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COMPOSITES THEORY AND PRACTICE

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STRUCTURE AND PHYSICAL PROPERTIES OF ALUMINA CERAMIC FOAMS DESIGNED FOR CENTRIFUGAL INFILTRATION PROCESS

In the case of liquid phase processes used for the production of Al alloy matrix composites, the important factors are the interaction on the boundary of the liquid metal-ceramic phase, mainly the wetting of the ceramic surface. From this point of view, in the case of IPCs obtained by centrifugal infiltration, modification of the matrix alloy, the structure and physical properties of the porous ceramic materials, additionally the technological parameters like the temperature and time of infiltration as well as the rotational speed of the mould are important. In turn, the main parameters that influenced the efficiency of the infiltration process are the density, porosity, absorbability and permeability of the porous ceramic medium. The structural characteristics and physical properties of alumina foams, important for the infiltration of liquid aluminum alloy, have been presented. Scanning electron microscopy (SEM) and computer microtomography (μCT) were used to evaluate the microstructure of the Al_2O_3 foams. Selected properties of the foams such as density, open porosity, total porosity, absorbability were determined. In addition, the Ergun equation to determine the permeability of the foams was used. Metal-ceramic interpenetrating composites (IPCs) were obtained by the centrifugal infiltration of alumina foams with liquid Al alloy.

Keywords: alumina foam, structure, physical properties, centrifugal infiltration

STRUKTURA I WŁAŚCIWOŚCI FIZYCZNE KORUNDOWYCH PIANEK CERAMICZNYCH PRZEZNACZONYCH DO PROCESU INFILTRACJI ODŚRODKOWEJ

W procesach ciekłofazowych wytwarzania kompozytów o osnowie stopów Al istotnymi czynnikami są oddziaływania na granicy faz, a zwłaszcza zwilżanie powierzchni ceramiki przez ciekły metal. Z tego punktu widzenia w przypadku kompozytów IPCs otrzymywanych w procesie infiltracji odśrodkowej istotne są modyfikacja stopu osnowy, struktura i właściwości fizyczne porowatej ceramiki, a także parametry technologiczne, takie jak: temperatura, czas infiltracji oraz prędkość wirowania formy. Z kolei głównymi parametrami, od których zależy wydajność procesu infiltracji, są gęstość, porowatość, nasiąkliwość oraz przepuszczalność ceramicznego, porowatego medium. W niniejszej pracy przedstawiono charakterystyczne cechy strukturalne i właściwości fizyczne korundowych pianek ceramicznych istotne z punktu widzenia infiltracji ciekłym stopem aluminium. Do oceny mikrostruktury pianek Al_2O_3 zastosowano metody skaningowej mikroskopii elektronowej (SEM) oraz mikrotomografię komputerową (μCT). Wykorzystując metodę ważenia hydrostatycznego, wyznaczono wybrane właściwości pianek ceramicznych, takie jak: gęstość pozorna, porowatość otwarta, porowatość całkowita, nasiąkliwość. Ponadto, w celu odpowiedniego doboru preform ceramicznych do procesu infiltracji odśrodkowej ciekłym stopem Al wyliczono wartość przepuszczalności pianek, korzystając z równania Erguna. Do wytworzenia kompozytów o dwóch wzajemnie przenikających się fazach metalowej i ceramicznej (IPCs) zastosowano proces infiltracji odśrodkowej.

Słowa kluczowe: pianka z tlenku aluminium, struktura, właściwości fizyczne, infiltracja odśrodkowa

INTRODUCTION

In recent years there has been a significant increase in interest in the production of various types of ceramic foam materials [1-6]. This is primarily due to the unique properties of these materials such as low density, high melting point, low thermal conductivity, high permeability, vibration damping ability and good resistance to aggressive chemicals - features used in various industries. The basic areas of porous ceramic foam applications are filters operating in aggressive chemical and high temperature environments, catalytic coating substrates, porous biomaterials, as well as porous foams designed to infiltrate with liquid metals [7-16] or polymers [17, 18] to create interpenetrating composites (IPCs).

The intensive development of the aerospace industry as well as the automotive and machine industries necessitates the use of increasingly better materials, not only with good mechanical and tribological properties, but also with durability, dimensional stability and heat stability at elevated temperatures, and what is important, at a relatively low weight and cost of the finished product. These characteristics can be attributed to modern composite materials with interpenetrating metal and ceramic phases obtained by centrifugal infiltration [14, 15]. This process compared to other proposed methods of producing IPCs [7-13] is characterized by a high flow rate of liquid metal through the porous medium at a relatively low infiltration pressure caused by centrifugal force during rotation of the mould. In addition, the short infiltration and solidification time limits the contact time between the liquid metal and the porous ceramic skeleton. Shortening the contact time between the metal and ceramic is a very important factor especially for reactive systems such as Al/SiC, Al/C, moreover, it prevents grain growth in the matrix alloy and thus enables obtaing higher strength properties of the composite casting. In comparison to the individual manufacturing process of IPCs described by the authors of [12, 13], the centrifugal infiltration process makes it possible to obtain a series of near-net shape castings, also locally reinforced [14,15], which considerably limits the difficult and costly machining process of the composite final product [19]. As with other liquid-phase processes for the manufacture of Al matrix composites, the important factor is the wetting of the ceramic surface and interaction at the boundary of the liquid metal and the ceramic phase. From this point of view, in the case of IPCs obtained by centrifugal infiltration, not only technological parameters (i.e. temperature and time of infiltration, speed of the centrifugal mould), but the matrix alloy modification as well as the structure and physical properties of porous ceramics are also very important. The main parameters that influence the effectiveness of the infiltration process are the density, porosity, absorbability and permeability of the porous ceramic medium. The permeability as the ability of the material to transport metal through the pores, can be determined by the flow function. Taking into account pressure drop ΔP that exists on the length of sample L with retained cross-section A, Darcy's law or the Forchheimer equation is used. These equations serve to calculate the permeability, taking into account the flow velocity and compressibility of the transported fluid [4]. It should be noted that the permeability of ceramic foam materials is closely related to their relative density, which decreases as the window size increases and as the ratio of open to closed cells increases [4, 5]. Therefore, to determine the permeability, the modified Ergun equation can be used, initially allowing correlation of the permeability with porosity and the mean particle diameter of the granular medium. Modification of the equation consists in converting the parameter related to the grain size to one of the geometrical parameters of the foam. It was shown in [6] that the parameter which has a significant impact on foam permeability is the modal diameter of the cell windows; it means the channels allowing the flow of medium through the foam. The modified Ergun equation, whose accuracy was experimentally confirmed for foams having porosity degree greater than 85% [6], takes the form of (1) and (2):

a) for a lower fluid flow velocity:

$$k_1 = \frac{0.025 d^2 p^3}{150(1-p)^2} \tag{1}$$

b) for turbulent flow with a higher velocity:

$$k_2 = \frac{0.037 d p^3}{1.75(1-p)} \tag{2}$$

where: d - value of modal diameter of connections between pores, [m], p - open porosity of foam.

This paper presents the structural characteristics and physical properties of alumina ceramic foams, important for the infiltration of liquid aluminum alloy. The scope of the work includes:

1. Evaluation of the ceramic foam microstructure by means of scanning electron microscopy (SEM) and X-ray microtomography and to determine their basic geometrical parameters.
2. Determination of the physical properties of the tested foams, i.e. density, absorbability and porosity.
3. Calculation of the permeability of the foams as a critical parameter of the infiltration process.
4. Modification of the Al alloy chemical composition by Mg and Sr additions to improve Al/Al_2O_3 wetting.
5. Production of metallic-ceramic composites with interpenetrating structures in the centrifugal infiltration process.
6. Structure analysis and determination of selected physical properties of Al/Al_2O_3 composites such as: density and porosity.

RESEARCH MATERIALS

The AlSi12CuNiMg alloy was selected for the centrifugal infiltration of Al_2O_3 foams for its good properties and wide range of applications in the aerospace and automotive industries. This alloy is characterized by high strength, good fluidity and low susceptibility to hot cracking. Before the infiltration process, the AlSi alloy was subjected to modification by the addition of 1% magnesium, and 0.03% strontium. The addition of magnesium improves the wettability of the ceramic surface by the liquid metal, in turn the strontium additive prevents Si primary grain growth. In addition, magnesium reacts with Al and O and forms the $MgAl_2O_4$ spinel phase at the interfacial boundary, which provides good bonding between the AlSi alloy and the ceramic.

In the experimental works Al_2O_3 foams were used. The alumina oxide foams intended for infiltration were made in the Institute of Ceramics and Building Materials in Warsaw. For their fabrication the polymeric sponge method was applied [3]. In the research studies, four types of polyurethane sponges (produced by the

Eurofoam Group) of different densities and pore sizes were used. This allowed the preparation of different kinds of ceramic skeletons.

Metal-ceramic interpenetrating composites (IPCs) were fabricated by centrifugal infiltration using MOV500 equipment belonging to the Laboratory of Metal Composites at the Faculty of Materials Engineering and Metallurgy of the Silesian University of Technology. The stand was designed and constructed as part of a research project financed by funds from the National Science Centre (Project No. N N508 630 540). The device has a variable centrifugation rotation adjustment between 500 and 4000 rpm. [14, 15]. The technological parameters of the centrifugal infiltration process are shown in Table 1.

TABLE. 1. **Technological parameters of centrifugal infiltration**
TABELA 1. **Parametry technologiczne procesu infiltracji odśrodkowej**

Technological parameters	Value	Unit
Centrifugal mould rotational speed	3800	[rpm]
Infiltration temperature	720	[°C]
Ceramic foam temperature	350	[°C]
Mould filling time	3	[s]
Infiltration pressures	3.48	[MPa]

RESEARCH METHODOLOGY

A scanning electron microscope (VP S-3600N HITACHI) working with an X-ray spectrometer (EDS from THERMO NORAN) was used to evaluate the most important structural features of the ceramic foams. A dozen or so microstructure images of individual foams were made and analysed at the same magnification (50x). Microstructures with a total content of at least 100 cells and 100 windows for each foam were used. On the assumption of the spherical shape of the cells, manual detection was performed (Fig. 1). Calculations were performed using Adobe Illustrator CS6 - trial version [20].

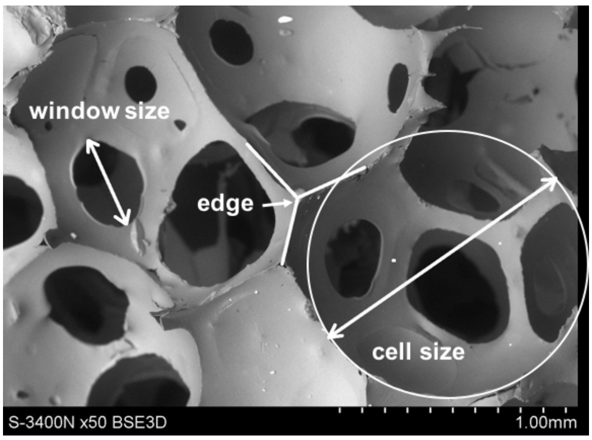


Fig. 1. Example of determining diameter of cells and windows in foam
Rys. 1. Przykład oznaczenia średnicy komórek i okien przelotowych w piance

Using the hydrostatic weighing method according to PN-EN1389, the physical properties of the tested alumina foams were determined such as apparent density, open porosity, total porosity and absorbability. The hydrostatic weighing process was carried out on a Radwag device (WAA100/C/1). The apparent density of the tested foams was calculated according to formula (3):

$$d_p = \frac{m_s}{m_n - m_w} \cdot d_0 \quad (3)$$

where: d_p - apparent density [g/cm^3], m_s - dry mass of sample [g], m_n - mass of sample saturated with water [g], m_w - mass of sample weighed in water [g], d_0 - water density at measurement temperature [g/cm^3].

The relative density values of the tested foams were calculated according to formula (4). Depending on the literature data, the real density of the corundum is between 3.97 and 4.02 g/cm^3. It was decided to take an average value d_{rz} = 3.98 g/cm^3:

$$d_w = \frac{d_p}{d_{rz}} \cdot 100\% \quad (4)$$

where: d_w - relative density [%], d_p - apparent density [g/cm^3], d_{rz} - real density [g/cm^3].

The absorbability of foam materials (N) was calculated according to formula (5), and open porosity of alumina foams (P_o) as a function of the calculated masses on the basis of equation (6):

$$N = \frac{m_n - m_s}{m_s} \cdot 100\% \quad (5)$$

$$P_o = \frac{m_n - m_s}{m_n - m_w} \cdot 100\% \quad (6)$$

The total porosity was calculated on the basis of the knowledge of real density (d_{rz}) and apparent density (d_p) according to formula (7):

$$P_c = \frac{d_{rz} - d_p}{d_p} \cdot 100\% \quad (7)$$

In turn, modified Ergun equation (2) was used to determine the permeability of the ceramic foams. The hydrostatic weighing method was also used to determine the density and total porosity of the IPC materials.

RESULTS AND DISCUSSION

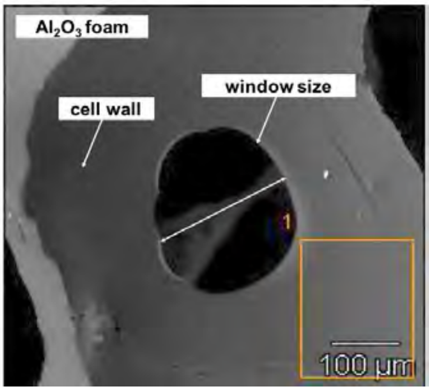
Structural studies

All the tested porous ceramic materials had the same chemical composition (Fig. 2). The SEM observations of the Al_2O_3 foams enabled determination of their characteristic structural features such as Plateau edges, shape and size of the cells, and the shape and size of the windows (Fig. 3). All the ceramic foams were characterized by an open cellular pore structure predominantly spherical in shape and of different sizes. In turn, the windows of the cells were spherical in shape and their size ranged from a few to almost 800 μm.

However, it is worth remembering that the microphotographs taken with the scanning electron microscope show only the outer surface of the foam. Each cell may have a different degree of openness. Therefore, X-ray microtomography (SkyScan 1174 tomograph) was used to determine the level of cell openness. The investigation was carried out in cooperation with Prof. Anna Boczkowska at the Faculty of Materials Science, Warsaw University of Technology. It was shown that all the tested foams were characterized by the presence of less than 1% closed pores. Figures 4-7 show selected macro- and microscopic images of the examined alumina foams together with their 3D models. The largest cell size was found in the foam marked as S28133 (Fig. 7). This foam had a pore size within 1mm. The foam marked with the symbol S31048 exhibited the smallest cell diameters (Fig. 4). The pore size in this foam did not exceed 550 μm.

As with the cell size, the highest value of window diameter (reaching even 790 μm) was exhibited by the foam marked as S28133 (Fig. 7). The windows of the remaining foams, i.e. S31048, S311062 and S28089, had diameters from 50 μm to nearly 300 μm. It was found that with an increase in pore size inside the ceramic foams, the window size in the cell increases.

a)



b)

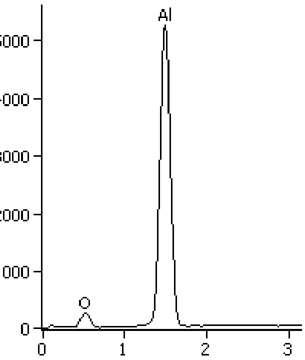


Fig. 2. Al_2O_3 foam marked as S28089: a) SEM image, b) results of EDS chemical analysis in area 1

Rys. 2. Pianka Al_2O_3 oznaczona symbolem S28089: a) mikrostruktura, SEM, b) wyniki analizy chemicznej w obszarze 1 (EDS)

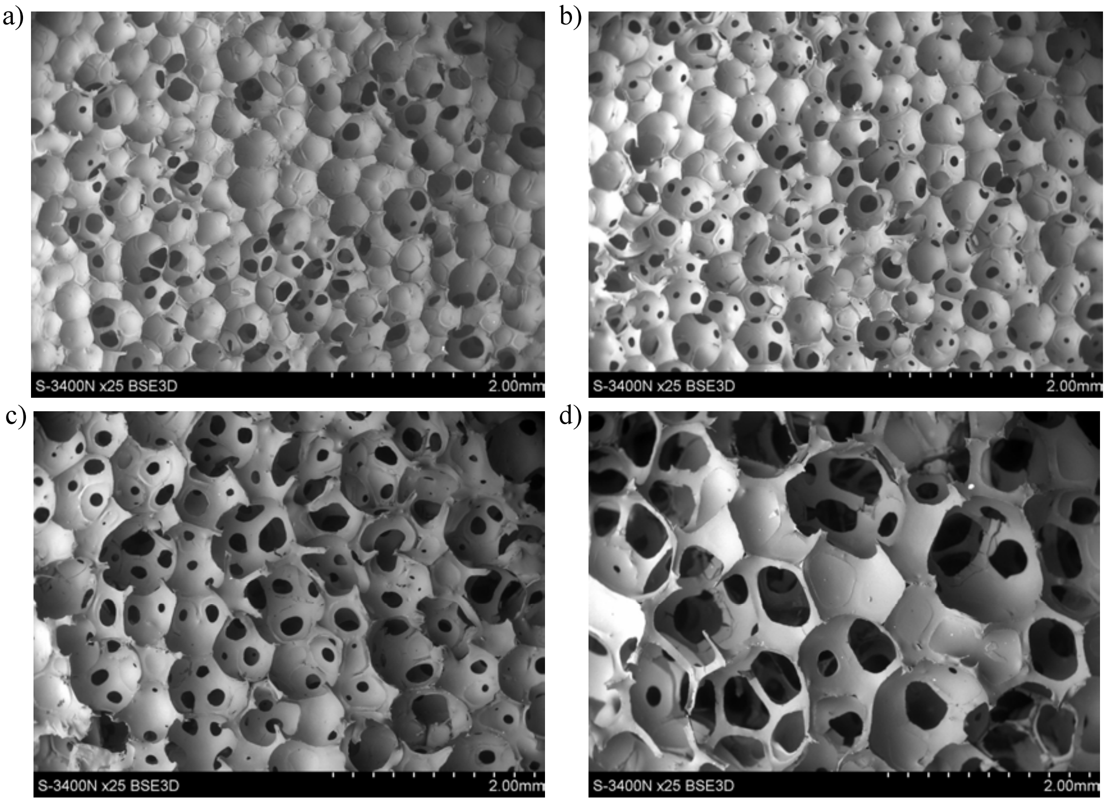


Fig. 3. SEM microstructure of Al_2O_3 foams: a) S31048, b) S311062, c) S28089, d) S2813

Rys. 3. Mikrostruktura SEM pianek Al_2O_3: a) S31048, b) S311062, c) S28089, d) S2813

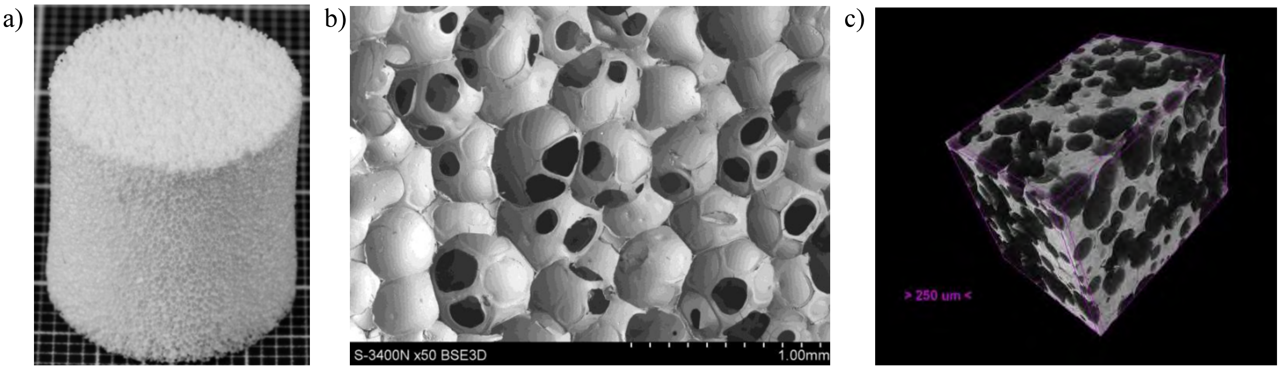


Fig. 4. Al_2O_3 foam marked as S31048: a) macrostructure, b) microstructure, SEM, c) 3D model, μCT

Rys. 4. Pianka Al_2O_3 oznaczona symbolem S31048: a) makrostruktura, b) mikrostruktura, SEM, c) model 3D, μCT

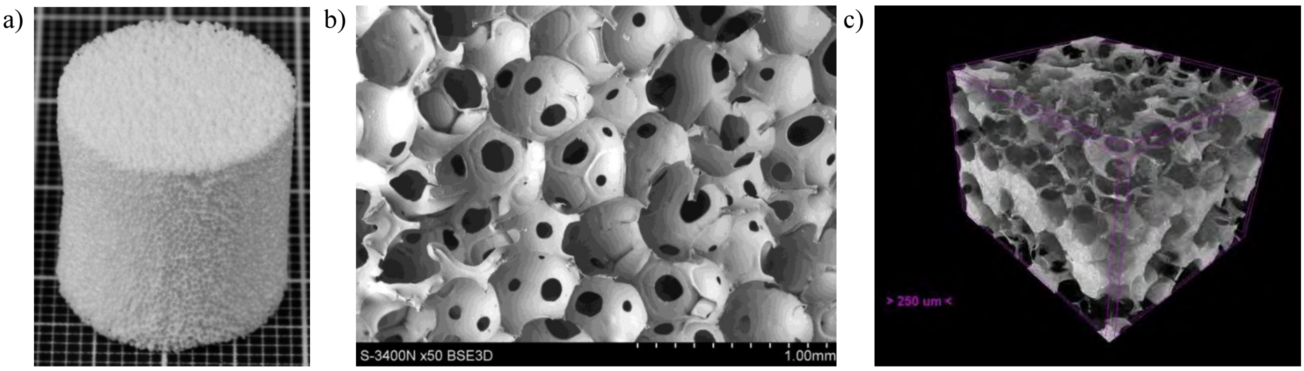


Fig. 5. Al_2O_3 foam marked as S311062: a) macrostructure, b) microstructure, SEM, c) 3D model, μCT

Rys. 5. Pianka Al_2O_3 oznaczona symbolem S311062: a) makrostruktura, b) mikrostruktura, SEM, c) model 3D, μCT

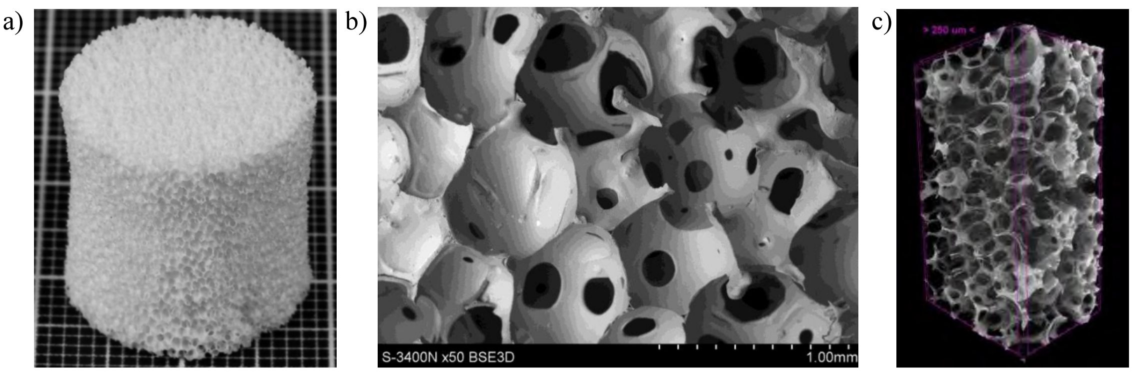


Fig. 6. Al_2O_3 foam marked as S28089: a) macrostructure, b) microstructure, SEM, c) 3D model, μCT

Rys. 6. Pianka Al_2O_3 oznaczona symbolem S28089: a) makrostruktura, b) mikrostruktura, SEM, c) model 3D, μCT

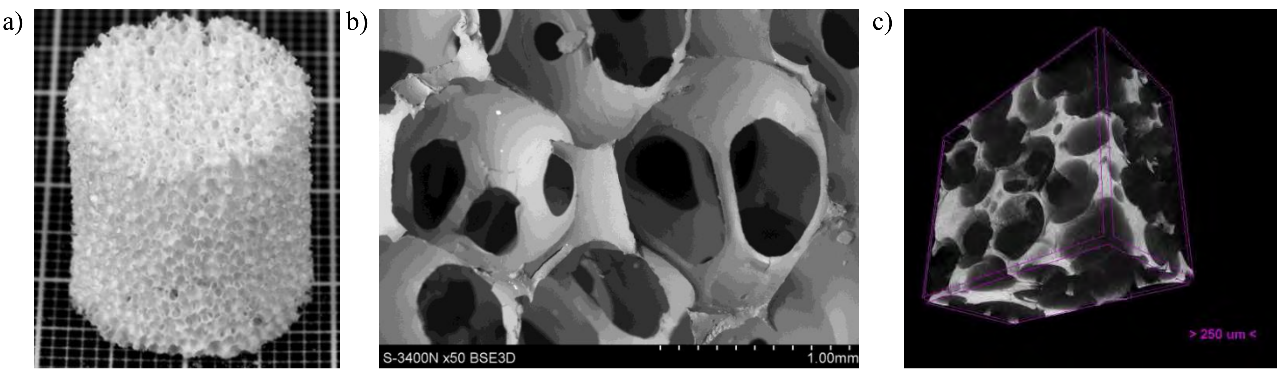


Fig. 7. Al_2O_3 foam marked as S28133: a) macrostructure, b) microstructure, SEM, c) 3D model, μCT

Rys. 7. Pianka Al_2O_3 oznaczona symbolem S28133: a) makrostruktura, b) mikrostruktura, SEM, c) model 3D, μCT

The characteristic feature of these ceramic foams is the presence of voids in the nodes (Fig. 8), which is related to their manufacturing process consisting in burning the expanded polymer substrate [3]. Voids in the nodes can reduce the strength of the ceramic skeleton. Therefore, knowledge of the ceramic foam structure is important to properly select the infiltration method.

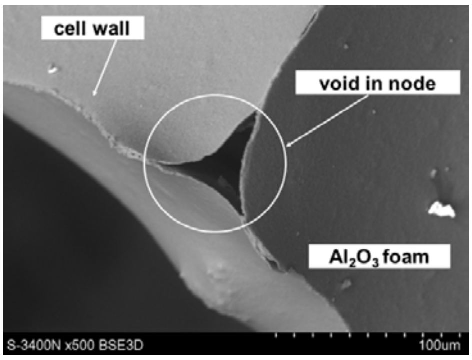


Fig. 8. Void in node in Al_2O_3 foam obtained by polymeric sponge method, SEM

Rys. 8. Pustka występująca w ściankach komórek pianek Al_2O_3 wytworzonych metodą odwzorowania polimerowej matrycy, SEM

Physical properties

The results of the physical property tests of the Al_2O_3 foams are shown in Table 2. By analysing the obtained results, it was found that the foams marked as S28133 and S28089 were characterized by the lowest apparent density values and the highest open and total porosity. In turn, the foam marked as S31048 possessed the highest apparent density values. The foam marked as S28089 had the greatest open porosity - over 79%, while the sample marked as S31048 had the smallest open porosity - 76.59%. The preform marked as S28089 had the highest absorbability - 97.89% and the preform marked S28133 - 96.65%. In turn, the foam designated as S31048 had lowest absorbability equal to 84.07%. To properly select ceramic preforms for the centrifugal infiltration process of liquid Al alloy, foam permeability was calculated using Ergun equation (2). The highest value of permeability (k_2) was calculated for the foam marked as S28133, while the preform denoted as S31048 had the lowest permeability. It should be noted that the calculation results of the non-Darcy permeability value for individual foams are only comparative because they were not confirmed experimentally.

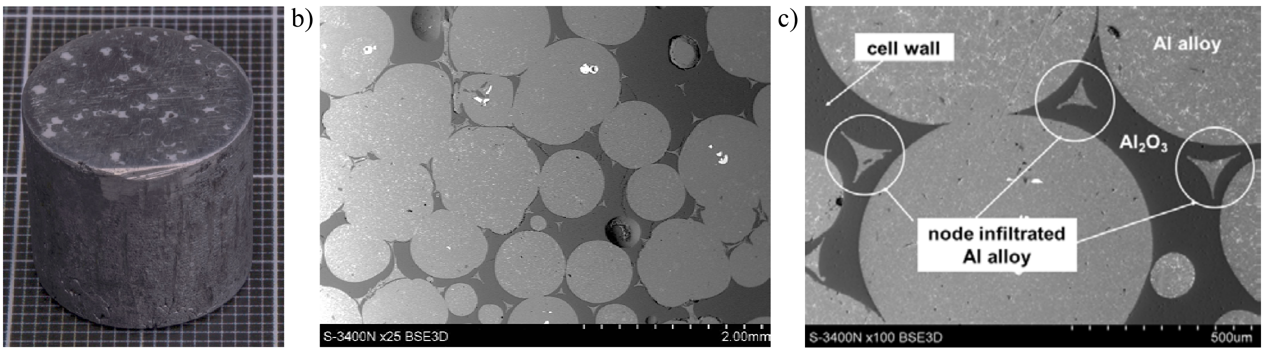
TABLE 2. **Physical properties of tested Al_2O_3 foams**
TABELA 2. **Właściwości fizyczne badanych pianek Al_2O_3**

Foam designation	Apparent density [g/cm^3]	Relative density [%]	Absorbability [%]	Open porosity [%]	Total porosity [%]	Permeability [10^{-7}m]
S31048 (F1)	0.910	22.86	84.07	76.59	77.14	4.79
S311062 (F2)	0.839	21.09	93.06	78.20	78.91	8.59
S28133 (F3)	0.813	20.44	96.65	78.83	79.56	28.7
S28089 (F4)	0.807	20.26	97.89	79.04	79.74	11.9

Structure of AlSi/Al_2O_3 infiltrated composites

In the centrifugal infiltration tests, two alumina foams with the highest open porosity and highest permeability (k_2) were used, that is the foam marked as S28089 (F4) and the foam denoted as S28133 (F3). The selected, characteristic structures of AlSi/Al_2O_3 composites with interpenetrating metal and ceramic phases are shown in Figures 9 and 10. On the basis of SEM observations, good and continuous connection between the ceramics and aluminum silicon alloy was confirmed. It was found that all the pores in both of the ceramic foams were completely filled by the AlSi alloy. Additionally, it was observed that the microporosity in the skeleton of the foams as well as the voids in the nodes are also filled with the metal alloy (Figures 9c and 10c). The above observations are compatible with the measurement results of density and porosity of the composites (Table 3). The experimental study was performed using the hydrostatic weighing method. The density of the modified AlSi alloy used for centrifugal infiltration was also determined (ρ_{AlSi} = 2.6 g/cm^3).

a) b) c)



Rys. 9. Infiltrowany kompozyt AlSi/Al_2O_3_(F4): a) widok próbki kompozytowej, b) mikrostruktura, SEM, c) widok wypełnionych stopem Al pustek obecnych w ściankach komórek, SEM

Fig. 9. AlSi/Al_2O_3_(F4) infiltrated composite: a) view of composite sample, b) SEM microstructure, c) view of filled voids present in cell walls, SEM

a) b) c)

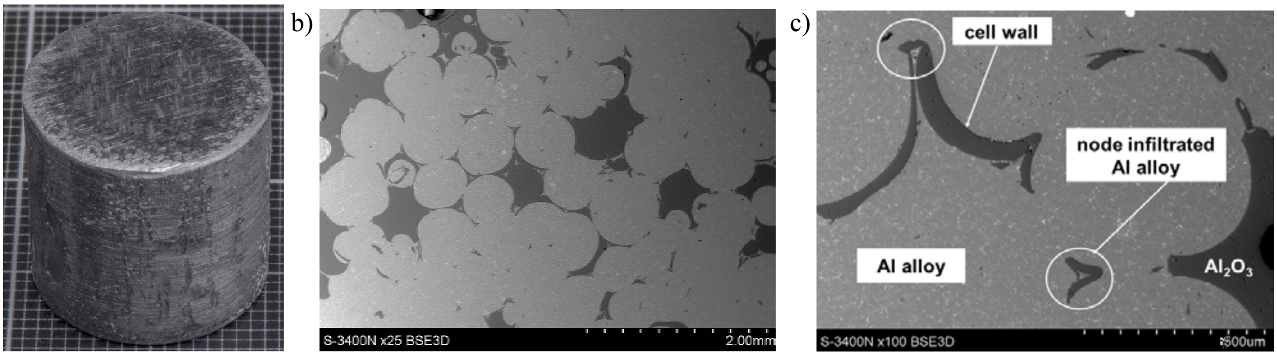


Fig. 10. $AlSi/Al_2O_3$_(F3) infiltrated composite: a) view of composite sample, b) SEM microstructure, c) view of filled voids present in cell walls, SEM

Rys. 10. Infiltrowany kompozyt $AlSi/Al_2O_3$_(F3): a) widok próbki kompozytowej, b) mikrostruktura, SEM, c) widok wypełnionych stopem Al pustek obecnych w ściankach komórek, SEM

TABLE 3. **Selected physical properties of $AlSi/Al_2O_3$ infiltrated composites**

TABELA 3. **Wybrane właściwości fizyczne infiltrowanych kompozytów $AlSi/Al_2O_3$**

Sample designation	Density [g/cm^3]	Total porosity [%]
$AlSi/Al_2O_3$_(F4)	2.79	3.39
$AlSi/Al_2O_3$_(F3)	2.83	1.94

SUMMARY

Based on analysis of the results of the described studies, the possibility of shaping a ceramic-metal interpenetrating composite structure using the centrifugal infiltration process was confirmed. The main advantage of the proposed manufacturing process is low pressure infiltration (p = 3.48 MPa), which, rather than damaging the structure of the ceramic skeleton, makes it possible for all the open spaces to be filled by the liquid AlSi alloy and the same allows obtaining functional composite materials.

It has been confirmed that the permeability of the alumina foams determined on the basis of the modified Ergun equation allows estimation of their relative susceptibility to the infiltration process. It has been shown that the physical properties such as density and total porosity of the obtained composites remain closely correlated with the structure and physical properties of the applied alumina foams. It was found that with an increasing window diameter in the ceramic cell, as the primary geometrical parameter which determines the permeability of the porous medium, it decreases the total porosity of the composites. It has an impact on increasing their density and thereby on the degree of infiltration. Further work on developing the technology of locally reinforced molded composite castings will be continued in the TANGO 2 project funded by NCBiR.

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