

# Modern composite materials manufactured by pressure infiltration method

#### L.A. Dobrzański\*, M. Kremzer, M. Drak

Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland \* Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

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## ABSTRACT

**Purpose:** The purpose of this paper is to present the technique of manufacturing the composite materials based on porous ceramic preforms infiltrated by liquid aluminium alloy and examination of the structure and corrosion resistance of those materials.

**Design/methodology/approach:** The material for investigations was manufactured by pressure infiltration method of ceramic porous preforms. The eutectic aluminium alloy EN AC – AlSi12 was use as a matrix while as reinforcement were used ceramic preforms manufactured by sintering of  $Al_2O_3$  Alcoa CL 2500 powder with addition of pore forming agents as carbon fibres Sigrafil C10 M250 UNS manufactured by SGL Carbon Group Company. To determine the corrosion resistance, corrosion test by potentiodynamic method were made consisting in registering the anode polarization curves using the measurements system consisting of the potentiostat PGP-201 working with the Radiometer Copenhagen VoltaMaster 4 software.

**Findings:** The received results show the possibility of obtaining the new composite materials with required structure and corrosion resistance depends of the volume fraction of the reinforcing phase.

**Practical implications:** The composite materials manufactured by the developed method can find application among the others in automotive, aircraft or marine industry as the alternative material for elements fabricated from unreinforced aluminium alloys.

**Originality/value:** The obtained results show the possibility of manufacturing the composite materials by the method of porous sintered framework pressure infiltration based on the ceramic particles, characterized with the better corrosion resistance than aluminium alloy used as the matrix.

Keywords: Composite materials; Infiltration; Structure; Corrosion resistance

## **1. Introduction**

Light metal matrix composite materials with ceramic reinforcements have received extensive studies during the past decades because of their improved mechanical properties. Among them, the  $Al_2O_3$  particulate reinforced aluminium composites ( $Al_2O_3/Al$ ) may belong the most successful type of MMCs. The  $Al_2O_3$  reinforced aluminium alloys matrix composites have been increasingly used in the automotive or aircraft industry because of their high strength-to-weight ratio, good castability and better tribological properties over the unreinforced alloys [16, 17].

Composite is a material formed by two or more different chemically components, and the properties of the final material are better than those of components separately. Composite materials are different from many others available materials because of the possibility to design their properties accordingly to the closest desired requirements for the condition of the practical use. Producing and estimating the properties of the materials is not simple and is connected with complicated engineer's calculations because it have to be taken into consideration many parameters among which are: components properties, their portion, type of connection between them, the used technique of manufacturing and its parameters, furthermore treatment and many others [2, 5, 7, 8, 13].

Very often elements of different devices made from composite materials work not only in the conditions of carried out high loads or in the conditions of abrasive wear but also in extreme corrosion environments. Aluminium and its alloys are characteristic of high corrosion resistance in many conditions; thin and strongly adhering aluminium oxide layer developing spontaneously features a protection and slows down the electrochemical corrosion processes, deciding their corrosion resistance but the introducing of reinforcement phase can also significantly change their corrosion resistance consisting higher corrodibility. That is cause by many factors such as:

- creating of galvanic cell between active aluminium matrix, and more noble material of reinforcement,
- creating of intermetalic phases (good conducting zones on the passive layer),
- high density of dislocation and residual stress around the reinforcement phase,
- porosity on the matrix/reinforcement boundary,
- technology of composite material manufacturing and the shape reinforcement phase.

Analyzing the results of research works involving the corrosion of aluminium matrix composite materials the negative influence of reinforcement phase on their corrosion resistance can be estate [1, 3, 4, 6, 9-12, 14, 15, 18].

Aluminium matrix composite materials exhibit excellent strength, modulus, wear and corrosion resistance, and damping capacity. These materials have been proposed for many industrial applications in the recent few years. In order to examine the susceptibility of these materials in corrosive environments, corrosion studies need to be performed. Hence the goal of a work is to present the technique of manufacturing the Al-Al<sub>2</sub>O<sub>3</sub> composite materials by pressure infiltration method of ceramic performs with liquid alloy and examination of structure and corrosion properties of those materials.

## 2. Material and experimental procedure

#### 2.1. Material and technology

The material for investigation was produced by he method of pressure infiltration of porous ceramic preforms with liquid aluminium alloy. The composite's matrix consisted of euthetic alloy EN AC – AlSi12 and as the reinforcement the porous ceramic frameworks consisted of sintered  $Al_2O_3$  particles were used.

The ceramic preforms were manufactured by sintering of powder  $Al_2O_3$  Condea CL 2500 with the addition of pores and canals structure forming agent in the form of carbon fibres Sigrafil C10 M250 UNS of Company SGL Carbon Group. The properties of the used carbon fibres were shown in Table 1.

The addition of the carbon fibres was 30, 40 and 50 % of weight. Into  $Al_2O_3$  suspension were added the addition of anti-

forming agent of the set of carbon fibres Dolapix CE 64 of Company Zschimmer und Schwarz GmbH Company, eliminating their electrostatic interactions. In order to make pressing easier, 1% polyvinyl alcohol Moviol 18-8 solvable in water was added. The prepared powder was dried by freezing and water sublimation method under the lowered pressure. Next the mixture was sifted through the sieve of 0.25 mm. To activate the polyvinyl alcohol, the powders were humidified with distillated water, packed into the plastic bags and put away for 24 h. The powders were uniaxially pressed in the hydraulic press "Nelke" in steel mold with the inside diameter of 30 mm. The maximum pressure was 100 MPa and pressing time was 15 s. Compacts were sintered in "Gero" pipe furnace in air atmosphere (20 l/min). The temperature during the sintering process ensured the carbon fibres degradation (heating by 10 h in temperature 800 °C) and Al<sub>2</sub>O<sub>3</sub> powder sintering in temperature of 1500 °C by 2 h. The porosity of the ceramic performs depends on the carbon fibres content 69% at 30% of carbon fibres addition, 75% at 40% of carbon fibres addition and 80% at 50% of carbon fibres addition, respectively.

Obtained ceramic preforms were heated in furnace up to temperature of 800 °C. Covered by graphite form was warmed up to 450 °C (maximal temperature of the press plates) and then fulfilled with preform and liquid alloy EN AC – AlSi12 with temperature of 800 °C, which chemical composition is presented in Table 2.

Table 1.

Properties of Sigrafil C10 M250 UNS carbon fibres [17]

Fiber	Mean fiber	Fiber	Tensile	Young's	Carbon
diameter	length	density	strength	modulus	content
μm	μm	g/cm <sup>3</sup>	GPa	GPa	%
8	135	1.75	2.5	26	>95

Table 2.

Chemical composition of EN AC-AlSi12 aluminium alloy

Mean mass concentration of elements, wt.%							
Si	Fe	Cu	Mn	Zn	Ti	Others	Al
12	≤0.55	≤0.05	≤0.35	≤0.15	≤0.2	≤0.15	The others

The whole was covered by the stamp and placed in hydraulic plate press Fontune TP 400. The maximum infiltration pressure was 100 MPa and load 120 s. After solidification obtained materials were removed from the form and cool down under pressured air stream. The scheme of MMC's manufacturing process is presented in Fig. 1.

For better identification of materials and cleaner interpretation of their characteristic in tables and figures the following denotation is used:

- C30 composite material reinforced by ceramic preform manufactured by sintering of Al<sub>2</sub>O<sub>3</sub> powder with 30% mass portion of carbon fibres,
- C40 composite material reinforced by ceramic preform manufactured by sintering of Al<sub>2</sub>O<sub>3</sub> powder with 40% mass portion of carbon fibres,
- C50 composite material reinforced by ceramic preform manufactured by sintering of Al<sub>2</sub>O<sub>3</sub> powder with 50% mass portion of carbon fibres.

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Fig. 1. The scheme of MMC's manufacturing process

#### 2.2. Methodology

Metallographic examinations of the composite materials with a luminium alloy EN AC – AlSi12 matrix reinforced by  $Al_2O_3$ porous preforms were made on the light microscope LEICA MEF4A. Specimens were not etched to observe the structure (ceramic phase distribution in the metal matrix) and the infiltration degree.

Corrosion investigations were made by the use of potentiodynamic method (permanent current method) with recording of anodic polarization curves. The measurement were made by the use of PGP-201 potentiostat cooperating with software VoltaMaster 4 Radiometer Copenhagen Company. Corrosive solution with chemical composition 0.1M Na<sub>2</sub>SO<sub>4</sub>, 0.05M NaCl with the pH 7.7 was prepared from analytically pure reagents POCh Gliwice and distillated water. The measurements were made in three electrode glass vessel (electrochemical cell) (Fig. 2).

The electrode was the sample of composite materials or the matrix, reference electrode - saturated calomel electrode, and the auxiliary electrode - the platinum. The preparations of materials to examinations consisted off cut of the samples which were connected with cooper wire and poured with epoxy resin. Samples were grinded and polished applying the metallography preparatic. Measurements were made in room temperature by the changes of potential 1mV/s . Before the registered polarization curves the corrosion potential of material E<sub>cor</sub> was evaluated in non-current conditions by 60 min. The potentiodynamic curves were registered by polarization in the positive value direction with the velocity of potential changes 1mV/s from Ecor value minus 100mV. After obtaining the current density of 1mA/cm<sup>2</sup> the polarization was changed into the negative values with the same velocity, the return curve was registered. On the base of obtained anodic polarization curves, by the use of Tafel's extrapolation method near the corrosive potential, the quantitative data describing the electrochemical corrosion of examined materials were evaluated: corrosion potential  $E_{cor}$  (mV), overpotential  $E_{(i=0)}$ (mV), pitting potential E<sub>n</sub> (mV), density of corrosion current i<sub>cor</sub>  $(\mu A/cm^2)$ , and polarization resistance R<sub>P</sub> (k $\Omega/cm^2$ ).



Fig. 2. Scheme of corrosive measuring position

#### **3. Results and discussion**

Results of metallographic examinations of the composite materials obtained by infiltration of porous ceramic preforms with the EN AC – AlSi12 alloy, carried out on the light microscope are presented in Fig. 3. The observations show the uniform distribution of reinforcing phase (sintered  $Al_2O_3$  Alcoa CL 2500 particles) in matrix. Moreover on the basis of observations it was found that infiltration process takes place at full level what have been proved by the lack of pores not filled with metal.

To obtain the influence of ceramic phase on the corrosion receptivity of composite materials the corrosion test by the potentiodynamical method were investigated. During the measurements the corrosion potential  $E_{cor}$  was evaluated by 60 min (Fig. 4.) and registered the anodic polarization curves (dependence of current density versus potential) for matrix and composite materials (Figs. 5 and 7-9). On The base of obtained anodic polarization curves by the use of Tafel's extrapolation method near the corrosive potential, the quantitative data describing the electrochemical corrosion phenomena of examined materials were evaluated (Fig. 6).

For EC AC – AlSi12 alloy, corrosion potential is equal -655 mV, current density in the passivation range 0.387  $\mu$ A/cm<sup>2</sup> and the corrosion resistance 96.5 kΩ/cm<sup>2</sup>. The best electrochemical properties, in comparison with the matrix shows composite material C30 with the highest portion of reinforcement. Corrosion potential of that material is equal -607mV, current density in the passivation range 0.021  $\mu$ A/cm<sup>2</sup> and the corrosion resistance 656.5 kΩ/cm<sup>2</sup>. The higher reinforcement portion in composite materials, the higher corrosion resistance (Tab. 3). Al<sub>2</sub>O<sub>3</sub> phase, the reinforcement of composite materials, does not conduct the current, so the part of surface, which it stand, does not undergo the electrochemical corrosion.





Fig. 4. Example of corrosion potential changes versus time



Fig. 3. Structure of the composite materials with the aluminium alloy matrix reinforced with the  $Al_2O_3$  porous preforms coated by nickel with different porosity: a) 68.80%, b) 75.60% and c) 80.80%

Fig. 5. Anodic loop: a) and the anodic polarization curve, b) of matrix

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Fig. 6. Example of corrosion parameters evaluated by the Tafel's extrapolation method



Fig. 7. Anodic loop: a) and the anodic polarization curve, b) of C30 material

Table 3.				
Corrosion p	arameters of the	matrix and c	composite	materials

Material	Corrosion potential, E <sub>cor</sub> [mV]	Overpotential, $E_{(i=0)}$ [mv]	Pitting potential, E <sub>n</sub> [mV]	$\begin{array}{c} Corrosion \ current, \ i_{cor} \\ [\mu A/cm^2] \end{array}$	Polarisation resistance, $R_p [k\Omega/cm^2]$
EN AC-AlSi12	-655	-717	26.5	0.387	96.5
C30	-607	-597	255	0.021	656.5
C40	-632	-659	50	0.060	395.6
C50	-658	-668	-56	0.077	193.4



Fig. 8. Anodic loop: a) and the anodic polarization curve, b) of C40 material



Fig. 9. Anodic loop: a) and the anodic polarization curve, b) of C50 material

The value of pitting corrosion potential  $E_n$  (Tab. 3) by which on the surface of samples pitholes are creating, was evaluated on the basis on anodic loops of materials. The highest pitting corrosion receptivity shows material with highest portion of ceramic phase for which the pitting potential is equal 255 mV, when the pitting potential for matrix is equal 26.5 mV. Introducing to the matrix the small portion of reinforcement causes the decrees of pitting corrosion resistivity ( $E_n$  for C50 material is equal -56 mV). The higher portion of ceramic phase, the higher pitting potential up to 50 mV for C40 material, and the highest value 255 mV shows C30 material.

Both zones near the reinforcement and small pore are privileged places for corrosion centre creating and that determinate the pitting corrosion resistivity of composite materials in comparison with matrix. The improvement of that properties, with the higher portion of ceramic phase, is cause by the smaller portion of sample's surface conducting the current. All investigated materials do not show the repassivation, so it was impossible to evaluate the repassivation potential  $E_{cp}$ , under which on the samples surface do not exist the active corrosion centres.

## 4.Conclusions

The metallographic observation of the obtained composite materials shown that the presented technology of fabricating the composite material with the EN AC – AlSi12 alloy matrix reinforced with the  $Al_2O_3$  preforms, obtained by the pressure infiltration, ensures the required structure - uniform distribution of reinforcing phase in the metal matrix and lack of pores in the material.

On base of corrosion tests it was found that the higher reinforcement portion in composite materials, the higher general and pitting corrosion resistance, because the reinforcement phase does not conduct the current, so the part of surface, which it stand, does not undergo the electrochemical corrosion.

The obtained results show the possibility of manufacturing the composite materials by the method of porous sintered framework pressure infiltration based on the ceramic particles, characterized with the better corrosion resistance than aluminium alloy used as the matrix.

#### <u>References</u>

- A. Albiter, A. Contreras, M. Salazar, J.G. Gonzalez-Rodriguez, Corrosion behaviour of aluminium metal matrix composites reinforced with TiC processed by pressureless infiltration, Journal of Applied Electrochemistry 36 (2006) 303-308.
- [2] M.F. Ashby, D.R.H. Jones, Engineering Materials 2 Forming of the structure and properties, Materials Selection, WNT, Warsaw, 1998.
- [3] J. Bieniaś, Aluminium matrix composite materials Chojen structure and corrosive aspects, Materials Engineering 3 (2006) 561-654 (in Polish).
- [4] J. Bieniaś, B. Surowska, M. Walczak, The corrosion characteristics of aluminum matrix composites reinforced with SiC particles, Proceedings of the 12<sup>th</sup> International Scientific Conference "Achievements in Mechanical and Materials Engineering" AMME'2003, Gliwice–Zakopane, 2003, 99-102.
- [5] A. Boczkowska, J. Kapuściński, Z. Lindemann, D. Witemberg-Perzyk, S. Wojciechowski, Composites, Warsaw University of Technology Press, Warsaw, 2003.

- [6] J.M.G. De Salzar, A. Urena, S. Manzanedo, M.I. Barrena, Corrosion behaviour of AA6061 and AA7005 reinforced with Al<sub>2</sub>O<sub>3</sub> particles in aerated 3.5% chloride solutions: potentiodynamic measurements and microstructure evaluation, Corrosion Science 41 (1999) 529-545.
- [7] L.A. Dobrzański, M. Kremzer, A. Nagel, Aluminium EN AC - AlSi12 alloy matrix composite materials reinforced by Al<sub>2</sub>O<sub>3</sub> porous performs, Archives of Materials Science and Engineering 28/10 (2007) 593-596.
- [8] L.A. Dobrzański, M. Kremzer, A. Nagel, Application of pressure infiltration to the manufacturing of aluminium matrix composite materials with different reinforcement shape, Journal of Achievements in Materials and Manufacturing Engineering 24/2 (2007) 183-186.
- [9] L.A. Dobrzański, A. Włodarczyk, M. Adamiak, Corrosion resistance of sintered composite material of EN AW-AlCu4Mg1(A) alloy matrix reinforced by Al<sub>2</sub>O<sub>3</sub> and Ti (C, N), Proceedings of the 3<sup>rd</sup> Scientific Conference "Materials, Mechanical and Manufacturing Engineering" M3E, Gliwice–Wisła, 2005, 217-224 (in Polish).
- [10] L.A. Dobrzański, A. Włodarczyk, M. Adamiak, Properties and corrosion resistance of PM composite materials based on EN AW-Al Cu4Mg1(A) aluminum alloy reinforced with the Ti(C,N) particles, Proceedings of the 11<sup>th</sup> International Scientific Conference "Contemporary Achievements in Mechanics, Manufacturing and Materials Science" CAM<sup>3</sup>S'2005, Gliwice–Zakopane, 2005, CD-ROM.
- [11] L.A. Dobrzański, A. Włodarczyk, M. Adamiak, Structure, properties and corrosion resistance of PM composite materials based on EN AW-2124 aluminum alloy reinforced with the Al<sub>2</sub>O<sub>3</sub> ceramic particles, Proceedings of the 13<sup>th</sup> International Scientific Conference "Achievements in Mechanical and Materials Engineering" AMME'2005, Gliwice–Wisła, 2005, 203-206.
- [12] L.A. Dobrzański, A. Włodarczyk, M. Adamiak, Structure, properties and corrosion resistance of PM composite materials based on EN AW-2124 aluminum alloy reinforced with the Al<sub>2</sub>O<sub>3</sub> ceramic particles, Journal of Materials Processing Technology 162–163 (2005) 27-32.
- [13] A. Dolata-Grosz, J. Wieczorek, Tribological properties of hybryd composites containing two carbide phases, Archives of Materials Science and Engineering 28/3 (2007) 149-155.
- [14] P. Hyjek, P. Kurtyka, S. Wierzbiński, H. Krawiec, Corrosion resistance of Al-Zn-Mg alloy matrix composite materials reinforced by Al<sub>2</sub>O<sub>3</sub> particles, Composites 2 (2005) 43-46 (in Polish).
- [15] K. Łuczak, P. Liberski, J. Śleziona, The influence of volumetric content and particle size on corrosion resistance of aluminium-ceramic particles, Composites 6 (2003) 75-78 (in Polish).
- [16] Z.P. Luo, Y.G. Song, S.Q. Zhang, A TEM study of the microstructure of  $SiC_p/Al$  composite prepared by pressureless infiltration method, Scripta Materialia 45 (2001) 1183-1189.

- [17] K.N. Tandon, Z.C. Feng, X.Y. Li, Wear behavior of SiC particulate reinforced aluminium composites sliding against steel balls under dry and lubricated conditions, Tribology Letters 6 (1999) 113-122.
- [18] A. Włodarczyk-Fligier, L.A. Dobrzański, M. Adamiak, Influence of the heat treatment on properties and corrosion resistance of Al-composite, Journal of Achievements in Materials and Manufacturing Engineering 21/1 (2007) 55-58.