) and mass loss (b) for $Al-Al_2O_3$ composites

Rys. 2. Wpływ rzeczywistej wielkości cząstek C na współczynnik tarcia (a) oraz ubytek masy (b) kompozytów $Al-Al_2O_3$

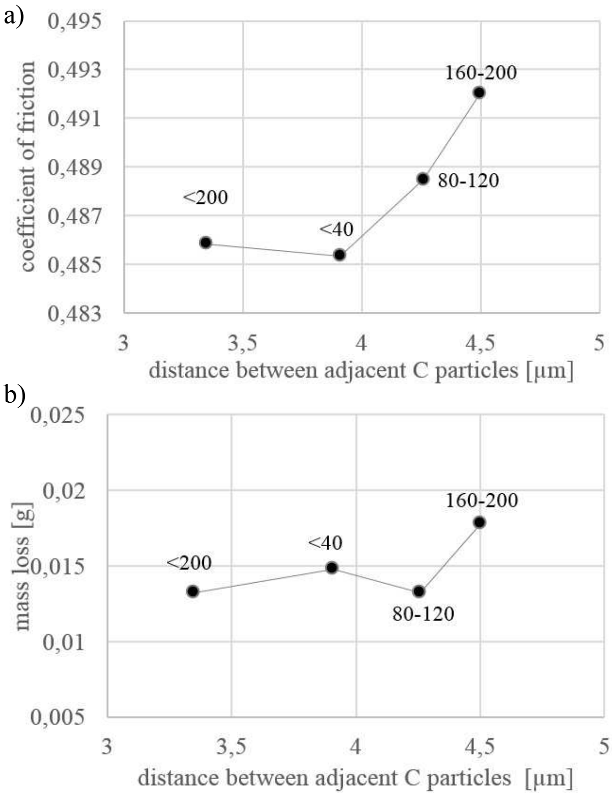

Fig. 3. Influence of average distance between adjacent C particles on friction coefficient (a) and mass loss (b) for $Al-Al_2O_3$ composites

Rys. 3. Wpływ średniej odległości pomiędzy sąsiednimi cząstkami C na współczynnik tarcia (a) oraz ubytek masy (b) kompozytów $Al-Al_2O_3$

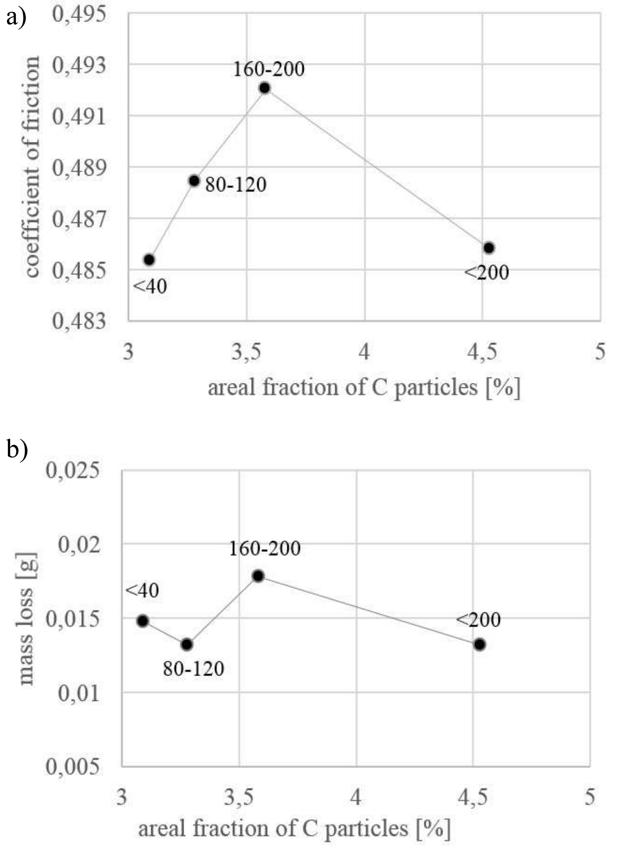

Fig. 4. Influence of C particle presence on composite surface on friction coefficient (a) and mass loss (b)

Rys. 4. Wpływ udziału powierzchniowego C na współczynnik tarcia (a) oraz ubytek masy (b) kompozytów $Al-Al_2O_3$

The analysis of variations of the COF instantaneous values showed a similar behaviour for all the materials (Fig. 5). The fluctuations in the coefficient of friction ranged between 0.43 and 0.53. Moreover, an increasing tendency was observed for all the composites. At the beginning, the materials revealed a COF value at the level of 0.45, then it slowly increased up to a stable value of 0.50. Similarities between the analysed composites can also be detected in the length of the lapping stage. COF stabilisation was achieved after 120÷÷150 meters. However, the lowest variance of COF after the lapping period was noticed for the 80÷120 composite, which was not correlated with any stereological parameters.

After the tribological test, the worn surfaces were analysed to gain a detailed description of the wear behaviour. The predominant wear mechanism was similar for all the materials (Fig. 6). On the surfaces, the effect of mechanical contacts of surface roughness were visible. Some areas were worn by a typical grinding mechanism, however, a significant part of surface was plastically deformed. High deformation of the matrix led to discontinuities created in the internal part of the surface, and processes of delamination in consequence.

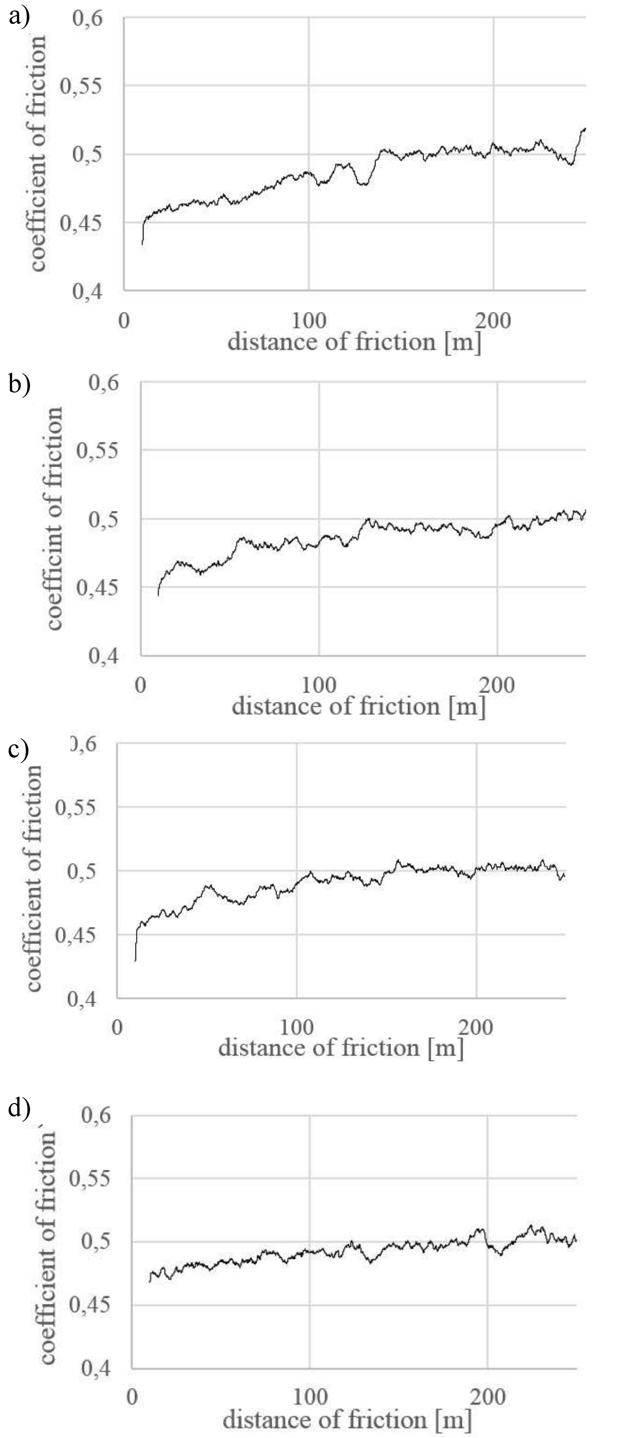

Fig. 5. Variations in friction coefficient on analysed distance for composites: < 200 (a), < 40 (b), 80÷120 (c), 160÷200 (d)

Rys. 5. Wahania wartości chwilowej współczynnika tarcia w funkcji przebytej drogi dla kompozytów: < 200 (a), < 40 (b), 80÷120 (c), 160÷200 (d)

The debris located on the surface was in the form of a loose powder. However, the delamination mechanism with the creation of debris was visible only in small microareas. Furthermore, analysis of the counter specimen revealed flakes of the composite adhering to the surface (Fig. 7). This was noticed for all the tested composites. This behaviour is profitable as it limits the wear of friction couple materials.

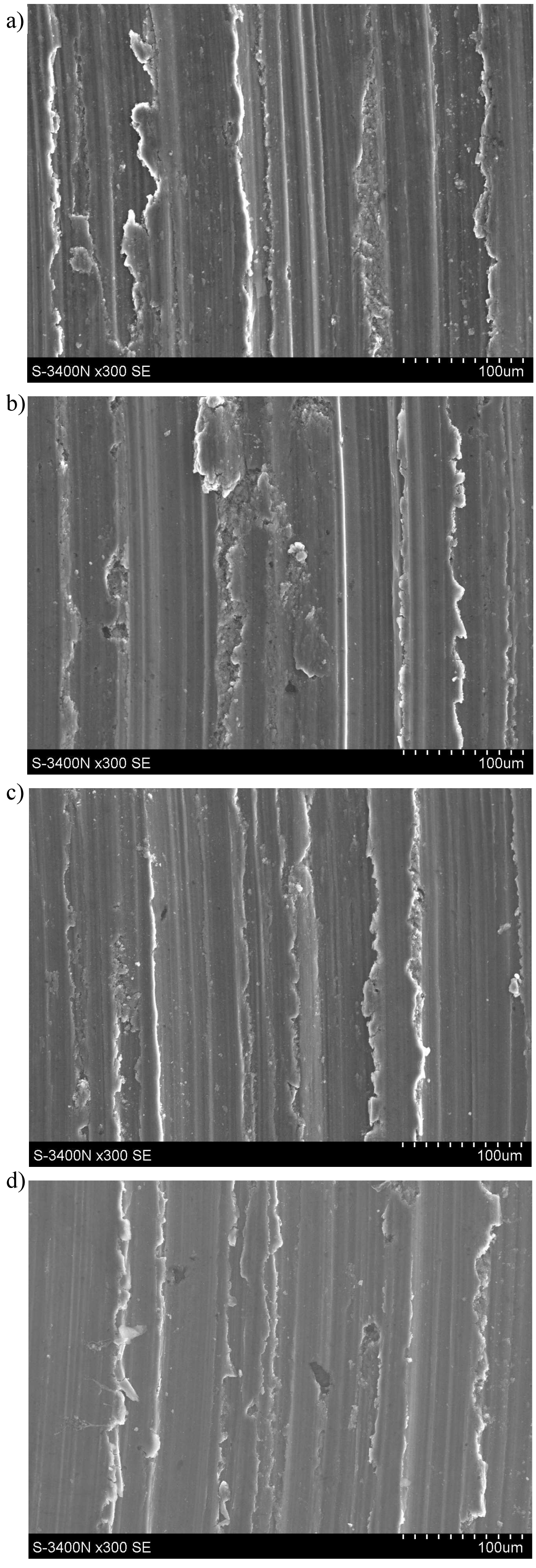

Fig. 6. Surfaces of < 200 (a), < 40 (b), 80÷120 (c), 160÷200 (d) composites after friction against steel ball

Rys. 6. Powierzchnia kompozytów < 200 (a), < 40 (b), 80÷120 (c), 160÷200 (d) po współpracy ciernej ze stalową kulką

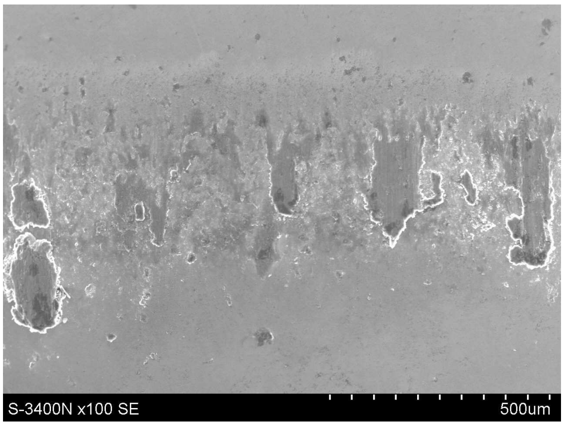

Fig. 7. Surface of steel ball after friction against <200 composite

Rys. 7. Powierzchnia kulki stalowej po współpracy ciernej z kompozytem < 200

SUMMARY

The research focused on the tribological behaviour of heterophase composites $Al-Al_2O_3+C$ depending on the fineness of the carbon component during the production stages. No significant variations in the tribological parameters were observed for the analysed composites. All the composites were characterised by a similar average COF value and similar COF variations along the analysed distance. The predominant wear mechanisms and level of mass loss were also almost the same. It suggests that in the case of particle size reduction techniques, like high energy ball milling, the sieving stage has an insignificant influence on the final properties of the materials. However, slight differences in the carbon stereological parameters should be taken into account. The areal fraction variations of C particles ranged between 3.09 and 4.53%, while the average C particle size was between 1.3 and 1.46 μm. Since the observed differences were not significant, the resultant final properties were similar. Nonetheless, a clear tendency of tribological property changes was discovered. The bigger the real size of C particles, as well as longer distance between adjacent particles, resulted in an increased friction coefficient. However, the most surprising effects were noticed during analyses of the areal fraction of C particles. There was no clear relation between the presence of C particles and the tribological properties. This result is not in agreement with the theoretical knowledge about the lubrication effects of glassy carbon particles. Verification and further analysis are necessary to confirm this thesis for other types of aluminum based composites reinforced by ceramic particles.

CONCLUSION

The presented analysis was carried out to describe the correlation between the changes in the stereological parameters of the carbon component in $Al-Al_2O_3+C$ composites and the tribological properties of those materials. Glassy carbon was applied as a solid lubricant which caused wear limitation and COF stabilisation. The presence of C particles, their size and homogeneous distribution are necessary to fulfil the strict requirements for materials applied in high loaded friction points. As a results of the presented research, some points were discovered:

1. It is possible to design and correct material properties, especially tribological properties, during powder metallurgy processes by selecting the appropriate initial size of particles.
2. The correlations between the stereological parameters of the materials and the initial size of the particles were discovered. The higher initial size of particles resulted in a higher average size of single C particles and greater distance between adjacent particles.
3. The stereological parameters (distance between C particles, size of C particles) had a significant influence on the COF value and wear level. The greater the distance between adjacent C particles and bigger size of those particles resulted in a higher of COF value. Proper distribution of the carbon component, as well as high fragmentation, improved COF stabilisation.
4. Due to the insignificant variations between the final properties of the obtained materials, the sieving stage can be omitted during designing and manufacturing composites powders by high energy ball milling. It may contribute to simplifying material production by powder metallurgy methods.

Acknowledgements

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