Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych

17: 2 (2017) 90-96

COMPOSITES THEORY AND PRACTICE

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Received (Otrzymano) 22.04.2017

TRIBOLOGICAL PROPERTIES OF Al MATRIX COMPOSITES REINFORCED WITH MAX TYPE PHASES

A method was developed to manufacture Al-Si alloy matrix composites reinforced with MAX phases by squeeze casting pressure infiltration of porous preforms. The MAX phases were synthesized using self-propagating high-temperature synthesis (SHS) in the microwave assisted mode. For the produced composites abrasive wear resistance tests were carried out using the pin-on-flat method with reciprocating motion for different load values (0.1, 0.2 and 0.5 MPa), while maintaining other parameters (sliding distance, speed) constant. The sliding distance equaled 2000 m with the average speed of 0.3 m/s, whereas the flat counterpart was made of CT70 tool steel with the hardness of 67 HRC and roughness Ra = 0.4÷0.6. Before testing both of the tribosurfaces were degreased with acetone. Volumetric sample consumption was investigated and changes in the structure of the working surfaces were analyzed. Optical and scanning electron microscopy analysis were also performed and elaborated in order to facilitate understanding and interpretation of the wear mechanisms. It was confirmed that the composite materials exhibit more than two times higher wear resistance than that of the matrix itself. The wear rate of the matrix falls within the range of $3.5÷5.5·10^{-4}$ mm^3/Nm, while for the composite material - $1.3÷2.4·10^{-4}$ mm^3/Nm. In the Al-Si matrix the main wear mechanism was identified to be based on plastic deformation composed of scaling and cracking processes, while for the MAX phase composite it is principally abrasive wear leading to pre-fracture, delamination and extraction of MAX phase platelets.

Keywords: MAX phases, SHS, microwave, porous structure, squeeze casting, wear resistance

WŁAŚCIWOŚCI TRIBOLOGICZNE KOMPOZYTÓW NA OSNOWIE STOPU Al UMACNIANYCH FAZAMI TYPU MAX

Opracowano metodę wytwarzania kompozytów na osnowie stopu Al-Si wzmocnionego fazami typu MAX metodą infiltracji ciśnieniowej porowatych preform. Fazy typu MAX syntezowano metodą samorozprzestrzeniającej się syntezy wysokotemperaturowej (SHS) wspomaganej mikrofalami. Dla wytworzonych kompozytów przeprowadzono badania odporności na zużywanie ścierne metodą pin-on-flat realizującą ruch posuwisto-zwrotny dla różnych wartości obciążenia (0,1, 0,2 i 0,5 MPa) przy zachowaniu pozostałych parametrów (droga ścierania, prędkość) stałych. Droga ścierania wynosiła 2000 m przy prędkości średniej 0,3 m/s, zaś przeciwpróbka wykonana była ze stali. Zbadano objętościowe zużycie próbki oraz przeanalizowano zmiany w strukturze powierzchni współpracujących. Przeprowadzono analizę mikroskopową metodami mikroskopii optycznej i skaningowej w celu ułatwienia zrozumienia i interpretacji mechanizmów zużycia. Potwierdzono, że materiały kompozytowe wykazują ponad dwa razy większą odporność na zużywanie ścierne od materiału osnowy. Współczynnik zużycia osnowy wynosił $3,5÷5,5x10^{-4}$ mm^3/Nm, podczas gdy dla materiału kompozytowego był równy $1,3÷2,4·10^{-4}$ mm^3/Nm. W przypadku osnowy Al-Si zaobserwowano mechanizm zużycia oparty na odkształceniu plastycznym, zaś dla kompozytu wzmocnionego fazami typu MAX było to głównie zużywanie ścierne, prowadzące do powstania pęknięć, delaminacji i ekstrakcji fragmentów płytek faz typu MAX.

Słowa kluczowe: fazy MAX, synteza SHS, mikrofale, porowata struktura, infiltracja ciśnieniowa, odporność na zużywanie ścierne

INTRODUCTION

In the majority of leading industries there is a significant demand for materials that are highly wear-resistant. Conventional solid lubricants such as noble metals (Ag, Au, Pt), inorganic fluorides (LiF, CaF_2, BaF_2) or metal oxides (TiO_2, NiO_2) are characterized by proper tribological properties, but they are brittle at elevated temperatures (above 350°C), which limits the possibilities of their application. Composite self-lubricating coatings are used to expand the application area. In these applications, the MAX phases, in particular Ti_2AlC, Ti_3AlC_2, Ti_3SiC_2 are ideal candidates because of the formation of oxide protective layers on them during the wear process. MAX phases are a relatively new group of materials consisting of ternary carbides or nitrides defined by the general formula of $M_{n+1}AX_n$, where M stands for an early transition metal, A is an element from the A group (i.e. Al, Ga, In, Ge, Sn, Pb, Si) and X is carbon or nitrogen. They are also called machinable ceramics with a molecular structure of layered three-component systems [1]. MAX phases combine the advantages of metals and ceramics in a unique way. They are characterized by high thermal and electrical conductivity, good machinability, high thermal shock, oxidation and corrosion resistance. They are also able to retain their high mechanical properties at elevated temperatures. To date there are approximately 70 MAX phases known. MAX type phases can be used, among others, as reinforcement for composite materials, structural ceramics for applications at elevated temperatures, nozzles, molten metal or hot gas filters, vibration-dampening material or biocompatible biomedical implants, heat exchangers, gas turbine engine components, electrical contacts, foil air bearings, furnace elements, outer surfaces of cylinders and pistons in diesel engines, directly heated catalyst support: ceramic based armors, patrol vehicles, helicopters or even battle tanks [1-5].

The wear properties of MAX phases have already been described extensively in the literature. El-Raghy et al. [6] tested the friction coefficient (μ) for fine and coarse Ti_3SiC_2 and found that it was in the range of 0.15÷0.4, while the wear rate (WR) equaled $1.34\cdot10^{-3}$ mm^3/Nm and $4.25\cdot10^{-3}$ mm^3/Nm, respectively for the pin-on-disc method. Sun, Zhang and Souchet [7, 8] defined the μ of Ti_3SiC_2 as 0.4÷0.5, although it turned out to be only 0.1 for a diamond counterpart [9]. Continuing ball-on-disc tests, Emmerlich et al. and Sarkar et al. [10, 11] then observed the formation of an Al_2O_3 oxide protective layer on the surface of Ti_3SiC_2 and determined μ as 0.5÷0.55. Zhai et al. [3] used a block-on-disc method at 0.1÷0.8 MPa on a low carbon steel counterpart for testing Ti_3SiC_2 to establish the impact of abrasion speed on μ and abrasive wear. It was found that as the speed increased from 5 to 60 m/s, μ decreased from 0.53 to 0.09. Gupta et al. [12] also investigated the tribological properties of other MAX-type phases (Ti_2AlC, Cr_2AlC, Ta_2AlC, Ti_3SiC_2, Ti_2AlN, Ti_4AlN_3, Cr_2GeC, Cr_2GaC, Nb_2Snc, Ti_2SnC) with the use of the tab-on-disc method. For all the MAX phases μ initially was 0.1÷0.2 and increased up to 0.4 with a WR lower than 10^{-4} mm^3/Nm. Hongxiang et al. [3] also investigated the tribological properties of Ti_3AlC_2 against low-carbon steels at 0.8 MPa, which resulted in a μ of 0.1 with a WR of $2.5\cdot10^{-6}$ mm^3/Nm. Although the wear resistance of MAX phases is high, the wear rate is lower than for other reinforcements i.e. Al_2O_3 or Si_3N_4 (in the order of 10^{-7} mm^3/Nm). When stabilized, the friction coefficients are, in general, lower than for other tribocouples for steel counterparts i.e. steel (0.57), aluminum (0.47) or copper (0.36) [13]. This feature makes reinforcements based on MAX phases the perfect solution to improve the wear properties of metal matrix composites.

Within the range of MAX phase composites the following combinations have been studied. Wang et al. tested Ti_3SiC_2/Cu composites manufactured by spark plasma sintering with 5, 10, 15 and 20% Cu. The WR was measured to be in the range of 2.79÷÷4.11$\cdot10^{-4}$ mm^3/Nm and μ equaled 0.48÷0.54. Sun et al. manufactured Ti_3SiC_2/TiC composites by vacuum sintering which exhibited a WR of $9.9\cdot10^{-5}$ mm^3/Nm and μ of 0.4÷0.5 [14]. The addition of Ti_5Si_3 reinforcement to a $Ti_3Si(Al)C_2$ matrix allowed μ to be reduced from 0.75 to 0.6 [15]. Hu et al. manufactured Ti_3SiC_2 composites strengthened with 10 or 20% Al_2O_3 particles, which resulted in both μ (0.3÷0.4) and WR improvements [16]. Ti_3SiC_2/WC-10Co composites, with 3 wt.% WC-10Co were subjected to the ball-on-disc test, which resulted in μ in the range 0.40÷0.48, and a WR ranging between 0.6-1$\cdot10^{-4}$ mm^3/Nm [17]. In another study Ti_3SiC_2/TiB_2 [18] composites were investigated. Studies of abrasive wear on the steel counterpart for the ball-on-flat test for the matrix and the composite matrix were conducted for contents of 15 and 20 wt.% TiB_2 and TiC. The strengthening significantly improved the tribological properties. Gupta and co-authors used 35, 20, 10 and 5 vol.% Ti_3SiC_2 reinforcement in Al composites produced by hot pressing [3]. The results of tab-on-disc abrasion resistance tests were in the range of 2÷6$\cdot10^{-4}$ mm^3/Nm, while the coefficient of friction decreased with an increase in Ti_3SiC_2 in Al.

In this paper the possibility of enhancing the tribological properties of Al based composites was investigated. Aluminum and its alloys are characterized by low wear resistance and therefore Ti_2AlC and Ti_3AlC_2 MAX phase reinforcements were applied. The reciprocating pin-on-flat test was performed under dry conditions in order to identify the wear behavior under three different loads. Dry conditions were applied in order to imitate the extreme environment, which is typical for emergency situations. Optical and scanning electron microscopy analyses were also conducted and elaborated in order to facilitate understanding of wear mechanisms.

EXPERIMENTAL METHODS AND APPROACH

Commercial Ti (99.5% Ti, −325), Al (99.9% Al, −325, Alfa Aesar) and graphite (99.5% C, −325SGL Carbon Ltd graphite) powders were used as the starting materials with molar ratios 2:1:1 to prepare a stoichiometric reactant mixture and fabricate Ti_2AlC and Ti_3AlC_2 by microwave assisted self-propagating high-temperature synthesis (MASHS). The Ti, Al, C powder amounts were firstly weighed with the accuracy to 0.001 g and mixed with ZrO_2 balls for 10 minutes. Subsequently, the powders were uniaxially cold-pressed in a hydraulic press into samples in the shape of pellets 22 mm in diameter under a pressure of 900 MPa for 10 seconds. Afterwards, the prepared samples were subjected to MASHS, which was conducted in a microwave reactor [19]. The temperature was measured by a Raytek Marathon MM pyrometer with a measuring spot diameter of 0.6 mm. The ignition temperature was reached and synthesis started at ~670°C when the melting point of Al was attained, while the combustion temperature exceeded 1600°C according to the reaction scheme described in a previous work [20]. The prepared samples were subsequently used as the reinforcement for composite materials based on MAX phases. Porous preforms were infiltrated with the use of the squeeze casting method with Al-Si eutectic alloy AC 44200 (10÷13.5% Si, 0.4% Fe, 0.05% Cu, 0.4% Mn, 0.1% Zn, 0.15% Ti). The alloy was not modified but casting in a permanent die at high pressure contributed to grain refining. The preforms were heated to 750°C. The melted metal at the temperature of 770°C was pressurized to fill up the die under the pressure of 40 MPa. The volumetric content of reinforcement in the composite material equaled approximately 60%. For the produced composites abrasive wear resistance tests were carried out using the pin-on-flat method (two-body abrasive wear test) with reciprocating motion for different load values (0.1, 0.2 and 0.5 MPa), while maintaining the other parameters (sliding distance, speed) constant. The sliding distance equaled 2000 m with the average speed of 0.3 m/s, whereas the flat counterpart was made of CT70 tool steel with a hardness of 67 HRC and roughness *Ra* = 0.4÷0.6. Before testing both of the tribosurfaces were degreased with acetone. Volumetric sample consumption was investigated and changes in the structure of the working surfaces were analyzed.

RESULTS AND DISCUSSION

The characteristic plate-like nanolaminate microstructure of Ti_2AlC and Ti_3AlC_2 MAX phases is shown in Figure 1a. The platelets have the proper length of 10-50 μm and width of approximately 5 μm. Apart from the desired phases TiC is also present in the material. Figure 1b presents the material structure after the infiltration process. The filling of the open porosity is complete and even. The dark areas represent the Al-Si matrix, while the bright platelets are the MAX phases. In Figures 2 and 3a comparison of the working wear tracks of the reference sample made of the matrix material and of the manufactured composite is shown. In the unreinforced reference sample (Fig. 2a), many deep grooves consistent with the abrasion test direction and sliding marks can be seen. In the composite sample (Fig. 2b) there is no such effect visible, which may implicate that the MAX phases act protectively to the matrix and improve its wear resistance. MAX phases bind the matrix, prevent plastic deformation and also increase the stiffness and Young's modulus of the material. Moreover, the topography of these two samples is also shown in Figure 3. For the Al matrix sample (Fig. 2a, Fig. 3a) the wear tracks are typical for conventional metallic materials, for which the wear behavior is dominated by plastic deformation [11].

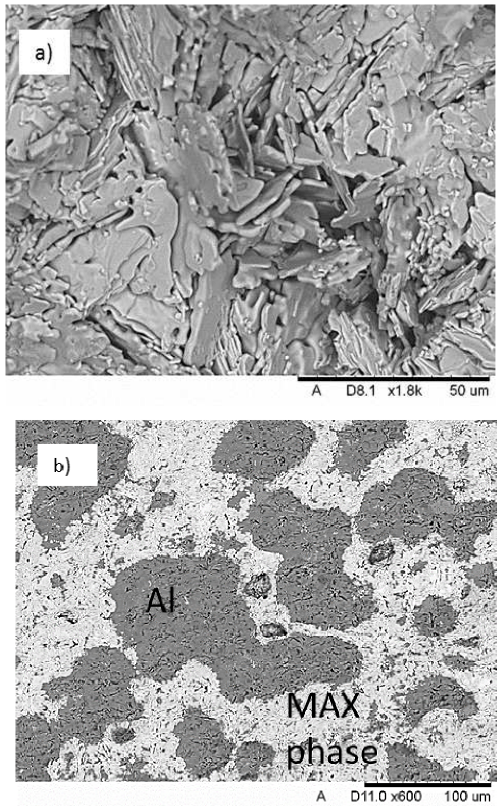


Fig. 1. View of microstructure of manufactured samples: a) MAX phase reinforcement, b) MAX phase (Ti_2AlC i Ti_3AlC_2) + Al-Si composite material

Rys. 1. Widok mikrostruktur wytworzonych próbek: a) wzmocnienie z fazy typu MAX, b) materiał kompozytowy na osnowie stopu Al-Si wzmocniony fazami typu MAX (Ti_2AlC i Ti_3AlC_2)

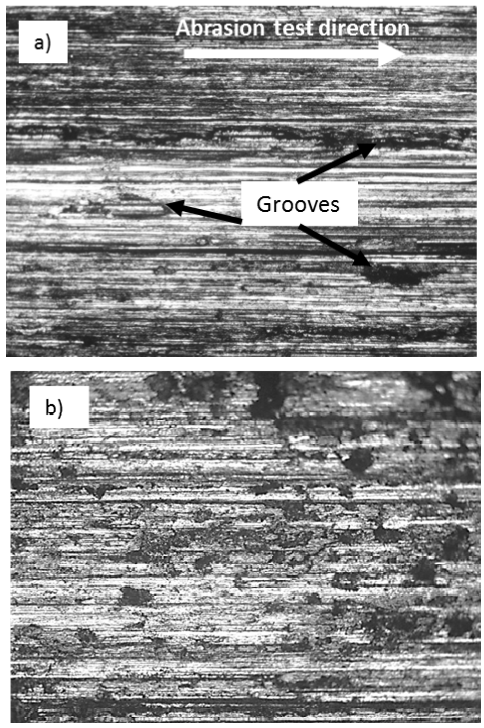


Fig. 2. View of working surfaces: a) Al-Si matrix, b) MAX phase + Al-Si composite material

Rys. 2. Widok powierzchni współpracujących: a) osnowa Al-Si, b) materiał kompozytowy na osnowie stopu Al-Si wzmocniony fazami typu MAX

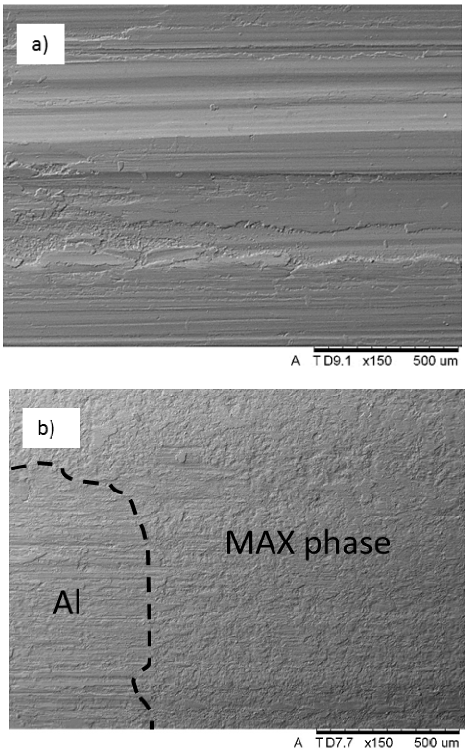


Fig. 3. Topography of working surfaces: a) Al-Si matrix, b) MAX phase + Al-Si composite material

Rys. 3. Topografia powierzchni współpracujących: a) osnowa Al-Si, b) materiał kompozytowy na osnowie stopu Al-Si wzmocniony fazami typu MAX

Many studies indicate that the main abrasive mechanism for MAX phase materials is abrasion of the working surfaces by grains removed from the MAX phase surface. The lower wear rate of the composite material in relation to the matrix is also believed to be caused by slow abrasion of the MAX phase platelets and the formation of tribofilms on the composite surface. Gupta proposed a classification of tribofilms into 4 groups, depending on their source [3]. For type I the triboreactions occur mainly in the MAX phase, while for type II they occur on the counterpart surface. When both of the working surfaces contribute to the formation of tribofilm, then it belongs to type III. Composite materials based on MAX phases predominantly form type IV tribofilms. In Figure 4 micrographs of the sample surfaces after the wear tests with three different loads are set together. As can be stated, the wear of the tribosurfaces increases with the load. On the micrographs presenting the Al-Si matrix, a massive amount of loose, powdered material can be seen, as debris is accumulated on the surface of the tested sample. Cracks, scratches and scale formation are also visible with propagation congruent with the wear test direction. The sliding marks consist of grooves of even a 50÷100 μm width. As for the MAX phase composite samples such effects are not present. Only remote precipitations result from the slight micro-fracture of the MAX phase platelets.

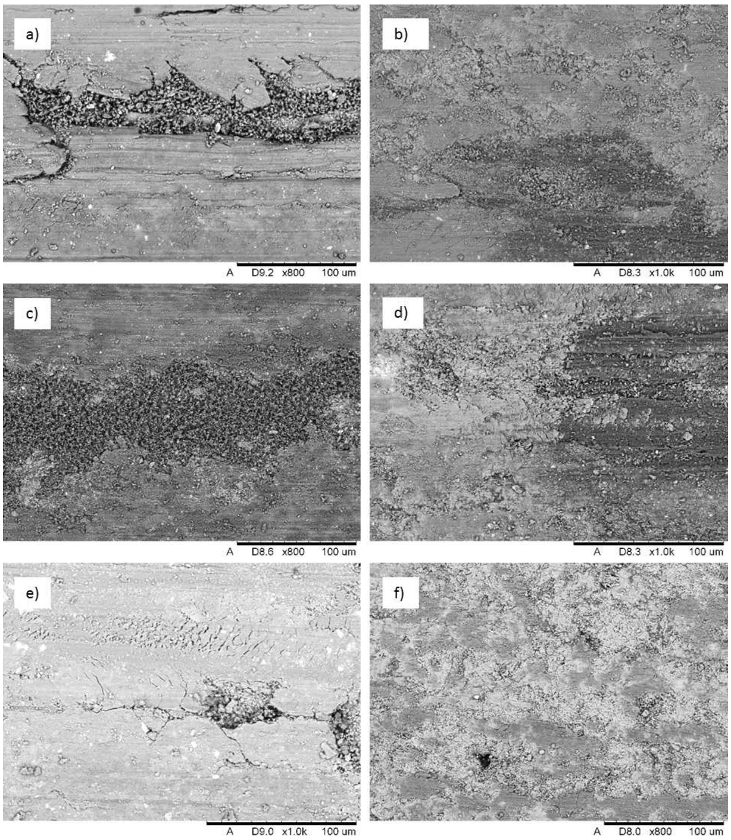
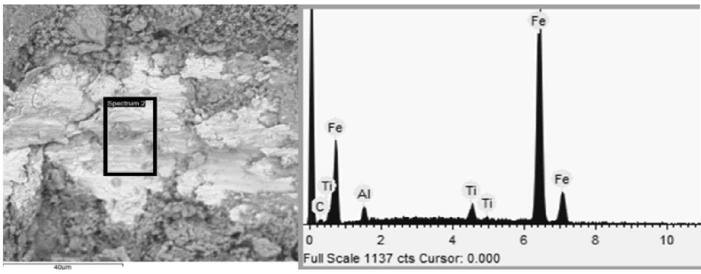


Fig. 4. Comparison of working surfaces for different loads: a) 0.1 MPa, matrix, b) 0.1 MPa, composite, c) 0.2 MPa, matrix, d) 0.2 MPa, composite, e) 0.5 MPa, matrix, f) 0.5 MPa, composite

Rys. 4. Porównanie powierzchni współpracujących dla różnych obciążeń: a) 0,1 MPa, osnowa, b) 0,1 MPa, kompozyt, c) 0,2 MPa, osnowa, d) 0,2 MPa, kompozyt, e) 0,5 MPa, osnowa, f) 0,5 MPa, kompozyt



Element	Atomic %
Carbon	10.401
Aluminum	3.025
Titanium	2.442
Iron	84.131

Fig. 5. Chemical analysis of third body on working surface

Rys. 5. Analiza chemiczna wtrąceń na powierzchni współpracującej

The small white particles on the studied surfaces come from the steel counterpart as confirmed in Figure 5, since the material transfer to the composite material occurred during sliding, which was followed by adhesive wear [7]. The chart in Figure 6 presents the dependence of the wear rate on the applied load. The wear behavior is strongly dependent on the load. In general the enhancement in wear resistance for the composite material is more than 2 times in comparison to the matrix. The wear rate of the matrix and the composite fall within the range of $3.5 \div 5.5 \cdot 10^{-4}$ mm^3/Nm and $1.3 \div 2.4 \cdot 10^{-4}$ mm^3/Nm, respectively. Initially, the wear rate increases together with the load but then it declines when the applied load reaches 0.5 MPa. The observed drop in the wear rate for the greater load remains in agreement with the results reported previously by other researchers [16]. It could be caused by the formation of protective sliding tribolayers.

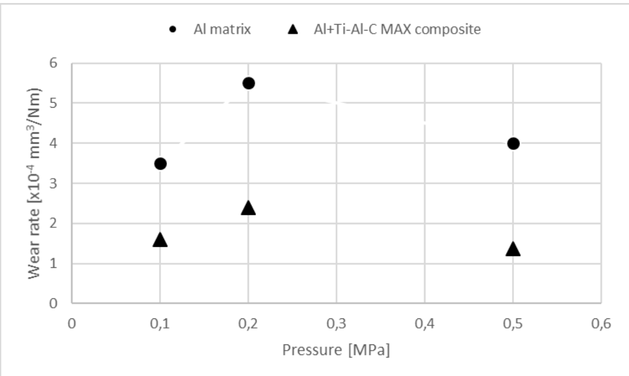


Fig. 6. Wear rate in function of applied load

Rys. 6. Współczynnik zużycia w funkcji przyłożonego obciążenia

In Figures 7 and 8 the cross sections of the tested samples are shown in order to compare the wear behavior. In the Al-Si matrix the main wear mechanism is composed of plastic deformation consisting of scaling and cracking processes, which can lead to the formation of a separated layer on the surface of the sample. During further abrasion this layer will be pulled out and the process will repeat. For the MAX phase composite the wear behavior is principally conventional abrasive wear leading to the pre-fracture and extraction of MAX phase platelet fragments. Although according to El-Raghy et al. [6] since the coarse platelets of MAX phases cannot be easily pulled out, they increase the wear resistance better than fine particles. Moreover, before grain extraction delamination also takes place, postponing the fracture process [9]. Such fragments are stacked in the grooves or porosities of the material, leading to the formation of an accumulated debris area. Compacted debris is believed to act as a third body, which results in abrasion of the steel counterpart [8]. According to the classification of tribofilms, it belongs to type IV because it originates from the composite sample. Figure 8a and 8b show the SEM micrographs of the Al-Si matrix, while 8c and 8d refer to the composite material. In Figure 8d the abrasive deformation of MAX phase reinforcement is presented.

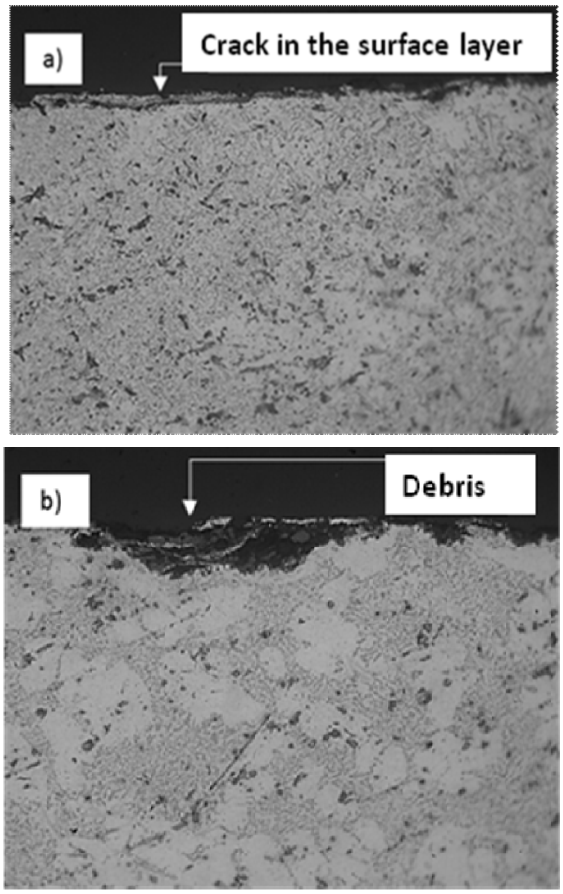


Fig. 7. View of cross sections of samples: a) Al-Si matrix, b) MAX phase + Al-Si composite material

Rys. 7. Widok przekrojów próbek: a) osnowa Al-Si, b) materiał kompozytowy na osnowie stopu Al-Si wzmocniony fazami typu MAX

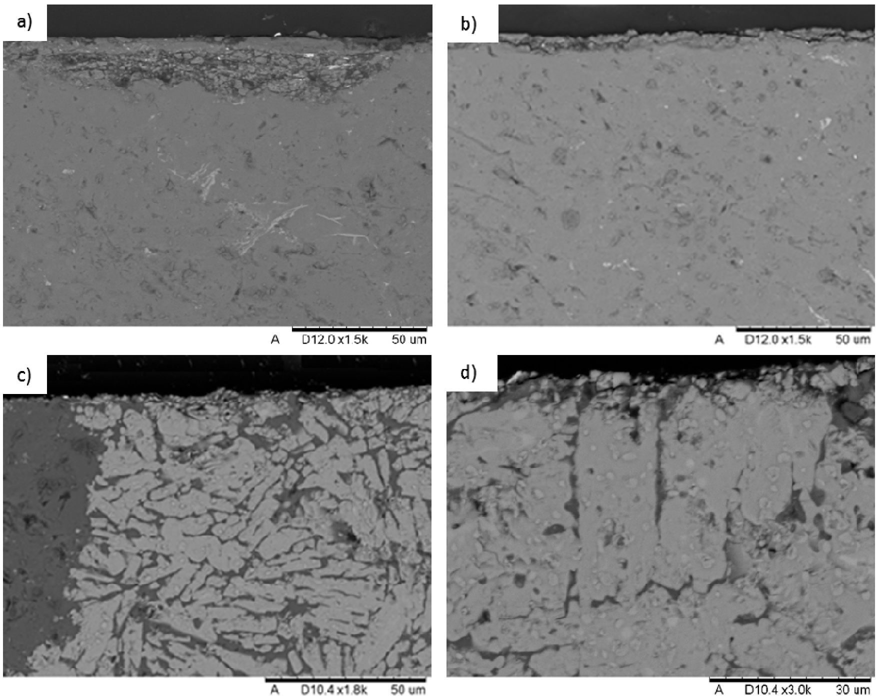


Fig. 8. SEM micrographs of cross sections of samples: a) Al-Si matrix - debris, b) Al-Si matrix - separated layer after crack, c) MAX phase + Al-Si composite material, d) MAX phase + Al-Si composite material - degradation of MAX phase platelets

Rys. 8. Mikrografie SEM przekrojów próbek: a) osnowa Al-Si - skumulowany skruszony materiał, b) osnowa Al-Si - oddzielona warstwa materiału po pęknięciu, c) materiał kompozytowy na osnowie stopu Al-Si wzmocniony fazami typu MAX., d) materiał kompozytowy na osnowie stopu Al-Si wzmocniony fazami typu MAX - wykruszające się płytki faz typu MAX

CONCLUSIONS

Ti_2AlC and Ti_3AlC_2 MAX phase composites were successfully fabricated by squeeze casting infiltration with Al-Si alloy of porous preforms manufactured with the use of microwave assisted self-propagating high-temperature synthesis. The effect of the load applied during sliding was studied. After performing tribological reciprocating pin-on-flat tests for three different loads (0.1, 0.2, 0.5 MPa), it was confirmed that the composite materials exhibit more than two times higher wear resistance than that of the matrix itself. The wear rate of the matrix fall within the range of $3.5\div5.5\cdot10^{-4}$ mm^3/Nm, while for the composite material - $1.3\div2.4\cdot10^{-4}$ mm^3/Nm. Furthermore, the wear of the counterpart was also tested. In the Al-Si matrix the main wear mechanism is based on plastic deformation composed of scaling and cracking processes, while for the MAX phase composite it is principally abrasive wear leading to energy-dissipating mechanisms such as pre-fracture, delamination and extraction of MAX phase platelet fragments. The entrapped debris between the tribocouple acts as a third-body causing further abrasion.

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