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MICROSTRUCTURE AND PHYSICAL PROPERTIES OF AlMg/Al_2O_3 INTERPENETRATING COMPOSITES FABRICATED BY METAL INFILTRATION INTO CERAMIC FOAMS

MIKROSTRUKTURA I WŁAŚCIWOŚCI FIZYCZNE KOMPOZYTÓW AlMg/Al_2O_3 OTRZYMANYCH PRZEZ INFILTRACJĘ METALU DO CERAMICZNEJ PREFORMY O BUDOWIE PIANKI

This work presents aluminium alloy-alumina (AlMg5/Al_2O_3) composites, where both phases are interpenetrating throughout the microstructure. Ceramic preforms for metal infiltration were produced by a new method of manufacturing of porous ceramics known as gelcasting of foams. Porous ceramics fabricated by this method is characterized by a continuous network of spherical cells interconnected by circular windows. Alumina (Al_2O_3) preforms used for infiltration process, were characterized by 90% porosity. The median diameter of spherical cell was 500 μm, while the median diameter of windows was 110 μm.

A direct pressure infiltration process was used to infiltrate the preforms with an AlMg5 alloy resulting in an interpenetrating microstructure. Due to the open cell structure of the Al_2O_3 foams, macropores in alumina preform were completely filled by metal. Microstructural characterization of the composites revealed a special topology of skeleton and good integrity of metal/ceramic interface. The density of AlMg5/Al_2O_3 composites was 2.71 g/cm^3, while the porosity was less than 1%.

Keywords: Interpenetrating composites, Al-Mg alloys, Al_2O_3 foams

W pracy opisano kompozyty metalowo-ceramiczne (AlMg5/Al_2O_3) o strukturze infiltrowanej charakteryzującej się wzajemnym przenikaniem szkieletów obydwu faz. Preformy ceramiczne (Al_2O_3) do infiltracji roztopionymi metalami wytworzono nową metodą otrzymywania wysokoporowatej ceramiki, którą jest żelowanie spienionej zawiesiny (ang. *gelcasting of foams*). Porowata ceramika wytworzona ww. metodą charakteryzuje się występowaniem sferycznych makroporów, zwanych także komórkami pianki, połączonych okienkami na wspólnych ściankach komórek, co sprawia, że porowatość próbki jest otwarta. Preformy korundowe przeznaczone do infiltracji charakteryzowały się porowatością 90%, modalną średnicą komórek pianki 450 μm i modalną średnicą okien na ściankach komórek 115 μm.

W procesie infiltracji ciśnieniowej stopu AlMg5 do pianek korundowych uzyskano kompozyty metalowo-ceramiczne o strukturze infiltrowanej charakteryzujące się pełnym wypełnieniem sferycznych makroporów preformy ceramicznej przez metal i dobrym przyleganiem na granicy faz ceramika-metal, co potwierdziły obserwacje morfologiczne zgładów i przełomów. Gęstość kompozytów wynosiła 2,71 g/cm^3, a porowatość mniej niż 1%.

1. Introduction

There is a need to improve the mechanical and physical performance of light metals such as aluminium and its alloys in order to extend their service limits in aerospace applications such as aeroengine and airflame components, spacecraft structures, heat sinks for electrical and electronic devices, sliding electrical contacts and many others [1, 2]. A popular way to achieving this is through the addition of ceramic reinforcements. Commercial approaches to producing these composites involve stirring ceramic particles or fibres into molten metals followed by casting. These approaches yielding a discrete phase dispersed in otherwise homogeneous matrix material. More recently, interest has arisen in composites where both phases are continuous, resulting in an interpenetrating microstructure [3-9]. Using Newnham's taxonomy which is based on phase connectivity, such materials are designed as (3-3) composites since both phases have connectivity in three dimensions [10]. The development of interpenetrating network composites is thus a logical step in the evolution of such materials that began with the fabrication of particulate (0-3) composites. There are some promising advantages resulting from the interconnectivity of the phases. Each phase contributes to the properties of the final composite, with the

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metal part increasing the strength and the fracture toughness relative to the monolithic ceramics, and the ceramic part increasing the dimensional and mechanical stability at high temperatures relative to the pure metal [9].

One method to achieve an interpenetrating metal-ceramic composite is the infiltration of a molten metal into a porous ceramic body called a preform which has a network of continuous open porosity. Provided that the structure of the initial porous material can be controlled sufficiently in terms of the degree of porosity, the shape and size of the pores, the size of the windows between them and the nature of the strut separating them, there is the opportunity to design and fabricate interpenetrating composites with customised structures. Hence the infiltration of porous ceramics offers the potential for producing tailored (3-3) interconnectivity interpenetrating composites.

In this work the ceramic preforms were made by a new forming technique of highly porous ceramics known as gel-casting of foams [11-12]. The gel-casting of foams process involves mechanical foaming of ceramic suspension containing foaming agents and gelling agents. The in situ polymerisation of gelling agents, led to fast solidification, resulting in strong porous bodies. The next step is calcinations of organic additives followed by sintering at high temperature. The obtained ceramic foams consist of interconnected porous network of spherical cells interconnected by circular windows. Due to the open cell structure of the gel-cast foams, the spherical cells may be completely filled by metal melts.

The main ecological problem of gelcasting process is the toxicity of the commonly used gelling agent – acrylamide, which is a neurotoxin. Hence, in this work, the environmentally friendly biopolymer (agrose) was proposed as the gelling agent [13]. This modified technology make it possible to produce ceramic performs in an environmentally friendly manner.

2. Materials and methods

2.1. Preparation of ceramic preforms and their characterization

Ceramic suspensions of alumina powder (CT 3000 SG, Alcoa Chemie, Ludwigshafen, Germany) were prepared to a solid loading of 55 vol.% by dispersing them with 0.5 wt.% of dispersant (Darvan 821A, R.T. Vanderbilt, U.S.A.). Their homogenisation was carried out by ball milling using alumina balls and jar. Agarose solutions (2.5 wt.%) were prepared by mixing agarose powder (POCH, Gliwice, Poland, Cat. No 111720491) with distilled water followed by heating for 1 hour at 95°C. The 2.5 wt.% agarose solution was added to the 55 vol.% alumina slurry maintaining the temperature of all constituents at 60°C. The final alumina content was fixed at 35 vol.%. Total concentration of active gelling matter in the final slurry was equal to 0.75 wt.% with regard to dry ceramic powder, which corresponded to 0,96 wt.% of agarose with regard to water.

Foaming was carried out at 60°C through agitation using a double-blade mixer. Details of the developed foaming process resulting in preparation of highly porous (~90%) ceramics were described in our earlier work [13]. Addition of non-ionic surfactants (Tergitol TMN-10, Aldrich, Germany and Simulsol SL-26, Seppic, France) was necessary to stabilize the foams. The concentration of each surfactant was 0.34 g/100ml of slurry. The foamed suspension containing agarose was poured into a mould. The mould was cooled down using flowing cold water (15°C) to transform the foam from a liquid state to a gelled state. The green bodies were then de-moulded and left in room conditions to dry. Sintering was performed at 1575°C with 2 h soaking time using heating rate of 2°C/min to 600°C and then 4°C/min up to sintering temperature.

The density of porous bodies was calculated from the mass and dimensions of a minimum of five samples having regular shapes. The theoretical density of fully densified alumina (3.98 g/cm^3) was used as a reference to calculate the total volume fraction of porosity.

The microstructure of ceramic foams was observed by scanning electron microscopy (SEM). The fractured samples first were coated with thin layer of gold. Pictures for monitoring the cellular structure were taken for estimation of cell and window sizes. This allowed window and cell size to be estimated from cells which presented an equator in the fracture surface and from windows by taking the major axis of oblique windows as the true diameter. The diameter of minimum 150 cells and 350 windows were measured for each sample and the pore and window size distributins were calculated.

The flexural strength of sintered Al_2O_2 foams was determined from three point bending. The bars with dimensions of 70 mm×15 mm×8 mm were used and the span was 40 mm. The crosshead speed was 0.5 mm/min for all samples. A universal testing machine (Instron 8080) was used.

The compressive strength was measured on un-axial compression, using 0.5 mm/min crosshead speed. The samples consisted of rectangular bars with dimensions of 15 mm×15 mm×20 mm. The loaded surfaces were covered with a thin sponge layer to obtain uniform load distribution throughout the faces. In all mechanical determinations results were based on average of five samples.

2.2. Preparation of metal-ceramic interpenetrating composites and their characterization

The metal used was AlMg5 alloy with technical purity. Magnesium is known to be a good surfactant, increasing the wetability of the aluminium alloy. The addition of magnesium to an aluminium melt improves wetting because of the lower surface tension of magnesium compared with that of aluminium. The addition of 3 wt. % magnesium to aluminium reduces its surface tension from 0.760 to 0.620 N m^{-1} at 720°C [14].On the other hand, the addition of Mg leads to the formation of $MgAl_2O_4$ spinel phase, which improves the adherence at the AlMg alloy/Al_2O_3 interface [15].

The composites were fabricated by pressure casting of AlMg5 alloy into the ceramic (Al_2O_3) foams with porosity level of 90%. The ceramic foam was placed in the graphite die and preheated together with a die to a temperature of 700°C. Before preheating and infiltration the reaction chamber was evacuated to vacuum of 2.67 Pa. The molten aluminium alloy with a temperature of 740°C was then poured into the preheated ceramic foam using the Degusa press. After the infiltration process a maximum pressure of 4 MPa was maintained until the die cooled and the alumina phase solidified.

The samples for microstructural observations were polished and examined using the SEM .

The density of the composites was determined using Archimedes method.

The bulk hardness of AlMg5 alloy and the interpenetrating composites was measured using a standard Rockwell hardness tester (scale B).

The compression strength of the AlMg5 alloy and the interpenetrating composites was measured at room temperature for each five cylindrical shaped samples (diameter 15 mm and height 30 mm) (Instron 8080). The speed of the cross-head was set to 0.5 mm/min.

3. Results and discussion

3.1. Characterization of ceramic (Al_2O_3) preforms

The total porosity of the obtained porous body was 90%, while the apparent density was 0.40 g/cm^3. The microstructure of the sintered foams is presented in Fig. 1. The alumina foams were typically composed of approximately spherical cells (pores) with no preferred orientation, resulting in isotropic properties, which was a typical result of direct foaming method [16-17] The spherical cells were interconnected by circular windows. The presence of windows is necessary for infiltration process.

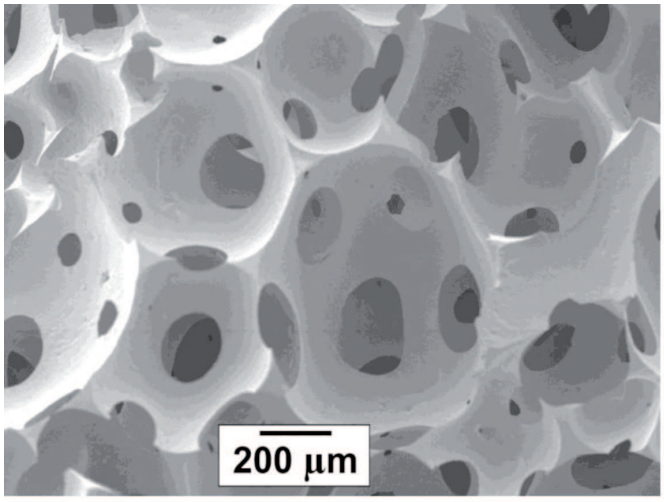

Fig. 1. SEM image of porous ceramics obtained by the gelcasting of foams method

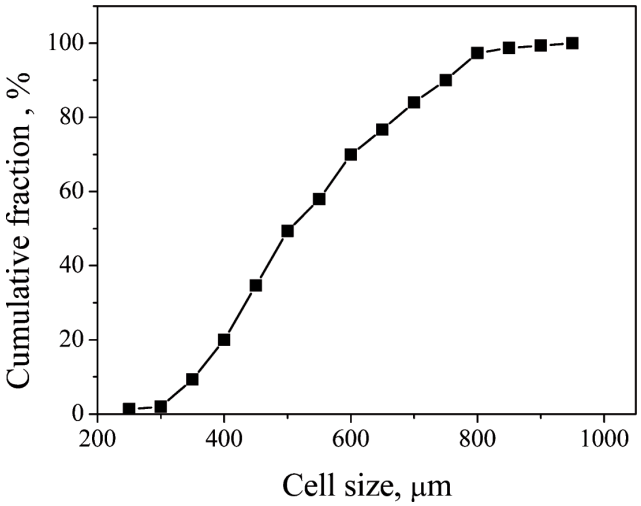

Fig. 2. Cell size distribution of Al_2O_3 foam measured on the base of image analysis

The degree of open porosity, the size of the spherical pores and the size of the windows between them are the critical parameters for infiltration process. Size distribution of the spherical pores is presented in Fig. 2 by the cumulative curve obtained from the results of image analysis. The foam exhibited a pore size distribution ranging from 100 to 1000 μm. The median spherical pore size was approximately 500 μm. The window size distribution of the alumina preforms is presented in Fig. 3. The mode window diameter was found to be approximately 110 μm.

The measurements of water permeability as well as the measurements of compressive and flexural strengths of alumina foams showed that the alumina foam ensured high mechanical strength and simultaneously high permeability. The obtained value of water permeability was $9{\cdot}10^{-10}$ m^2, while flexural and compressive strengths were 2.7 and 4.0 MPa, respectively. These parameters are very important for infiltration process. Due to the

non-wetting nature of most metal melts, especially the technologically interesting aluminium, this requires an external pressure to fill the pores. Therefore the mechanical strength of porous ceramic should be higher than threshold infiltration pressure. On the other hand, a high permeability is desirable because of the reduction in hydrodynamic pressure. For example, porous ceramics made by sintering of coarse powders have been reported with permeabilities in order of 10^{-15} – 10^{-14} m^2 and it is obvious that will be unsuitable for infiltration with low pressures [3].

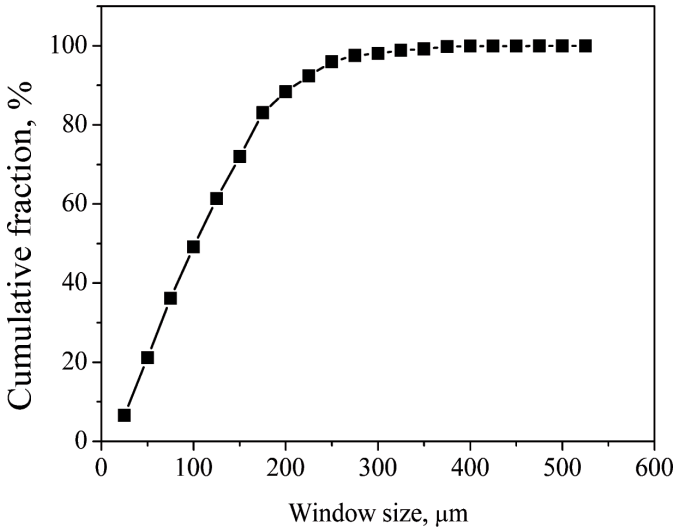

Fig. 3. Window size distribution of the Al_2O_3 foam measured on the base of image analysis

3.2. Characterization of metal-ceramic composites

The density of AlMg5/Al_2O_3 (10 vol% of Al_2O_3) composites determined by water immersion method was found to be 2.71 g/cm^3. The remaining porosity was measured as less than 1%. A small remaining porosity may be the closed porosity of the ceramic preforms.

The microstructure of the AlMg5/Al_2O_3 interpenetrating composites obtained by pressure infiltration of metal into porous Al_2O_3 preform is presented in Figs. 4 and 5. Due to open cell structure of the Al_2O_3 foams, macropores in alumina preform were completely filled by metal melts (Fig. 4). The fracture-section of the composite (Fig. 5) shows that the dense walls of Al_2O_3 cells does not allow the AlMg5 alloy to infiltrate but leaves fully dense struts in the composite to form a three-dimensionally continuous ceramic phase while the metal phase forms the other. Solidification under pressure condition appeared to provide a sound interface between the AlMg phase and the ceramic struts.

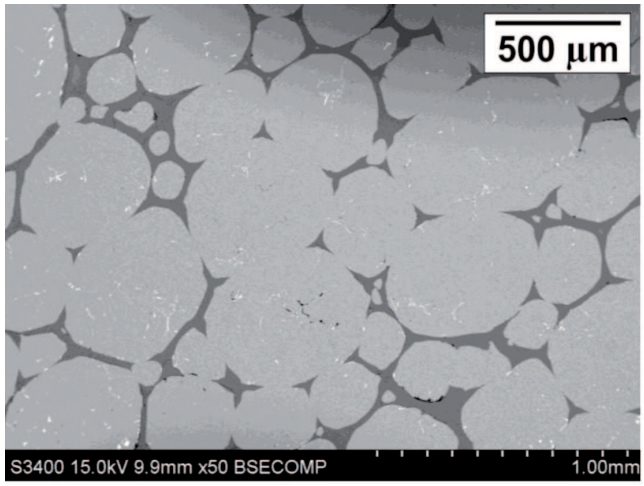

Fig. 4. SEM image (BSE mode) of the AlMg5/Al_2O_3 composite obtained by pressure infiltration of AlMg5 alloy into Al_2O_3 foam. Bright area is the metal phase; dark area is the ceramic phase

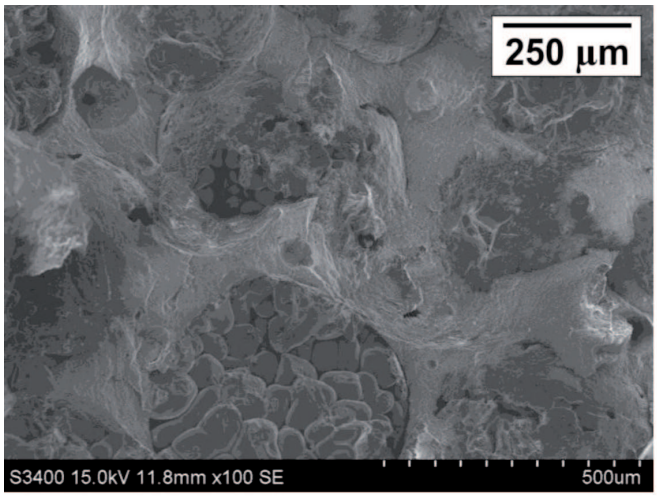

Fig. 5. Fracture section (SEM, SE mode) of the AlMg5/Al_2O_3 composite obtained by pressure infiltration of AlMg5 alloy into Al_2O_3 foam. Dark area is the metal phase; bright area is the ceramic phase

The Rockwell hardness of the AlMg5 alloy and the AlMg5/Al_2O_3 composite (Table 1), showed that the hardness of the composite was significantly higher than the alloy.

TABLE 1
Rockwell B hardness of the AlMg5 alloy and the AlMg5/Al_2O_3 composite

Materials	AlMg5	AlMg5/10vol%Al_2O_3
Rockwell B hardness	18 ± 4	32 ± 5

Static compression tests were performed on at least five specimens manufactured from the AlMg5 alloys and composites. Fig. 6 shows representative stress-strain curves for the AlMg5/Al_2O_3 composite and the metal alloy. Linear elastic regions have been approximated at

stresses below 100 MPa and 180 MPa for the AlMg5 alloy and the AlMg5/Al_2O_3 composite, respectively. At higher stresses the stress-strain behaviour is even more non-linear.

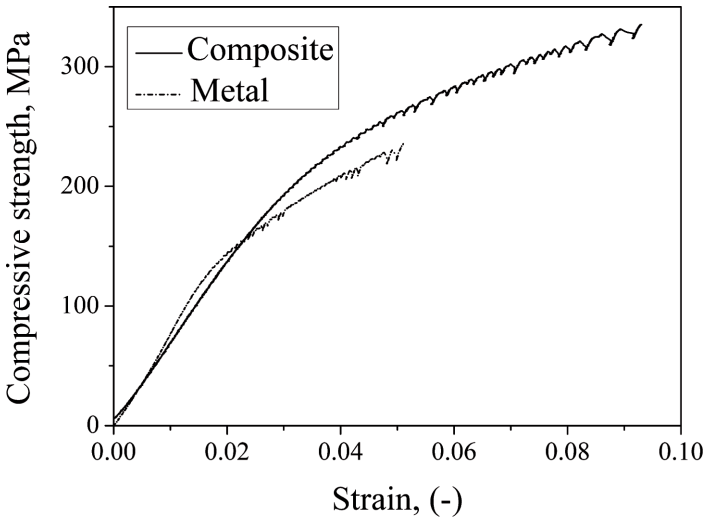

Fig. 6. Stress-strain curve under compressive load for the AlMg5 alloy and the AlMg5/Al_2O_3 composite

4. Conclusions

Open cell Al_2O_3ceramic foams were fabricated using gelcasting technique. The foams with porosity of 90% and compressive strength of 4 MPa served as preforms for molten AlMg5 alloy infiltration. Fully interpenetrating AlMg5/Al_2O_3 composites were obtained.

The hardness and compressive strength of the AlMg5/Al_2O_3 composite was significantly higher than that for the AlMg5 alloy.

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