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KOMPOZYTY
COMPOSITES

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Al/CF COMPOSITES OBTAINED BY INFILTRATION METHOD

Aluminium alloys reinforced with carbon fibres can find applications in a variety of light constructions operating in complex load conditions. The methods of obtaining these types of composites are mainly based on liquid-phase methods, where the connection between the composites is achieved as a result of wetting the reinforcement phase with a liquid metal. For these processes, the aluminium matrix should be characterized by high strength, the possibility for heat treatment and adequate technological properties (high castability, good wettability of carbon fibre surface by molten metal). A following important aspect in the Al/CF discussed system is a reduction of reactivity between the liquid aluminium and carbon fibre.

In the article, the results of infiltration tests on nickel coated carbon performs by a liquid Al alloy have been presented. In the examinations, aluminium with a silicon alloy (226D) modified by magnesium and strontium were used. The infiltration process was carried out on a Degussa press. The manufactured composite plates were characterized a regular shape, without surface casting defects. The nickel coating prevents the destruction of the fibres, but the reaction of Ni with the liquid aluminum alloy in the boundary area leads to the precipitation of a ductile phase of the Al-Ni system. Decohesion takes place among the matrix and fibres, which allows the supposition that the composite will be characterized by good mechanical properties. This requires experimental verification, which is planned in the successive stage of the research in the project "3D-textile reinforced aliminium matrix composites (3D-CF/Al-MMC) for complex stressed components in automobile applications and mechanical engineering" in the frames of the programme supported by the Polish Ministry of Science and Higher Education and by the DFG in Germany in the Polish-German Bilateral Project.

Keywords: **AMMCs, carbon fibers, composite structures, infiltration process**

KOMPOZYTY Al/CF OTRZYMANE METODĄ INFILTRACJI

Stopy aluminium wzmacniane włóknami węglowymi mogą znaleźć zastosowanie w wielu lekkich konstrukcji, pracujących w złożonych warunkach obciążenia. Metody wytwarzania tego rodzaju kompozytów są oparte głównie na metodach ciekłofazowych, gdzie połączenie pomiędzy komponentami uzyskuje się w wyniku zwilżania fazy zbrojenia przez ciekły metal. Dla tego typu procesów aluminiowa osnowa powinna charakteryzować się wysoką wytrzymałością, możliwością przeprowadzenia obróbki cieplnej i odpowiednimi właściwościami odlewniczymi (wysoką lejnością, dobrą zwilżalnością powierzchni włókien węglowych, niską lepkością). Kolejny ważny aspekt w omawianym układzie komponentów to konieczność obniżenia reaktywności występującej pomiędzy ciekłym stopem aluminium a włóknami węglowymi.

W artykule przedstawiono wyniki badań procesu infiltracji ciekłym stopem Al tkanin węglowych z powłoką niklową. Do infiltracji tkanin węglowych zastosowano stop aluminium z krzemem (226D) modyfikowany dodatkami magnezu i strontu. Proces infiltracji przeprowadzono na prasie Degussa. Wytworzone płytki kompozytowe charakteryzowały się regularnym kształtem bez powierzchniowych wad odlewniczych. Potwierdzono korzystny wpływ powłoki niklowej, która zabezpiecza włókna przed zniszczeniem. Jednak reakcja Ni z ciekłym stopem aluminium w obszarze granicznym prowadzi do powstania kruchych faz z układu Al-Ni. Zniszczenie materiału kompozytowego zachodzi poprzez osnowę i włókna, co pozwala przypuszczać, że kompozyty będą charakteryzowały się dobrymi właściwościami mechanicznymi. Wymaga to jednak dalszej weryfikacji eksperymentalnej planowanej w kolejnych etapach badawczych. Prace zostały zrealizowane w ramach projektu finansowanego w programie DFG: „Kompozyty o osnowie aluminiowej ze wzmocnieniem tekstylnym typu 3-D (3D-CF/Al-MMC) dla elementów podlegających złożonym obciążeniom w przemyśle samochodowym i w budowie maszyn".

Słowa kluczowe: **AMMCs, włókna węglowe, struktura kompozytów, infiltracja**

INTRODUCTION

Aluminium alloys reinforced with carbon fibres can find their application in a range of light constructions operating in complex load conditions. The methods of obtaining these types of composites are mainly based on liquid-phase methods, where the connection between the composites is achieved as a result of wetting the reinforcement phase with a liquid metal [1-7]. In the case of aluminium alloys, the main problem encountered while executing liquid-phase processes is the low wettability in the Al/CF system limited additionally by an oxide film which forms on the alloy surface. The improvement of wettability is possible through the right selection of chemical composition of the alloy on the one hand, and the production of suitable protective coatings on the carbon fibres on the other hand [8, 9]. Pressure infiltration and pressing in liquid crystal state methods (squeeze casting) are most often used to produce aluminium-carbon fibre composites [3]. Pressing in the liquid crystal state (squeeze casting) requires the use of special presses to generate very-high-press pressures (50÷100 MPa). Pressures which are that high may damage the fibres or lead to an uneven distribution of reinforcement in the composite. They do not prevent the formation of air voids which arise as a result of gas confinement in the porous pre-mould during infiltration with a liquid metal. In the case of pressure or gas-pressure infiltration, it is possible to avoid this effect by reducing the pressure in the mould in which the porous shape of the reinforcement was placed and into which a liquid metal is forced as a result of the gas pressure [6, 10-12]. In this case, the time of contact between the liquid metal and the reinforcement, as well as adequate preparation of the matrix and carbon fiber components have essential importance. In our experimental works, we confirmed that the reaction between the aluminium and the carbon fibre can bring about a brittle $A1_4C_3$ phase.

$$4Al_{(l)} + 3C_{(s)} \rightarrow Al_4C_{3(s)}$$

$$\Delta F_{933K} = -172 \text{ kJmol}^{-1} \quad (1)$$

Figure 1 shows the composite structure with the aluminium alloy matrix containing a carbon fibre - without coating - which was formed in the infiltration process in the Degussa induction-vacuum press. The observations of the structure made clearly show the building-up of aluminium carbide on the fibre-matrix boundary and its diffusion into the aluminium alloy. The hydrophilic and unstable $A1_4C_3$ carbide, which is formed as a result of the reaction of carbon fibres with a liquid aluminium (1), is destroyed in contact with steam - which as a consequence reduces the properties of the composite considerably and disqualifies the composite obtained as a construction material [8, 9, 13].

RESEARCH ASSUMPTIONS

On the basis of literature data and the authors' own research, three fundamental ways to possibly improve the wettability and reduce the reactivity in the aluminium-carbon fibre were selected [10-12, 14-17].

The first one is the adequate modification of the chemical composition of the matrix, which lies in introducing alloy additives to improve the wettability of the fibre surface, reduce the surface tension or take part in the *in-situ* reactions in the synthesis of compounds which can play the part of diffusion barriers. The limitation of reactivity in the Al/CF system is possible when the matrix alloy contains at least 7% wt. of silicon. The silicon additive reduces the tendency for the aluminium carbide to form on the boundary of phases. However, magnesium, strontium and titanium additives should provide a reduction of the surface tension and wetting angle. Additional chemical elements such as Mg and Ti reduce the reactivity of the Al/CF system, producing more probable phases from the thermodynamics point, that is: MgO, $MgA1_2O_4$ or TiC [13]. The second way has to do with modification of the carbon fibre surface, which lies in producing (forming) a layer that constitutes a diffusion barrier for the reaction between aluminium from the composite matrix and the reinforcement. As it turns out, from the review of literature, the latest research carried out in many research centers is related to SiC, Ni and Cu coatings [8, 9, 17].

The third one, which is important from the point of selecting the parameters concerning the production of a composite, is the optimization of technological conditions to reduce the temperature and reduce the time of processing fibre infiltration with an Al liquid alloy as much as possible. The solution to the problems described reflects the main trends of research within the DFG program in the project: “3D-textile reinforced Al-matrix composites (3D-CF/Al-MMC) for complex stressed components in automobile applications and mechanical engineering”. In the article, selected results of research achieved by the Department of Material Technology at the Silesia Technical University were presented.

a) b) c)

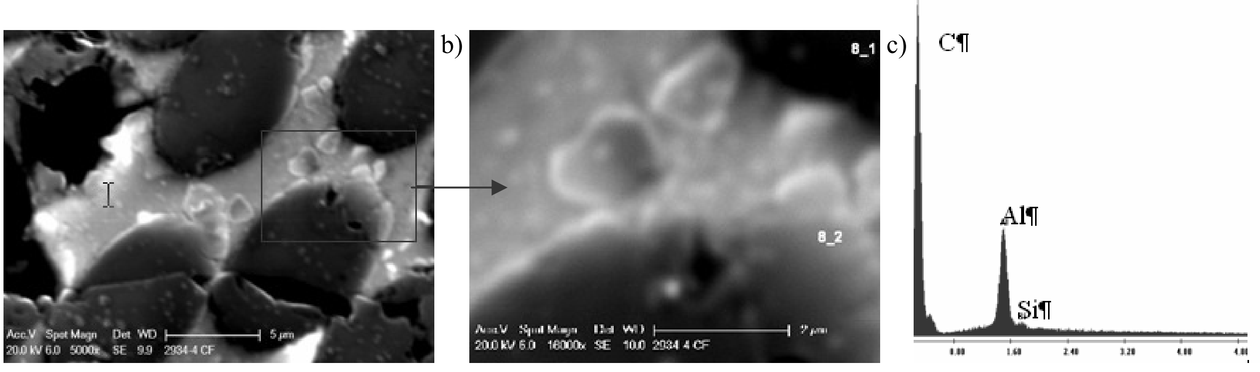


Fig. 1. Al/CF composite: a) and b) SEM micrograph showing destruction of carbon fibers by reaction of liquid aluminium and growth of carbides on boundary between phases; c) EDX spectrum from 8_2 selected points (*authors' own research*)

Rys. 1. Kompozyt Al/CF: a) i b) mikrostruktura pokazująca destrukcję włókien węglowych poprzez reakcję z ciekłym aluminium i narastanie węglików na granicy rozdziału faz; c) EDX z punktu 8_2 (*badania własne*)

The main objective of the research carried out is to develop a chemical composition of Al alloy for the matrix of Al/CF composites received as a result of a porous fibrous structure infiltrated with a liquid matrix in the process of pressure and gas infiltration (GPI) [6, 7, 10-12, 16, 17]. It was assumed that the matrix material must ensure the right wetting conditions, react with carbon fibres to the least possible extent and it must also have the right technological properties (castability, viscosity, solidification time) that the conditions of the infiltration process depend on.

EXPERIMENTAL PROCEDURE

Materials for tests

Taking into consideration the preliminary technological assumptions: the application of methods of pressure infiltration (Degussa induction-vacuum press), gas-pressure infiltration (GPI) and the methods of pressure die-casting, as the basic material of the matrix, the alloy AlSi9Cu(Fe) was selected (marked as 226D). In the background of the eutectic α(Al)+β(Si) and the solution α(Al), grey silicon precipitates are visible, (Fig. 2a). Carbon preforms were used as reinforcement prepared in the Institute of Lightweight Engineering and Polymer Technology (ILK), TU Dresden [6]. For the preform preparation, carbon fibres with an Ni coating produced by the TENAX firm (HTS40 A23 12K) were applied, (Fig. 2b,c).

Modification of matrix alloy

The alloy additives were selected in such a way as to improve the wettability of the surface of carbon fibres by the aluminium alloy and to achieve the proper strength and technological properties taking into consideration the heat treatment of the composite at a later stage. AlMg25%, AlSr10% and TiBAl (having weight composition 4.8% Ti and 1.0% B, 0.2% Fe, 0.1% Si) master alloys were chosen to modify the chemical composition. The modification was carried out in such a way as to change the chemical composition of the basic alloy by introducing 1% and 2% Mg, 0.5% Ti, 0.1% B and 0.03% Sr of the weight ratio, respectively. Moreover the modification, and preparation of the basic alloy also included its refining and degassing [15]. It was found that the modification through alloy additives and, irrespective of their participation and chemical composition, improves the alloy castability in comparison to the basic alloy (Fig. 3). The best castability was achieved in the modified alloys of 1% Mg and 1% Mg + 0.03% Sr. The introduction of both strontium and magnesium or titanium into the basic alloy caused refining of its structure. The observations of the structure of modified alloys indicate diversification during the disintegration of phase α and the size of the eutectic silicon crystals depending on the kind of modification proposed. Depending on the chemical composition, the structure of silumin is built in different proportions from the grain mixture of solid solution α, the eutectic (α+Si) and precipitations of the phases of intermetallic compounds from the Cu-Al, Fe-Al systems and from the Mg_2Si phase. The most beneficial structure - with a high level reduction of eutectic - was achieved by using strontium as a modifying additive in the amount of 0.03%. The selected structures are demonstrated in Figure 4.

The influence of the type of modification and the structure refinement on the mechanical properties was confirmed by the results of strength tests. The summary results of the tensile strength test of the samples in the state after casting are presented on in Figure 5a. It was found that the modifiers increased the tensile strength of the samples in comparison to the initial 226D alloy, the ultimate tensile strength of which does not exceed 140 MPa. The highest tensile strength (202 MPa) was achieved by the alloy having a great eutectic disintegration that is modified with strontium. Beneficial strength properties were also achieved in the alloys modified with titanium and boron (UTS = 200 MPa), whose structure is shown in Figure 4c. The lowest strength properties, which were near the R_m level in the initial alloy, were achieved in the alloy with the additive of 2% Mg (UTS =142 MPa), which can be caused by the formation of a brittle Mg_2Si phase (Fig. 3f). In spite of the beneficial effect of magnesium from the point of wettability in the Al-C system, it is necessary to reduce its content because of the strength properties. As the tests carried out showed, the modified 1% Mg alloy is characterized by a better strength at a 180 MPa level (Fig. 5).

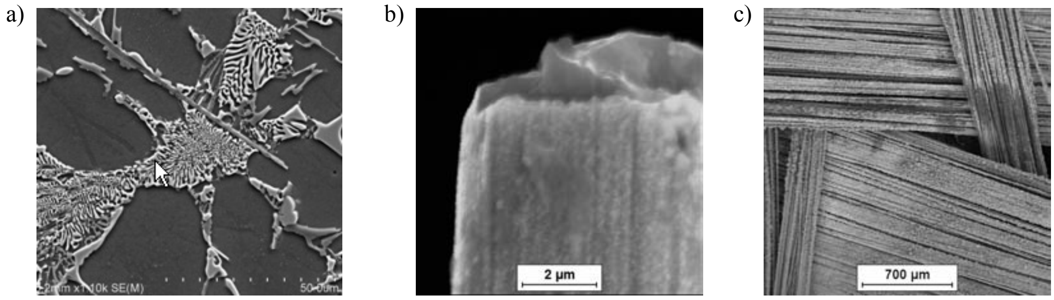


Fig. 2. Structure of AlSi9Cu(Fe) matrix alloy in initial state: a) SEM image. Structure of HTS40 A23 12K carbon fibers: b) view of single fiber with nickel coating, c) view of carbon fibers weave

Rys. 2. Struktura stopu osnowy AlSi9Cu(Fe) w stanie wyjściowym: a) obraz SEM. Struktura włókien węglowych HTS40 A23 12K: b) widok pojedynczego włókna z powłoką niklową; c) widok splotu włókien węglowych

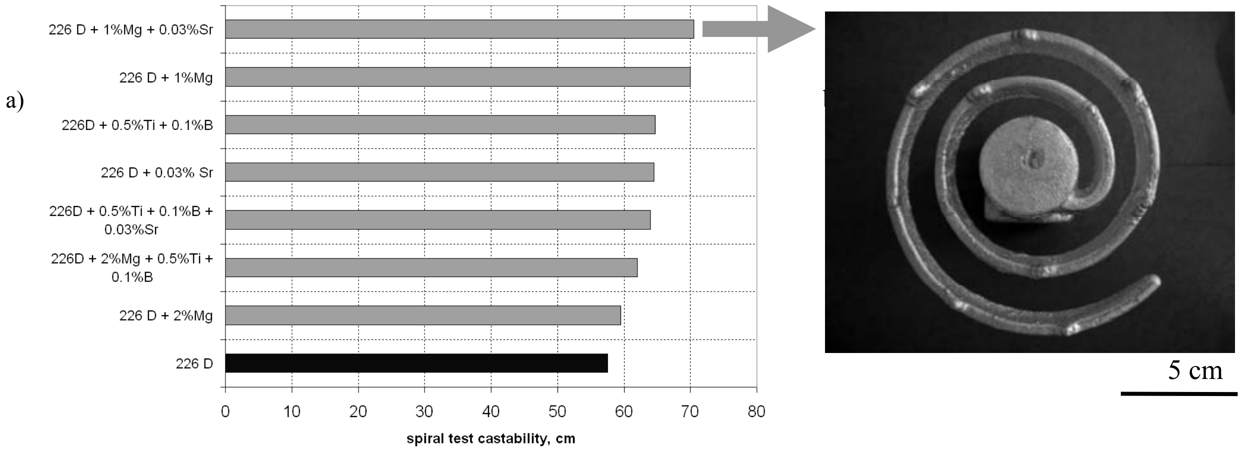


Fig. 3. a) Comparison of castability tests results of base AlSi9Cu(Fe) alloy and inoculated alloys; b) view of spiral castability test obtained for AlSi9Cu(Fe) alloy modified by 1% Mg+0.03% Sr

Rys. 3. a) Porównanie wyników prób lejności bazowego stopu AlSi9Cu(Fe) i stopów modyfikowanych; b) widok spirali lejności dla stopu AlSi9Cu(Fe) modyfikowanego 1% Mg+0.03% Sr

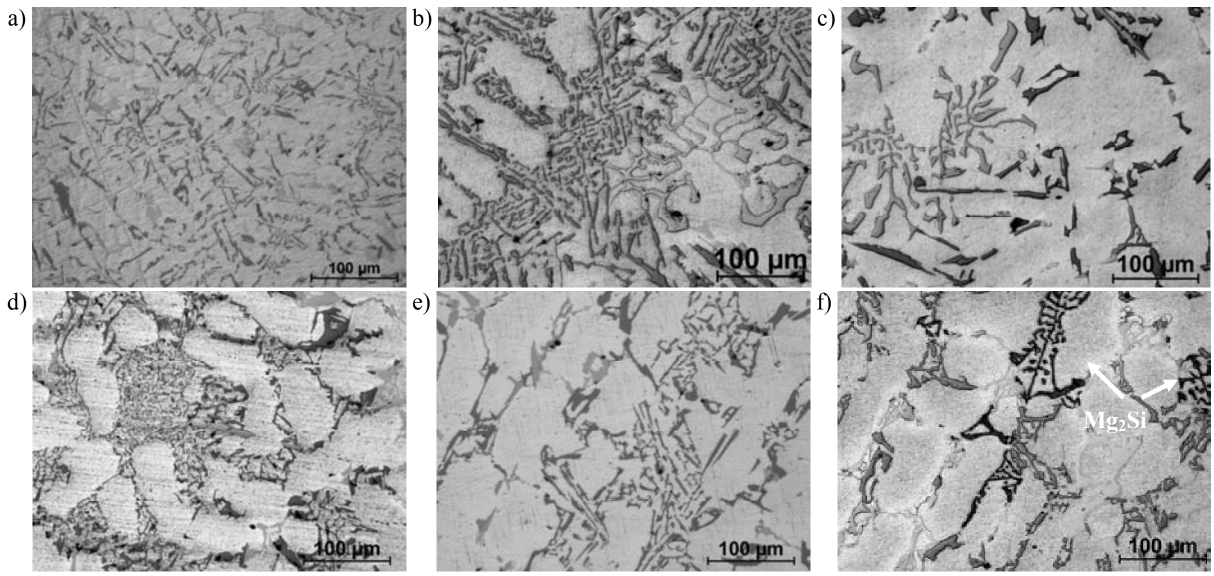


Fig. 4. Microstructure of AlSi9Cu(Fe) alloys: a) in initial state and modified: b) 0.03% Sr addition; c) 0.5% Ti + 0.1% B additions; d) 1% Mg+ + 0.03% Sr additions; e) 1% Mg addition; f) 2% Mg addition

Rys. 4. Mikrostruktura stopów AlSi9Cu(Fe): a) w stanie wyjściowym i modyfikowanym: b) dodatkiem 0.03% Sr; c) dodatkiem 0.5% Ti + 0.1% B; d) dodatkiem 1% Mg + 0.03% Sr; e) dodatkiem 1% Mg; f) dodatkiem 2% Mg

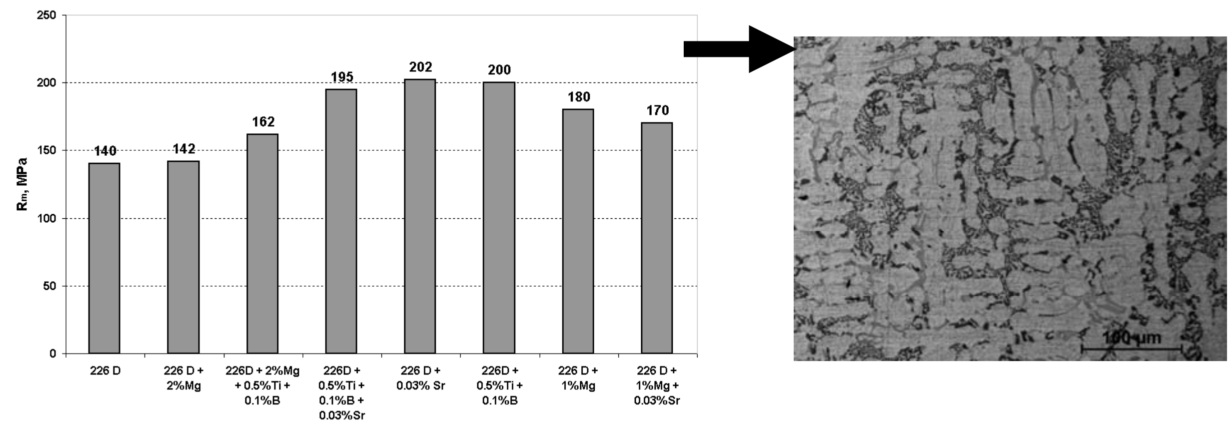


Fig. 5. a) Comparison of tensile strength of AlSi9Cu(Fe) alloys at initial and modified state; b) structure of AlSi9Cu(Fe) alloy modified by 1% Mg + 0.03% Sr

Rys. 5. a) Porównanie wytrzymałości stopu AlSi9Cu(Fe) w stanie wyjściowym i po modyfikacji, b) struktura stopu AlSi9Cu(Fe) modyfikowanego 1% Mg + 0.03% Sr

Process of producing Al/CF composites

The process of carbon textile infiltration with a nickel coating was performed in a graphite mould taking advantage of the Degussa press [11]. The selected alloys of the matrix were superheated to a temperature of T = 720°C and under a pressure of p = 15 MPa, at a time not longer than 15 minutes and forced into the mould where the preform made of carbon fibres was placed. From the infiltration process, composite plates $Al/CF_{(Ni)}$ with the dimensions of 40x40x2.6 mm were received. The scheme illustrating the experimental set-up is shown in Figure 6.

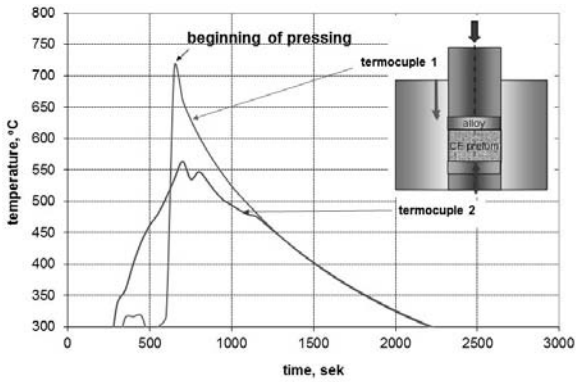


Fig. 6. Liquid infiltration cycle and schematic illustration of experimental set up

Fig. 6. Krzywe cyklu nasycania i schemat procesu infiltracji

Structure of composite received in pressure infiltration process

The test results of the composite microstructure received in the process of pressure infiltration, one layer and two layers of carbon textile perform obtained from commercial fibres with nickel coatings (HTS 40 A23 12K), are presented in Figures 7-10. The composite plates obtained featured a regular shape without surface casting defects. The presence of carbon fibres with the nickel coating caused the anticipated changes in the structure of the alloy matrix. The surface distribution of elements revealed in the structure observed, made with the application of SEM+EDX is presented in Figure 7. In the matrix, a traceable presence of nickel was found as well as irregular bright phases containing Al and Ni, which indicate the dissolving of the nickel coating and precipitation in the form of intermetallic phases of the Al_xNi_y type.

The dark precipitations which can be seen in Figure 7a, consisted of silicon (Fig.7b) and magnesium (Fig. 7c), indicating the presence of the intermetallic compound Mg_2Si. In turn, small grey precipitations, which can be seen in Figure 7a, show the silicon plates confirmed by the picture of the surface distribution of this element shown in drawing 7b. In the composite matrix, numerous complex compounds of the Al-Ni-Cu, Al-Mg-Si, Al-Ni-Fe-Mn and also Al-Si-Fe-Cu type, which correlate with the chemical composition of the alloy were identified.

a) b) c) d)

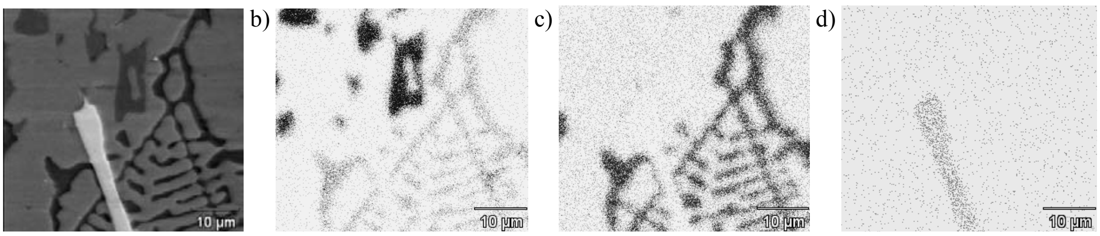


Fig. 7. Surface distribution of elements in $AlSi9Cu(Fe)/CF_{(Ni)}$ composite matrix alloy: a) SEI; b) SiK; c) MgK; d) NiK

Fig. 7. Rozkład powierzchniowy pierwiastków w osnowie kompozytu $AlSi9Cu(Fe)/CF_{(Ni)}$: a) SEI; b) SiK; c) MgK; d) NiK

a) b)

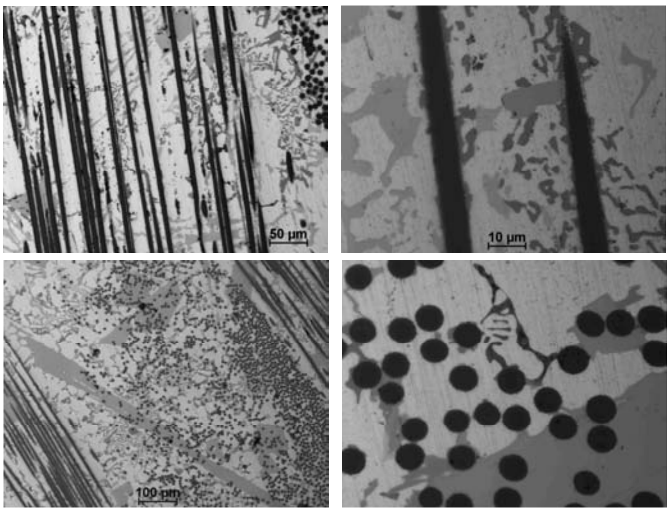


Fig. 8. Structure of composites reinforced by carbon textile obtained by pressure infiltration in Degussa press (fibres coated with nickel): a) single layer of $CF_{(Ni)}$ reinforcement; b) two layers of $CF_{(Ni)}$ reinforcement

Fig. 8. Struktura kompozytów wzmacnianych tkaniną węglową (włókna pokryte niklem) uzyskanych przez infiltrację w prasie Degussa: a) jedna warstwa zbrojenia $CF_{(Ni)}$; b) dwie warstwy zbrojenia $CF_{(Ni)}$

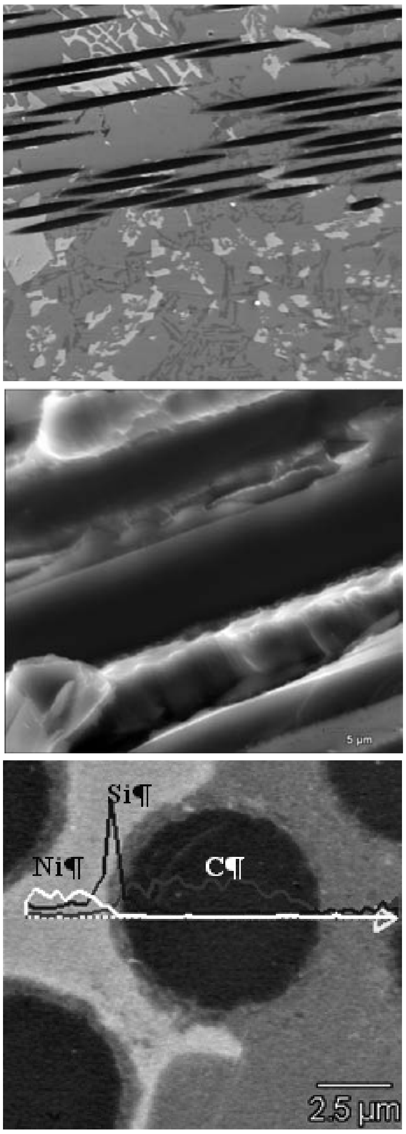
a)
b)
c)


Fig. 9. SEM micrographs of AlSi9Cu(Fe)/$CF_{(Ni)}$ composite microstructures: a) OM; b) fracture surface, SEM; c) linear distribution of elements on carbon fibres-Al matrix interface, SEM

Fig. 9. Mikrostruktura kompozytu AlSi9Cu(Fe)/$CF_{(Ni)}$: a) OM; b) powierzchnia przełomu, SEM; c) rozkład liniowy pierwiastków na granicy włókno węglowe-osnowa Al, SEM

On metallographic specimens made of the Al/CF(Ni) composite, a good filling of the space between the fibres with the matrix metal and a continuous connection on the carbon fibres-matrix boundary were observed. On the basis of the observation of the composite microstructure, an alternate fibre arrangement was found - characteristic for the rowing weave applied and the number of layers consisting of the carbon preform (Fig. 8). A non-porous matrix between single bundles of rowing was found, which infiltrated the space between the fibres at the same time. This shows good wetting on the macroscopic scale. Similar observations of the metallographic specimens with the use of a scanning microscope showed good adhesion of the matrix to the fibres and a good connection on the phase boundary as a consequence (Figs. 9 and 10).

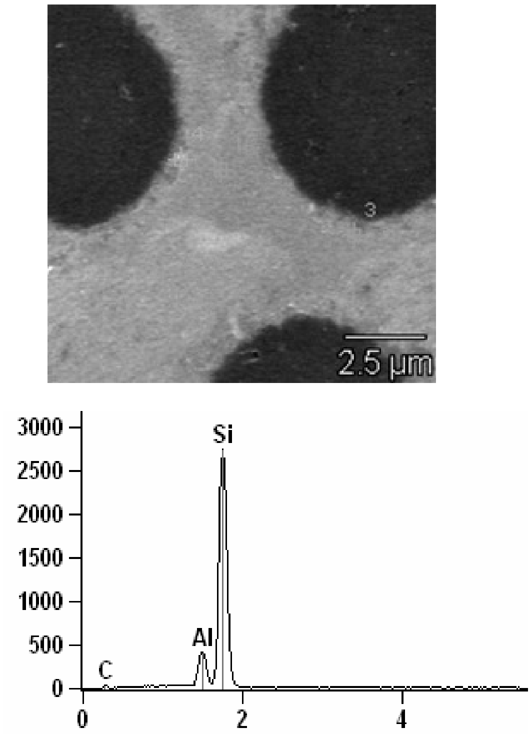
a)
b)


Fig. 10. a) SEM micrographs of AlSi9Cu(Fe)/CF(Ni) composite microstructure; b) EDX from point 3

Fig. 10. a) Mikrostruktura kompozytu AlSi9Cu(Fe)/$CF_{(Ni)}$; b) EDX z punktu 3

The preferred connection between the components was obtained due to proper process parameters. It was confirmed in the presented images of a fracture in which there was no separation of the bundle fibers or their drawing, and the process of destruction ran through the fibers (Fig. 9b). The microanalysis of the chemical composition of a linear phase separation on the border of the carbon fiber-matrix Al (Fig. 9c) showed an increased concentration of silicon, and the presence of nickel, which is confirmed by the analysis made at point 3 (Fig. 10). In the border area, there was no degradation of the fibers nor visible products of the reaction of carbon with aluminium.

CONCLUSIONS

1. In the infiltration pressure process, the connection in the aluminium carbon fiber system not only determines the wetting conditions, but also the chemical interaction between the components.
2. In the reaction of the liquid Al and uncoated carbon fibres, an Al_4C_3 phase formed, which improves the wetting conditions in the system, but it definitely reduces the strength of the material.
3. Nickel coating prevents the destruction of the fibres, but the reaction of Ni with liquid aluminum in the area leads to the precipitation of a ductile phase of the Al-Ni system. Reducing the undesirable effect appears to be possible after the

change of the technological parameters of the infiltration process. For a full evaluation of the durability, strength and stability of the composite structure and boundary, further investigations using transmission electron microscopy techniques are planned.
4. Currently, researches are being performed on the optimization of the temperature and time of infiltration, so as to obtain favorable wetting of the fiber-matrix system and simultaneously reduce the formation of brittle phases.
5. Further research will focus on the development of heat treatment parameters of Al/CF composites.

Acknowledgments

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