

The influence of Ni-P layer deposited onto Al_2O_3 on structure and properties of Al- Al_2O_3 composite materials

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Received 24.03.2011; published in revised form 01.06.2011

Materials

ABSTRACT

Purpose: The purpose of this work is to present the influence of wettability improvement of sintered Al_2O_3 particles by deposition of Ni-P coating.

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 used 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, SGL Carbon Group Company. The Al_2O_3 was coated with the Ni-P alloy to improve the wettability of sintered particles. Metallographic examinations were made in the transmission electron microscope (TEM).

Findings: The obtained results indicate the possibility of obtaining new materials with all advantageous properties of the particular composite constituents by infiltration of the ceramics with the liquid aluminium alloy.

Practical implications: The composite materials made by the developed method can find application as the alternative material for elements fabricated from conventional materials.

Originality/value: The obtained results show the possibility of manufacturing the composite materials by the pressure infiltration method of porous sintered preforms based on the ceramic particles with liquid aluminium alloy being a cheaper alternative for materials reinforced by fibres.

Keywords: Composites; Wettability; Infiltration

Reference to this paper should be given in the following way:

L.A. Dobrzański, M. Kremzer, J. Konieczny, The influence of Ni-P layer deposited onto Al_2O_3 on structure and properties of Al- Al_2O_3 composite materials, Journal of Achievements in Materials and Manufacturing Engineering 46/2 (2011) 147-153.

1. Introduction

Composite materials reinforced with ceramic particles are most often manufactured by casting methods, powder metallurgy or infiltration of the porous, ceramic preforms with liquid metals alloys being a connection of both methods [1-10]. The squeeze-

casting process, which involves the infiltration of the melt through a preform of fibres, whiskers or particles is a more suitable technique to employ, as it enables: the possibility of obtaining the composite products of precise shape mapping and the high-quality surface (near net shape), adaptation of the process to the mass scale production, free variability of reinforcing phase and matrix

material, high-productivity process with relatively low-cost of production, the possibility of local reinforcement of the product. But to ensure good properties of final product many parameters should be properly selected: preheating temperature of preform, temperature of liquid metal, infiltration pressure, time and speed. The big influence on composite properties have also preform characteristic (volume fraction, morphology and orientation of reinforcement) [11-16].

The connection of aluminum's properties as a matrix and particles or ceramic fibres as reinforcement in composite materials is dependent on the created during the technological process structure metal-ceramics. Taking into consideration fact, that aluminum does not free wet aluminum oxide, different technological treatments are employed, allowing to obtain resistant connection. To the main treatments can be included: increase of infiltration pressure, and deposition of coating into the reinforcement material. One of the coating conception, mainly described in literature, is deposition of Ni-P coating on the Al_2O_3 [17-20].

The most commonly described characteristic of the wettability degree of ceramics by liquid metal is the wettability angle θ (in the range 0° - total wettability to 180° no wettability). Conventionally systems metal-ceramic are divided into wettable system $\theta < 90^\circ$ and not wettable ones $\theta > 90^\circ$ (Fig. 1).

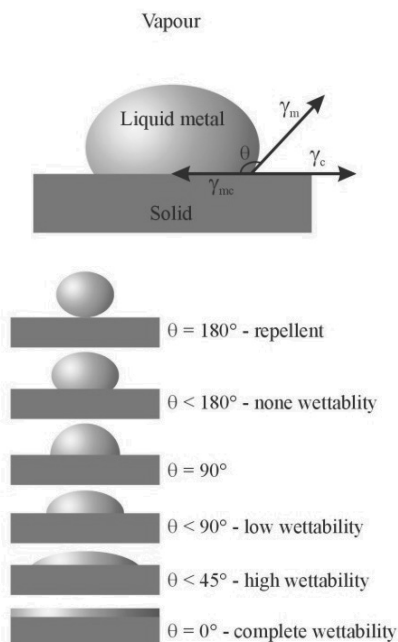


Fig. 1. Schema illustrates the definition of wettability angle θ according to the Young's equation of equilibrium and characteristic of metal ceramics systems depending on wettability angle θ value [21]

On the value of θ angle, in case of influence of liquid aluminum into Al_2O_3 , can impact the following factors: surface roughness, base porosity, contaminations, contact time, atmosphere, presence of oxide coating on the metal drop, what in the case of aluminum is the most essential factor deciding about the wettability. The reason

of that is the fact that aluminum has high value to oxidation, creating continuous and compact oxide coating, which is the barrier inhibiting the creation of sufficiently good contact for metal with the base. The coating makes difficult the interaction between phases contribute to increase of θ angle [7,14].

The goal of this work is to examine the structure and properties of aluminum matrix composites manufactured by pressure infiltration method of ceramic preform obtained by sintering of Al_2O_3 Alcoa CL 2500 powder with addition of pore forming agent in form of carbon fibres Sigrafil C10 M250 UNS. Moreover influence of Ni-P layer deposited onto reinforcement on the structure and properties of Al- Al_2O_3 composite materials was examined.

2. Experimental procedure

Material for experiments was made using the pressure infiltration of the ceramic preforms with the liquid aluminum alloy. The EN AC - AlSi12 eutectic aluminum alloy features the matrix material, which chemical composition is presented in Table 1; whereas the ceramic porous preform, was used as the reinforcement.

Table 1.
Chemical composition of EN AC-AlSi12 aluminum 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 ceramic preforms were manufactured by sintering of powder Al_2O_3 Alcoa CL 2500, with the addition of pores forming agent in the form of carbon fibres Sigrafil C10 M250 UNS of Company SGL Carbon Group. The use of carbon fibres as the pores forming agent decides of high purity process, because during their degradation only CO_2 is the oxidation product, while using the cellulose or sawdust, the furnace's walls are covered with hard to clean tar stains.

The manufacturing process of the ceramic preforms involved:

- preparation of Al_2O_3 powder and carbon fibres mixture,
- pressing of prepared mixture,
- sintering.

The properties and chemical composition of the used carbon fibres and ceramic powder are shown in Tables 2 and 3 respectively.

The porosity of the ceramic preforms depends on the carbon fibres content and is a part of 68.80% at 30% of carbon fibres addition, 75.60% at 40% of carbon fibres addition and 80.80% at 50% of carbon fibres addition.

The internal surfaces of ceramic preforms were coated with Ni-P in order to improve the Al_2O_3 wettability by the liquid aluminum alloy. Solutions containing metallic Pd were used for activation of the ceramics surface. Process of Ni-P coating deposition is shown in Fig. 2.

Table 2. Properties of Sigrafil C10 M250 UNS carbon fibres

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

Table 3. Chemical composition of Alcoa CL 2500 powder

Mean mass concentration of elements, wt. %						
Al_2O_3	Na_2O	Fe_2O_3	SiO_2	CaO	B_2O_3	The others
99.80	0.05	0.02	0.01	0.01	0.01	0.10

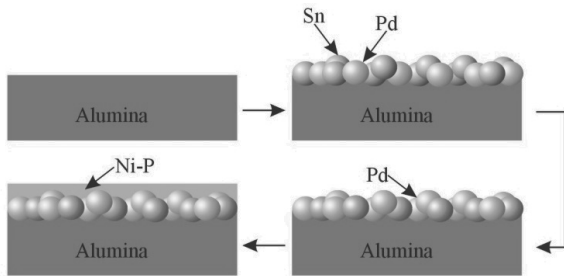


Fig. 2. Scheme of Ni-P deposition on the Al_2O_3 substrate

Firstly compacted Al_2O_3 plates covered by Ni-P were used to confirm proper process progress as shown in Fig. 3.

Deposition of internal surfaces of porous ceramic preforms was possible by application of a special device which allows to pump solutions over the preforms (scheme is shown in Fig. 4). The device was made from aluminum alloy, on internal parts (to avoid the reaction of aluminum with reagents) the ceramic lacquer coating was deposited. To assure the required temperature of the solution during coating deposition, on the device a copper spiral coil was installed.

All types of uncoated and coated by Ni ceramic preforms were heated in a furnace up to a temperature of 800°C . Covered by graphite form was warmed up to 450°C (maximal temperature of the press plates) and then filled with preform and liquid alloy EN AC - AlSi12 with a temperature of 800°C . The whole was covered by the stamp and placed in a hydraulic plate press Fontune TP 400. The maximum infiltration pressure was 100 MPa and its influence was 120 s. After solidification, obtained materials were removed from the form and cooled down under a pressured air stream.

Examinations of diffractions and the thin foils examination were made in a transmission electron microscope JEM 3010UHR by Jeol Company at the accelerating voltage of 200 kV. Thin films were made from plates cross-section electroerosively cut and mechanically thinned out from 1 mm to 0.2 mm. Obtained samples were then thinned out in a Disc Grynder to $80\ \mu\text{m}$ and ionically polished in Gatan devices.

The static tensile tests were made on the universal strength machine Zwick Z100 at room temperature according to PN-EN 10002-1:2004 standard.

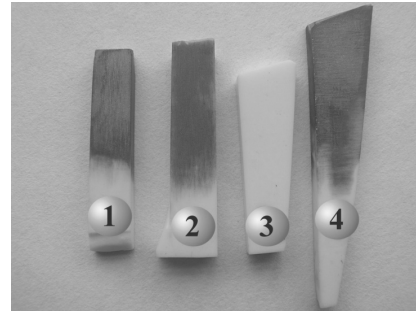


Fig. 3. Al_2O_3 plates with deposited Ni-P coating. No 1, 2, 4 samples were covered in full process, No 3 sample without activation process

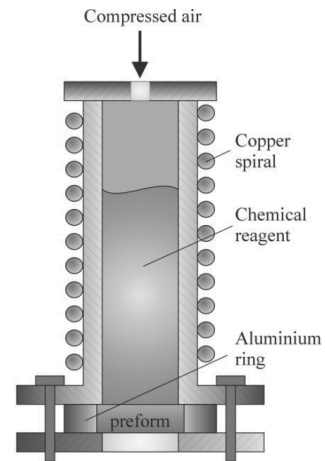


Fig. 4. Scheme of the device for deposition of the internal surface of porous ceramic preforms

3. Experimental results and their discussion

Investigation of thin foils in a transmission electron microscope of composite materials reinforced by porous ceramic preforms confirmed the presence of numerous Al_2O_3 grains in the structure (Fig. 5). Between Al_2O_3 fields is distributed a metal matrix (solid solution of Al) and Si grains with eutectic structure (Fig. 6), creating as a result of crystallization during cooling of composite materials after infiltration of ceramic preforms by liquid EN AC - AlSi12(A) alloy. In composite materials with Ni-P coating deposited on the Al_2O_3 , the presence of solid solution grains on the base of Ni (Fig. 7) is observed. Composite materials with Ni-P coating deposited on the Al_2O_3 in solid solution on the base of Al, the participation of Al_3Ni (Fig. 8) identified as a result of the reaction of the coating and aluminum infiltrating ceramic preforms.

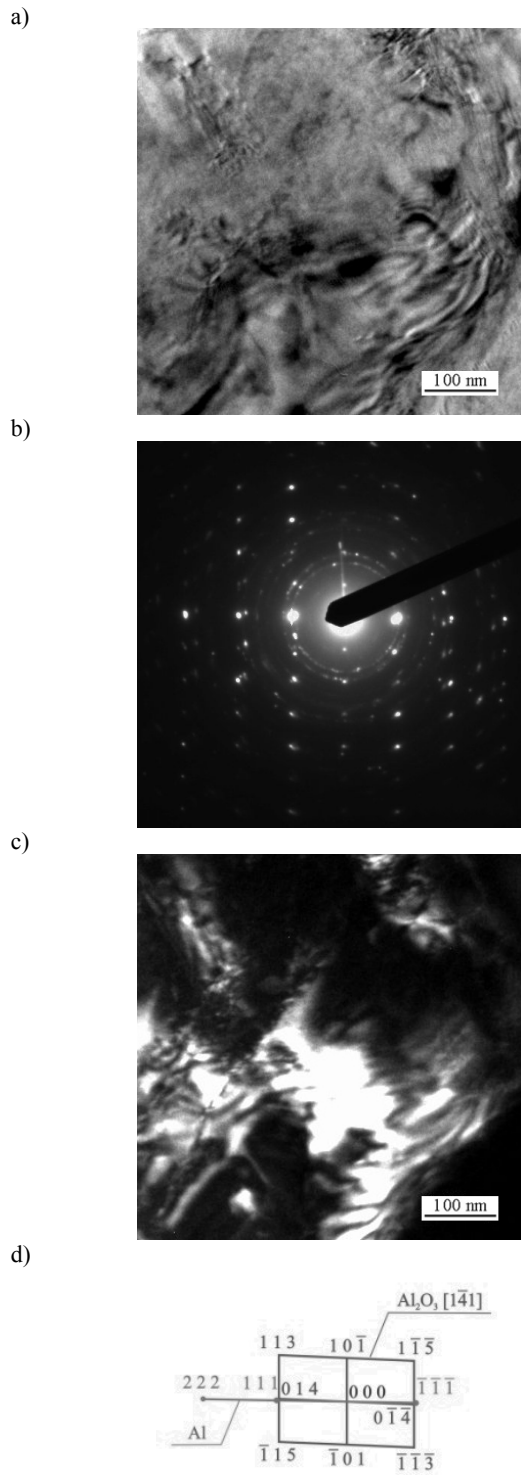


Fig. 5. Structure of thin foil, a) image in light field, b) diffraction pattern from area as in Fig a, c) image in dark field from 014 Al_2O_3 and 111 Al reflex, d) solution of the diffraction pattern from Fig. b

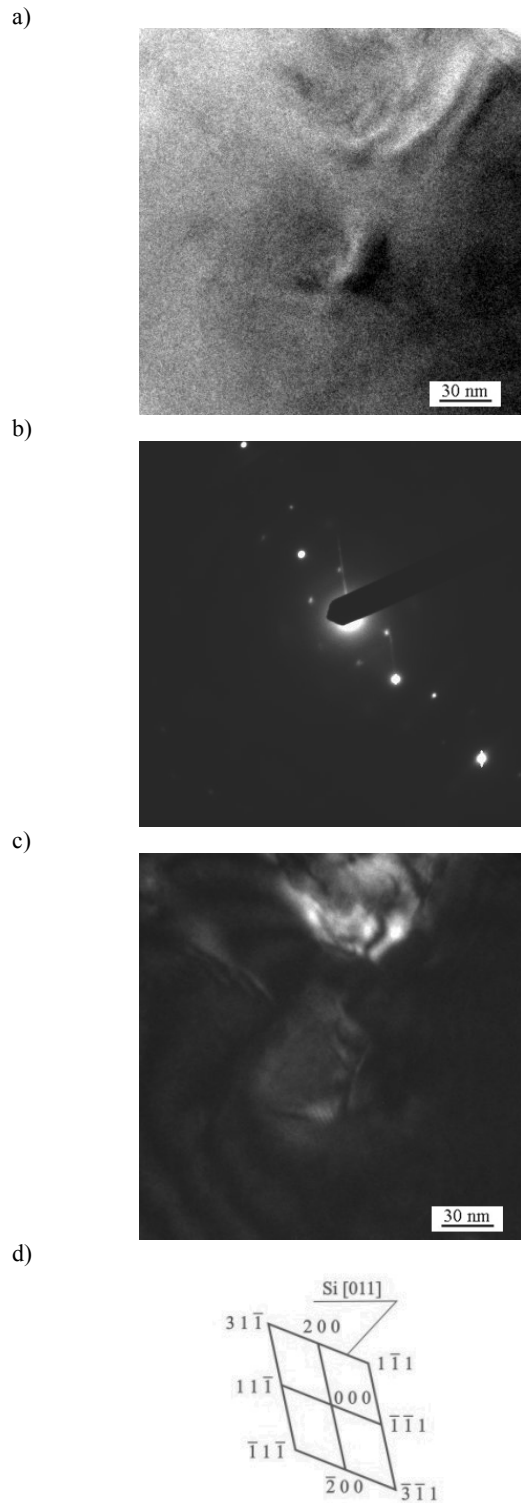


Fig. 6. Structure of thin foil, a) image in light field, b) diffraction pattern from area as in Fig a, c) image in dark field from 311 Si reflex, d) solution of the diffraction pattern from Fig. b

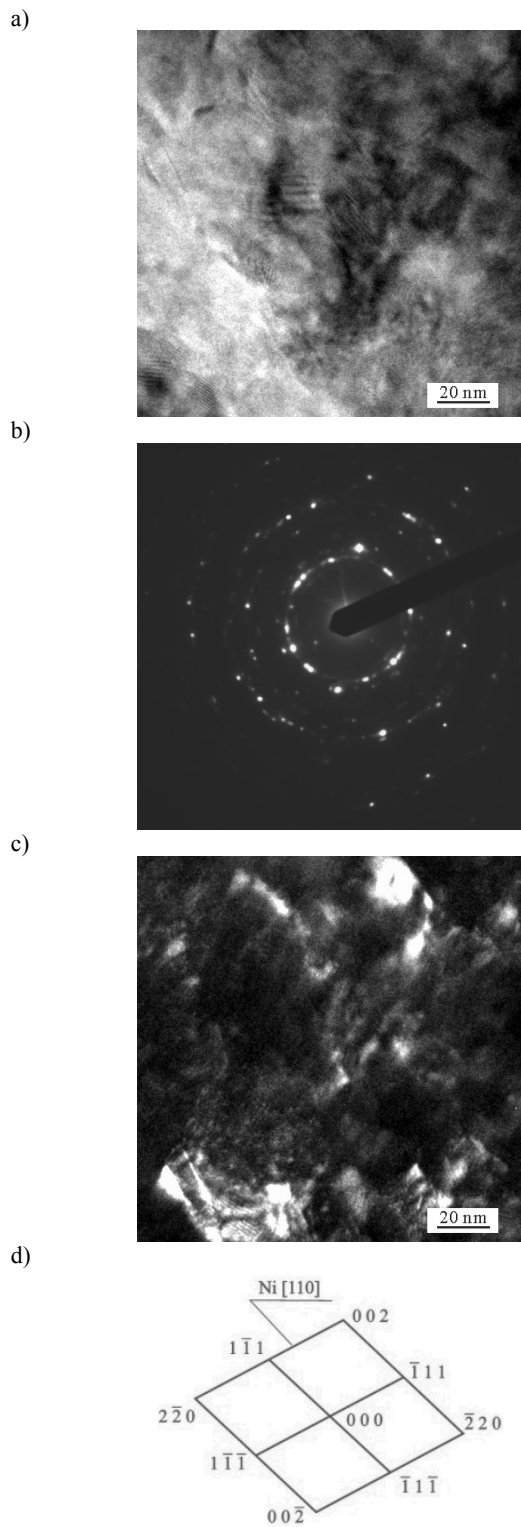


Fig. 7. Structure of thin foil, a) image in light field, b) diffraction pattern from area as in Fig a, c) image in dark field from $00\bar{2}$ Ni reflex, d) solution of the diffraction pattern from Fig. b

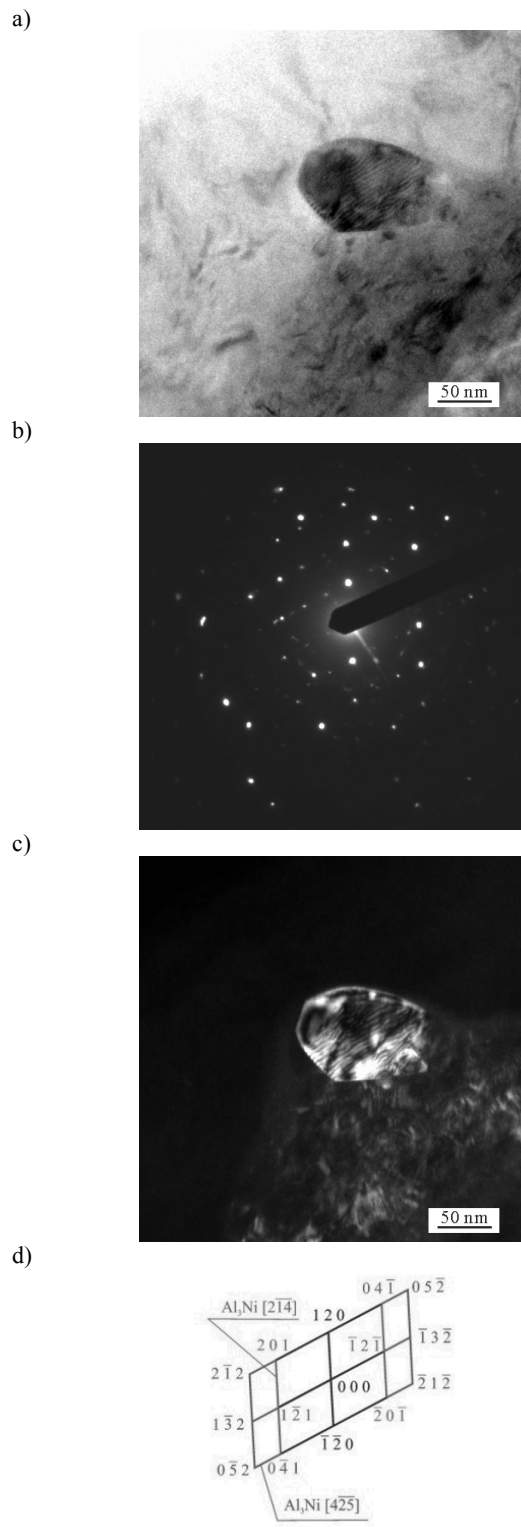


Fig. 8. Structure of thin foil, a) image in light field, b) diffraction pattern from area as in Fig a, c) image in dark field from $\bar{1}\bar{2}0$ Al_3Ni reflex, d) solution of the diffraction pattern from Fig. b

On the base of static tensile tests the strength of the obtained composite materials and the matrix material (EN AC - AlSi12 alloy) were established. The matrix is characterized by 198 MPa tensile strength. The maximum tensile strength show material reinforced by ceramic preform fabricated from particle with ~25% of Al_2O_3 phase covered by nickel (320 MPa). Further increase of ceramic phase in material cause the decrease of tensile strength (Fig. 9), that indicates, the reinforcement is not the main phase carried out the load, and the main mechanism of strengthening depend on the limitation of matrix to the plastic deformation. Results show also the purposefulness of covering internal surfaces of preforms by nickel to improve the wettability of Al_2O_3 by liquid aluminum alloy and amelioration of metal-ceramic connection.

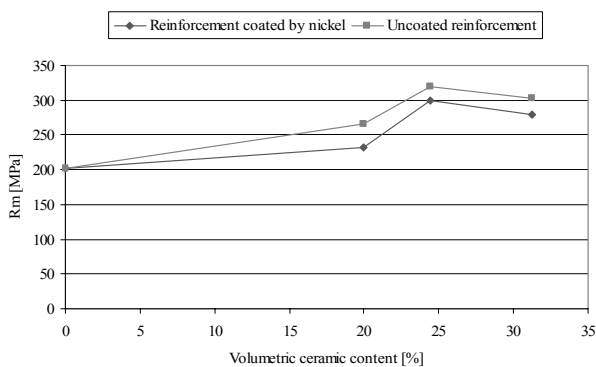


Fig. 9. The influence of ceramic phase content of the tensile strength of composite materials reinforced by Al_2O_3 preforms

4. Conclusions

As a result of thin foils investigation in transmission electron microscope of composite materials reinforced by ceramic preform the presence of Al and Si (matrix of composite material) is confirmed. The reinforcement was established to be Al_2O_3 . The presence of Ni in the Ni-P coating deposited onto Al_2O_3 is observed. There was also proved that its presence influence the improvement of Al_2O_3 wettability. Al_3Ni phase was identified as a product of reaction between coating and aluminum infiltrating ceramic preforms.

On the base of the static tensile test it could be observed significant increase of the strength of the obtained materials in comparison with the matrix. The maximum tensile strength show material reinforced by ceramic preform ~25% of Al_2O_3 phase content covered by nickel. Further increase of ceramic phase in material cause the decrease of tensile strength (Fig. 9), that indicates, the reinforcement is not the main phase carried out the load, and the main mechanism of strengthening depend on the limitation of matrix to the plastic deformation. Results show also the purposefulness of covering internal surfaces of preforms by nickel to improve the wettability of Al_2O_3 by liquid aluminum alloy and amelioration of metal-ceramic connection.

The obtained results indicate the possibility of obtaining new materials with all advantageous properties of the particular composite constituents by infiltration of the ceramics with the liquid aluminum alloy.

Acknowledgements

Part of investigations were made at Hochschule Aalen (Germany) in framework of Baden - Württemberg scholarships for International Students.

The paper has been realised in relation to the project POKL.04.01.01-00-003/09-00 entitled "Opening and development of engineering and PhD studies in the field of nanotechnology and materials science" INFONANO, co-founded by the European Union from financial resources of European Social Fund and headed by Prof. L.A. Dobrzański.



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