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INFLUENCE OF CASTING PROCEDURE ON WEAR OF MAGNESIUM MATRIX COMPOSITES REINFORCED WITH CARBON OPEN-CELLED FOAM

In this article the characteristics of the macrostructure, microstructure (LM, SEM) and selected properties of magnesium matrix composites reinforced with carbon open-celled foam with a porosity of 10 ppi, obtained by two casting techniques - gravity casting and pressure infiltration, were presented. The open porosity and hardness of the composites were determined. Tribological examinations in dry friction conditions were performed by the pin-on-disc method, and the coefficient of friction, weight loss of the sample as well as the cast iron countersample were determined, and the wear trace was characterized. Observations of the examined material surfaces after friction tests were conducted by SEM. In comparison to the gravity cast composite, a lower porosity, higher hardness and finer magnesium matrix size were found for the pressure infiltrated composite. The impact of the casting technique also concerned the tribological properties. Both the composites exhibited a lower coefficient of friction in comparison to pure magnesium, but for the pressure infiltrated composite the coefficient of friction, weight loss of the sample and countersample, as well as the depth of the wear trace were the lowest. Moreover, after the friction tests, different effects were observed on the surfaces. In the gravity cast composite the carbon component cracked and separated from the matrix, in contrast to the pressure infiltrated composite where uniform wear was observed while maintaining continuous bonding with the matrix, which explains the differences in the tribological properties.

Keywords: **magnesium matrix composite, carbon open-celled foam, pressure infiltration, gravity casting, wear resistance, tribological properties**

WPŁYW PROCEDURY ODLEWANIA NA ZUŻYCIE KOMPOZYTÓW MAGNEZOWYCH ZBROJONYCH PIANĄ WĘGLOWĄ

Scharakteryzowano makrostrukturę, mikrostrukturę (LM, SEM) i wybrane właściwości kompozytu na osnowie technicznie czystego magnezu zbrojonego otwartokomórkową pianą węglową o porowatości 10 ppi, otrzymanego dwiema metodami - odlewania grawitacyjnego oraz infiltracji ciśnieniowej. Określono porowatość i twardość kompozytów. Przeprowadzono badania tribologiczne w warunkach tarcia na sucho metodą pin-on-disc i wyznaczono współczynnik tarcia, ubytek masy próbki i przeciwpróbki żeliwnej oraz scharakteryzowano ślad wytarcia. Powierzchnie kompozytów po badaniach tribologicznych scharakteryzowano metodą SEM. Wykazano mniejszą porowatość, większą twardość i mniejsze ziarna osnowy magnezowej kompozytu otrzymanego metodą infiltracji ciśnieniowej w porównaniu z odlewnym grawitacyjnie. Wpływ techniki odlewania dotyczył również właściwości tribologicznych. Obydwa kompozyty charakteryzował mniejszy współczynnik tarcia w porównaniu z próbką referencyjną z materiału osnowy, ale dla kompozytu otrzymanego metodą infiltracji ciśnieniowej ten współczynnik, ubytki masy i głębokość śladu wytarcia były najmniejsze. Ponadto obserwowano różnice na powierzchni wytarcia, komponent węglowy ulegał wykruszeniu w przypadku materiału otrzymanego metodą odlewnia grawitacyjnego, a w infiltrowanym ciśnieniowo następowało jego równomierne zużycie przy zachowaniu ciągłego połączenia z osnową, co tłumaczy różne parametry tribologiczne.

Słowa kluczowe: **kompozyty magnezowe, otwartokomórkowe piany węglowe, infiltracja ciśnieniowa, odlewanie grawitacyjne, odporność na zużycie, właściwości tribologiczne**

INTRODUCTION

Magnesium matrix composites reinforced with carbon materials is a group of composite materials that exhibits better tribological properties and wear resistance compared to the matrix alloys [1-5]. Those composites are commonly reinforced with short carbon fibers [1, 2], glassy carbon particles [3, 5], graphite [6] or with 3D structures - foams [7-9]. Carbon open-celled foams (C_{of}) are widely used as thermal management materials [10, 12], catalysis support [13, 14] or biomaterials [15, 16], but their application as reinforcement in light metal matrix composites is also known [7-9, 17, 18]. This type of continuous, multiaxial reinforcement can be applied in a selected region of the final product (e.g. the outer region in a centrifugal cast) or in the whole composite volume.

Metal matrix composites reinforced with carbon [7, 9, 17, 18], ceramic [19-26] and hybrid [27, 28] foams can be fabricated by various casting techniques such as pressure infiltration [8, 9, 17, 18, 20, 22-25, 27, 28], centrifugal casting [21], squeeze casting [26] and gravity casting [19]. Applying foam reinforcement is especially beneficial where high wear resistance is expected or higher stiffness and compressive strength are required. In the case of carbon foam-magnesium systems, the effect of the matrix composition was earlier described in [9]. However, in composite fabrication the technique of component consolidation is equally important. The selection of an appropriate casting technique allows a proper infiltration ratio, low porosity, good bonding between the components to be obtained. In gravity casting and pressure casting different parameters such as mold temperature, foam temperature, liquid metal temperature and pressure, as well as cooling conditions can be applied, and they will influence the final composite microstructure [19]. The aim of the paper was to show how the casting procedure influences the macrostructure, microstructure and wear properties of a magnesium matrix composite reinforced with the same open-celled carbon foam of 10 ppi porosity, but fabricated by two different methods. Due to their future practical applications, gravity casting and pressure infiltration were used as the methods of component consolidation in the experiments. To reveal the effects of crystallization and exclude the influence of alloying elements [8, 29] technically pure magnesium was applied.

MATERIALS AND METHODS

The materials applied in the experiment were technically pure magnesium (Onyxmet, about 99.9% purity) and carbon open-celled foam with a porosity of about 10 ppi (Fig. 1), fabricated according to the procedure presented in [30]. Composite samples were obtained in the same conditions by the pressure infiltration process in a Degussa press and gravity casting as well. The infiltration process was performed in vacuum at the temperature of 690°C for 5 minutes followed by cooling under a 5 MPa load [31]. A composite sample with a 30 mm diameter was obtained. The gravity casting process was conducted in argon atmosphere at the temperature of 690°C and the casting mold with a carbon foam insert was preheated up to 300°C. The melting temperature of about 690°C was selected based on previous works [8, 9, 31]. The temperature of carbon foam preheating was selected owing to the low thermal stability of C_{of} in an oxygen-containing atmosphere [30]. In both experiments, graphite molds were applied.

The composite macrostructures were characterized on polished surfaces after etching in the authors' own designed solution (33 ml of distilled water, 66 ml of ethanol, 1 ml of nitric acid, 2 g of citric acid and 0.1 g of picric acid) using a stereoscope. Etching was performed during 30 seconds by immersion, repeated in two times. The mean grain surface area was determined on macrographs of etched cross-sections using Met-Ilo software (Prof. J. Szala, Silesian University of Technology). The microstructure of the composites was characterized on polished, unetched surfaces by light microscopy (LM, Nikon Eclipse MA200). The area fraction of carbon foam elements and pores was calculated on 30 LM micrographs by manual detection, using Met-Ilo software. The apparent density and open porosity of both magnesium composites were determined by the Archimedes' method. The Vickers hardness of the composite matrix was determined using a Duramin 5 microhardness testing machine with the load of 200 g.

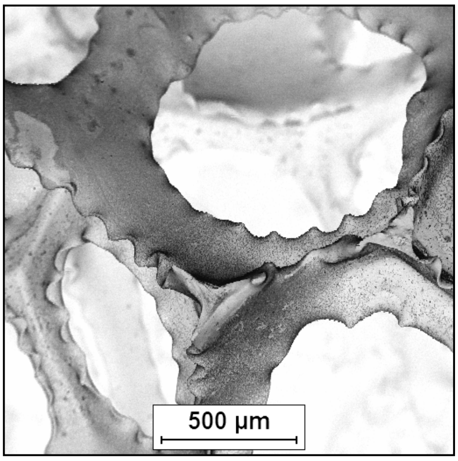

Fig. 1. SEM micrograph of open-celled carbon foam 10 ppi (C_{of}) used in experiment

Rys. 1. Mikrofotografia SEM otwartokomórkowej piany węglowej o porowatości 10 ppi (C_{of}) wykorzystanej w eksperymencie

The tribological properties in dry friction conditions in air such as the coefficient of friction, μ, and mass loss, Δm, were examined using the pin-on-disc method (Tribometer TM-01M). For the experiment, a cast iron pin (ϕ = 3 mm, friction radius = 4 mm) was used and the tribological parameters were measured for a pin load of 5 N, speed of 0.10 m/s and friction distance of 700 m. Finally, the wear surface was characterized using SEM (Hitachi S-4200). The depth of the wear trace was examined using optical profilometry (Non-contact 3D optical profiler Sensofar S NEOX with SensoSCAN and SensoMAP software).

RESULTS AND DISCUSSION

The macro and microstructure of the examined composites presented in Figures 2 and 3 show major differences between the obtained materials. For the gravity cast composite, a higher porosity around the carbon foam elements was detected (Figs. 2a and 3a), which suggests the influence of the shrinkage effect. Observation of the composite grain size (Fig. 2) shows that pressure infiltration causes significant grain size refinement in comparison with gravity casting. The measurements revealed that the mean grain surface area for the pressure cast composite is 0.32 ±0.31 mm^2, while for the gravity cast 0.70 ±0.70 mm^2. The grain size distribution (Fig. 4) in both composites shows a higher amount (about 15%) of a grains in the range from 0.1 mm^2 to 1 mm^2 in the pressure infiltrated composite than in the gravity cast one. Furthermore, in the composite obtained by gravity casting, the amount of grains in the range from 1 to 10 mm^2 is five times higher than in the pressure infiltrated one. In both processes the graphite mold temperature was different, in gravity casting it was preheated to 300°C and in pressure infiltration it was heated up to 690°C together with the magnesium.

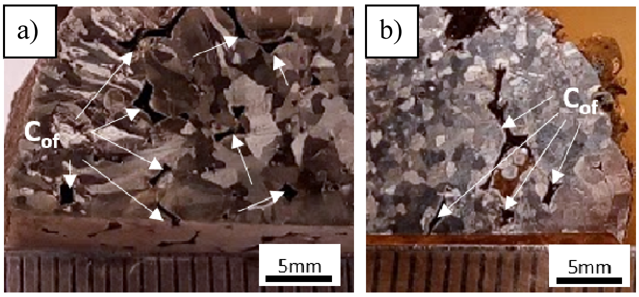

Fig. 2. Macrostructure of C_{of} –Mg composites obtained by gravity casting (a) and pressure infiltration (b), etched cross-sections

Rys. 2. Makrostruktura kompozytów C_{of} –Mg uzyskanych metodą odlewania grawitacyjnego (a) i infiltracji ciśnieniowej (b), przekroje poprzeczne trawione

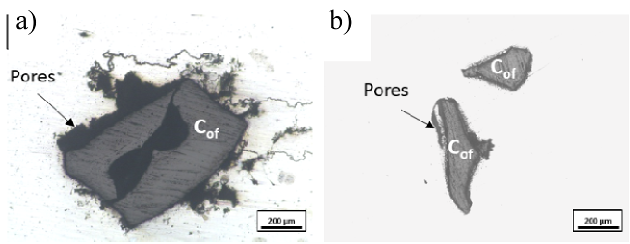

Fig. 3. Microstructure of C_{of} –Mg composites obtained by gravity casting (a) and pressure infiltration (b), polished unetched cross-sections, LM

Rys. 3. Mikrostruktura kompozytów C_{of}–Mg uzyskanych metodą odlewania grawitacyjnego (a) i infiltracji ciśnieniowej (b), przekroje poprzeczne nietrawione, LM

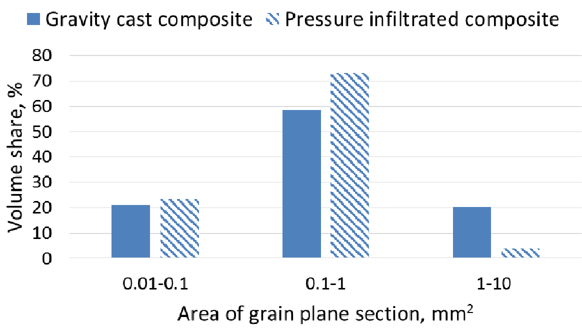

Fig. 4. Distribution of magnesium grain surface area in examined C_{of} –Mg composites

Rys. 4. Rozkład wielkości pola powierzchni przekroju płaskiego ziarna magnezu w badanych kompozytach C_{of} –Mg

Additionally, in both processes the heat transfer was different because the mold was cooled by water during processing by pressure infiltration, while in gravity casting it was cooled by air, which is slower. Moreover, this explains the observed differences in the magnesium matrix grain size. The results of the quantitative evaluation (Table 1) show that the area fraction of the carbon foam for the gravity cast composite was 3.13% (with a standard deviation of about 2.40) and for the pressure infiltrated composite it was 3.42% (with a standard deviation of about 2.63). Those results confirm proper infiltration of the applied carbon foam, whose porosity was calculated as approx. 97%. For analysis of the pores in the composites, quantitative metallography revealed their higher area fraction in the gravity cast composite than in the pressure infiltrated composite, which is in good agreement with the open porosity measured by the Archimedes' method (Table 1). The absence of an increase in hardness for the gravity cast composite in comparison with the sample of pure cast magnesium could be explained by the high porosity of this composite. Comparing the hardness of the composites, the pressure infiltrated one exhibits a higher hardness than the gravity cast composite, which can be due to the lower porosity and smaller grain size of the magnesium matrix.

TABLE 1. **Apparent density, open porosity, Vickers hardness and results of composite microstructure quantitative evaluation**

TABELA 1. **Gęstość pozorna, porowatość otwarta, twardość HV i wyniki ilościowej oceny mikrostruktury kompozytów**

	Pure magnesium	C_{of} – Mg obtained by gravity casting	C_{of} – Mg obtained by pressure infiltration
Apparent density [g/cm^3]	1.74 ±0.01	1.65 ±0.01	1.72 ±0.01
Open porosity [%]	0.00 ±0.00	5.72 ±0.10	1.55 ±0.08
Vickers hardness, HV0.2	31 ±2	31 ±4	38 ±2
Area fraction of carbon foam [%]	-	3.13 ±2.40	3.13 ±2.63
Area fraction of pores [%]	-	5.19 ±2.55	1.91 ±1.51

The results of the tribological examinations are presented in Figures 5-9. They shown that the coefficient of friction (Fig. 5 and Tab. 2) for the composites is lower than the reference magnesium, and evidently less for the C_{of}–Mg composite obtained by pressure infiltration. Moreover, in the case of that composite, the coefficient of friction vs. distance curve is more stable. The mass loss values presented in Table 2 shows that the highest wear of sample was observed for the gravity cast composite, even in comparison with the pure matrix, and could be explained by its relatively high porosity. Analysis of the countersample mass change revealed that the wear is less after dry friction with the composites than the reference sample, and in that case the pressure infiltrated composite was also the most advantageous.

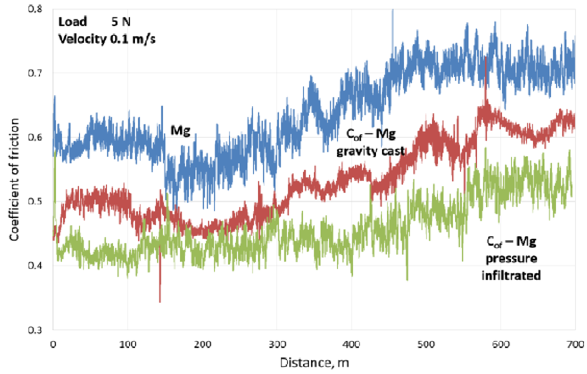

Fig. 5. Changes in coefficient of friction determined for composites and reference matrix over distance of 700 m

Rys. 5. Zmiany współczynnika tarcia określone dla kompozytów i materiału referencyjnego na drodze 700 m

TABLE 2. **Wear of materials examined in dry friction conditions**
TABELA 2. **Zużycie materiałów badane w warunkach tarcia suchego**

	Pure magnesium	C_{of} – Mg obtained by gravity casting	C_{of} – Mg obtained by pressure infiltration
Coefficient of friction [μ]	0.64 ±0.16	0.53 ±0.10	0.46 ±0.08
Weight loss of sample [g]	0.0098	0.0186	0.0071
Weight loss of pin [g]	0.0013	0.0006	0.0005
Average depth of wear trace [μm]	240	320	220

The results of wear trace characterization by optical profilometry presented in Figures 6-8 shows differences in the depth of the wear trace dependent on the examined material. The highest depth was observed for the gravity cast composite, less for the reference material and the least for the pressure infiltrated composite. The poor result obtained in the gravity casting process is caused by the high porosity of the composite manufactured in that way. Additionally, some differences are visible in the profiles, for pure magnesium grooves are noticeable, while in the composites they are absent.

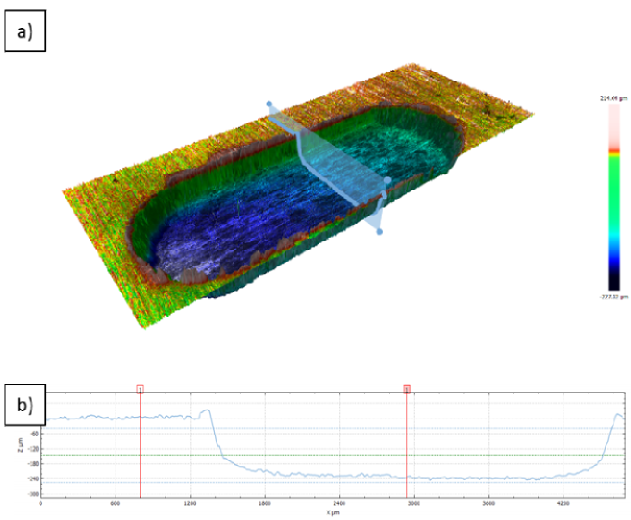

Fig. 6. 3D (a) and 2D (b) wear tracks of magnesium after dry friction tests

Rys. 6. Profile 3D (a) i 2D (b) śladu wytarcia magnezu po testach tarcia suchego

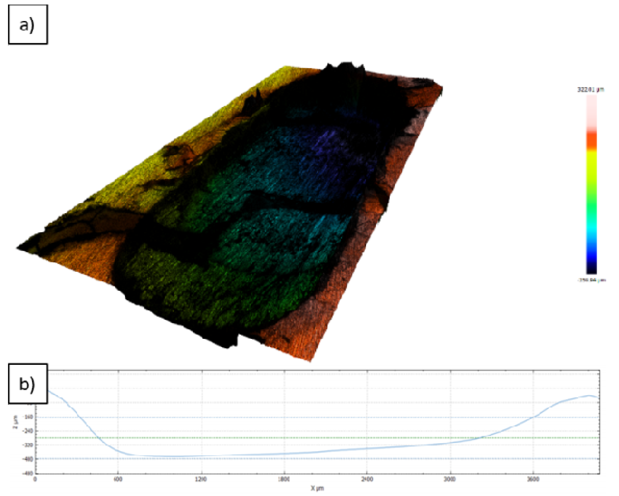

Fig. 7. Wear tracks 3D (a) and 2D (b) of C_{of} –Mg composite obtained by gravity casting after dry friction tests

Rys. 7. Profile 3D (a) i 2D (b) śladu wytarcia kompozytu C_{of} –Mg uzyskanego metodą odlewania grawitacyjnego po testach tarcia suchego

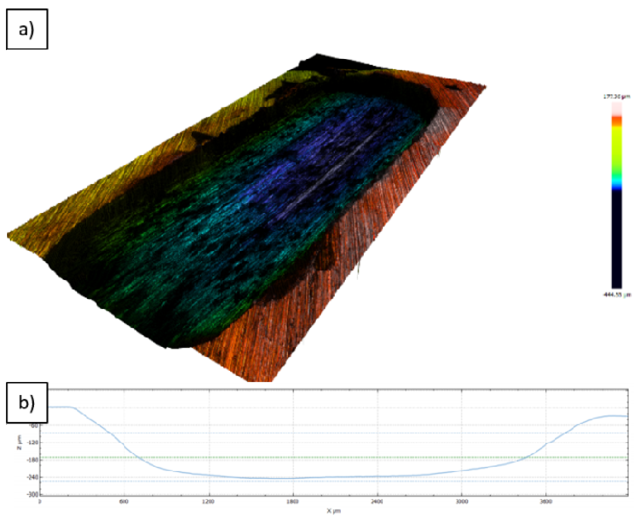

Fig. 8. 3D (a) and 2D (b) wear tracks of C_{of} –Mg composite obtained by pressure infiltration after dry friction tests

Rys. 8. Profile 3D (a) i 2D (b) śladu wytarcia kompozytu C_{of}–Mg uzyskanego metodą infiltracji ciśnieniowej po testach tarcia suchego

The surfaces of the examined materials after the dry friction tests are presented in the SEM micrographs in Figure 9. On the surface of the magnesium reference sample (Fig. 9 a,b) such effects as metal plastic deformation, cracks, grooves and delamination as well as friction products agglomerated in the grooves can be observed. Applying carbon foam as reinforcement limits plastic deformation and delamination independent of the casting procedure, however, major differences were observed between the composites. In the gravity cast composite the effects of plastic deformation and delamination were not observed, but the processes of carbon component degradation such as cracking and pulling out were visible and they were concentrated at the interface. The wear products formed mainly by metal oxide paticles and carbon fragments accumulated mainly in discontinuities at the interface boundary. The wear effects visible at the interface are probably caused by the presence of shrinkage pores in this area. The grooves formed in the magnesium matrix are distinctly shallower than in the reference sample. SEM observation of the pressure infiltrated C_{of}–Mg composite reveals that the morphology of the surface after the same friction tests is different. Degradation processes of the carbon foam and interface were not revealed. The open-celled foam is well connected with the matrix, and uniform wear occurs, which indicates a solid tribofilm formation.

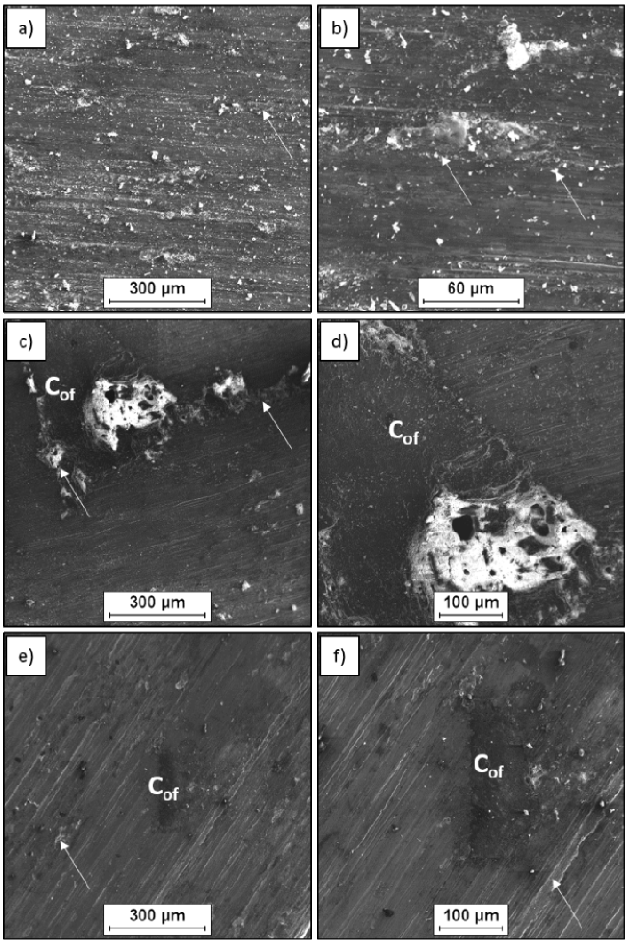

Fig. 9. SEM micrographs of material surfaces after dry friction tests: a-b) pure magnesium, c-d) C_{of}–Mg composite obtained by gravity casting, e-f) C_{of}–Mg composite obtained by pressure infiltration, cracks and delamination effects marked with arrows

Rys. 9. Mikrofotografie SEM powierzchni materiałów po testach tarcia suchego: a-b) czysty technicznie magnez, c-d) kompozyt C_{of}–Mg uzyskany metodą odlewania grawitacyjnego, e-f) kompozyt C_{of}–Mg uzyskany metodą infiltracji ciśnieniowej, pęknięcia i delaminacja wskazane strzałkami

The differences in the composite matrix wear in comparison with pure magnesium were detected, Figure 9e-f shows some grooves in the pressure infiltrated C_{of}–Mg composite, but their intensity is lower than in pure magnesium and that confirms the shape of the wear trace profile (Fig. 8b). The microstructure of the cross-section samples after the tribological test (Fig. 10) confirms the differences in the surface profile; for the gravity cast material it is more developed and contains relatively deep wear traces (Fig. 10a). Moreover, evident degradation processes in this composite are visible, they occur at the wear surface by breaking out of the carbon component, in the C_{of} by cracking propagation and at the interface also by cracking propagation and component separation. Those effects explain the worse tribological properties of the gravity cast composite and indicate the shrinkage pores, particularly at the interface, as the main source of microstructure degradation.

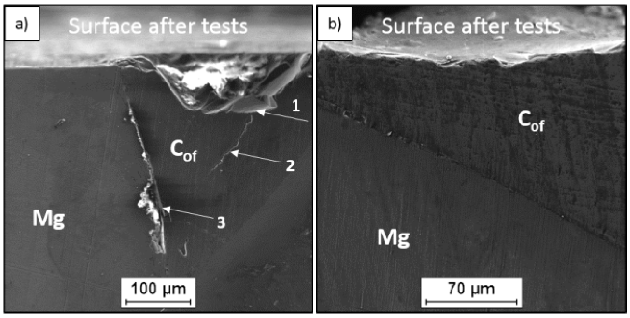

Fig. 10. SEM micrographs of C_{of}–Mg composite after tribological tests, cross-section, region near wear surface: gravity cast composite (a) and pressure infiltrated composite(b); 1 - gap in C_{of}, 2 - crack in C_{of}, 3 - crack at interface

Rys. 10. Mikrofotografie SEM kompozytu C_{of}–Mg po próbach tarcia, przekrój poprzeczny, strefa przy powierzchni tarcia: kompozyt odlewany grawitacyjnie (a) i kompozyt infiltrowany ciśnieniowo (b); 1 - ubytek w C_{of}, 2 - pęknięcie w C_{of}, 3 - pęknięcie na granicy rozdziału

SUMMARY

In this study, we presented a new ultra-light magnesium-based matrix composite that was reinforced with open-celled carbon foam (3 vol.%), which was developed using the pressure infiltration and gravity casting methods. We demonstrated the dependence of the composite microstructure and tribological properties on the applied casting procedure. Thus, the following conclusions have been drawn based on the obtained results:

1. The effect of the casting procedure on the carbon foam stability, open porosity, hardness, grain size and microstructure was observed. For the gravity cast composite, shrinkage pores were detected near the carbon foam and this effect was more intense than in the pressure infiltrated composite. Moreover, due to pressure infiltration the grain size of the magnesium matrix, characterized by the grain surface area, was approx. 50% lower than for gravity casting, and the hardness of composite was higher as well.
2. Applying carbon foam as the reinforcement of pure magnesium led to a decrease in the coefficient of friction and stabilization of its value, independent of the component consolidation method. That effect was stronger for the pressure infiltrated composite, and could be a result of the lower porosity, lower size of matrix grains, and explains the lower depth of the wear trace compared to the gravity cast composite.
3. After the friction tests on the examined materials surfaces, different structural effects were observed. Metal delamination and accumulation of the friction

products occurred on the reference magnesium, while that process was not evident on the composites. The examinations also revealed a distinct differences between the wear of the composites dependent on the technology because in the gravity cast material both cracks at the interface and local degradation of the carbon foam were observed, while in the pressure infiltrated composite the wear of the carbon foam occurred uniformly.

Acknowledgement

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