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Fabrication of ceramic preforms based on Al_2O_3 CL 2500 powder

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Materials

ABSTRACT

Purpose: The aim of a work is to elaborate the method of manufacturing the porous, ceramic preforms based on Al_2O_3 particles used as the reinforcement in order to produce modern metal matrix composite materials.
Design/methodology/approach: Semi-finished products were manufactured by the method of sintering of ceramic powder. The preform material consists of powder Condea Al_2O_3 Cl 2500, however, as the forming factor of the structure of canals and pores inside the ceramic, agglomerated framework the carbon fibres Sigrafil C10 M250 UNS were used. The investigations of the structure of powder Al_2O_3 Condea Cl 2500, the used carbon fibres and the obtained ceramic preforms on the scanning electron microscope (SEM) have been made. The measurement of permeability of the obtained materials on the specially designed station has also been made.
Findings: The obtained preforms are characterised by volumetric participation of ceramic phase of 15 – 31%, what is the result of differential addition of the pores forming factor, and the high permeability indicates on "the open porosity".
Research limitations/implications: The basic limit of the mentioned method is the possibility of obtaining preforms of porosity less than 85%, where in case of using the ceramic fibres the pores can be more than 90% of material volumetric.
Practical implications: The manufactured ceramic preforms are widely used as the reinforcement to produce the composite materials by the method of infiltration. That method allows manufacturing the metal elements locally reinforced and the near-net shape composite products.
Originality/value: The received results show the possibility of obtaining the new preforms being the cheaper alternative for semi-finished products based on the ceramic fibres and the use of carbon fibres as the pores forming agent indicate that it is the high-quality process.
Keywords: Composites; Ceramic preforms; Permeability

1. Introduction

Composite materials on the light metals alloys matrix reinforced by fibres and ceramic particles are characteristic of very good mechanic properties, rigidity, hardness and wear resistance by relatively low density. These materials are also characteristic of the smaller coefficient of thermal expansion in proportion to the matrix. With regard to their advantages, they have been widely used in motorization, air industry and also to produce sport equipment. From the composite materials on the matrix of light metal alloys can be produced elements exposed to the impact of high temperature, elements subjected to intensive wear and also elements in power transmission systems with low friction coefficient and high vibration damping capability.

Two main development directions of manufacturing technology of the metal composite materials are observed, powder metallurgy [1-3] and casting methods, with specific modification of the pressure infiltration of the porous, ceramic preforms with liquid metals alloys. That method is used more and more often in manufacturing of the composite materials with metal matrix and has also become the subject of many research projects [4-11].

The ceramic preforms, being a framework, are the base of the composite materials manufactured by infiltration method. These preforms mainly determine the structure and the properties of the final product. The properly manufactured semi-finished product should be characterized by the structure of open joined canals allowing the liquid metal to flow as easily as possible. The occurrence of the closed pores or blind canals causes the formation of areas with no metal. Several fundamental development ways of manufacturing of the porous ceramic material are observed [12-17]:

- Sintering rough powder with or rarely without binder – it's the easiest and the most common method depending on sintering of the unthickened ceramic powder, which allows obtaining preforms of 50% porosity.
- The method basing on making foam from ceramic suspension, which depends on the use of phenomenon of gas emanation as a result of chemical reaction or reaction of degradation, which takes place in high temperature, e.g.:

$$CaCO_3 \rightarrow CaO + CO_2 \quad (1)$$

That way enables to obtein preforms up to 90% porosity.

- Getting porous ceramic materials with the method of freezing and solvent sublimation (usually distilled water) from ceramic suspension. The porous structure is shaped by the volumetric participation of the solvent in suspension, the speed of freezing and ice sublimation under the lower pressure.
- Sintering of ceramic powders with the addition of pores forming agent structure.

The last of the presented methods is most flexible and allows obtaining the diverse structure materials and ceramic phase usage. The level of porosity and its character can be adjusted with different pore forming agents (PFA) addition, that are degradated in high temperature in the areas where the pores are originated. The most commonly used pore-forming substances are the materials of thermal degradation temperature considerably lower than sintering temperature of ceramics, like: polythene, wax, starch, cellulose, carbon fibres etc.

The goal of this project is the technology optimization of manufacturing of the ceramic preforms based on Al_2O_3 powder with the addition of carbon fibres as the pores forming agent. The obtained semi-finished products are going to be used as the framework of the composite materials manufactured by the pressure infiltration method with liquid metal alloys.

2. Experimental procedure

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 manufacturing of the ceramic preforms involved: the preparation of powders, their pressing and sintering. The powder Al_2O Condea Cl 2500 was wet - grinded with the use of distillated water in a ball grinder for 5 min. in order to break the particles concentrated in agglomerations. Into Al_2O_3 suspension were added the addition of anti-forming agent of the aglomerations of carbon fibres Dolapix CE 64 of Zschimmer und Schwarz GmbH Company, eliminating their electrostatic interactions. The addition of the carbon fibres was 30, 40 and 50 % of weight. 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 0.25mm. To activate the polyvinyl alcohol, the powders were humidified with distillated water, packed into the plastic bags and put away for 24h. The powders were uniaxially pressed in the hydraulic press "Nelke" in steel mold with the inside diameter of 30mm. Pressing pressure was 25, 50 and 100 MPa, and time of its influence was 15s. Sintering of the mouldings was done with the help of pipe furnace in air stream atmosphere of 20 l/min. The course of sintering process is presented in Figure 1. It consisted of slow heating to 800°C (20°C/h), keeping that temperature till 10h for burning out the carbon fibres, heating to 1500°C (300°C/h), sintering (2h) and cooling with furnace.

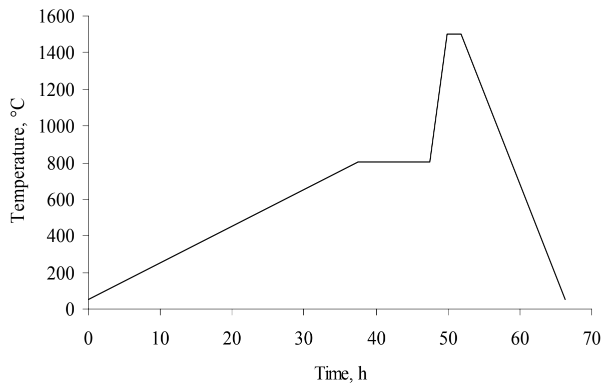


Fig. 1. Temperature performance during the sintering process

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 obtained ceramic preforms were measured and weighed, for a calculation of the amount of ceramic phase fraction and therefore its porosity.

The permeability measurements where made on especially designed device for pumping over the liquid through the porous materials. During the test, the flow time of 350ml water was measured, adequately under the pressure of: 0.5; 1; 1.5 and 2 bars at room temperature. The permeability of preforms was calculated with formula:

$$D = \frac{\eta \cdot L \cdot \dot{V}}{A \cdot \Delta p} \quad (2)$$

where: D – the permeability, η – liquid viscosity, L –thickness of preform, V – flow, A - surface of preform, Δp – pressure growth.

Table 1.
Properties of Sigrafil C10 M250 UNS carbon fibres

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

The observation of the structure of ceramic preforms fractures, manufactured by the sintering of Al_2O_3 Condea Cl 2500 powder method was carried out using the Leica Stereoscan 440 scanning electron microscope.

3. Experimental results and their discussion

Geometries and weight measurements of ceramic preforms manufactured by powder sintering method with the addition of 30, 40 and 50% carbon fibres pressed under pressure of 25, 50 and 100MPa, allowed to calculate the theoretical volume fraction of ceramic phase in a porous material volume, what is presented in Table 2. Regarding the optimal mechanical properties in the next researches the materials pressed only under 100Mpa were used.

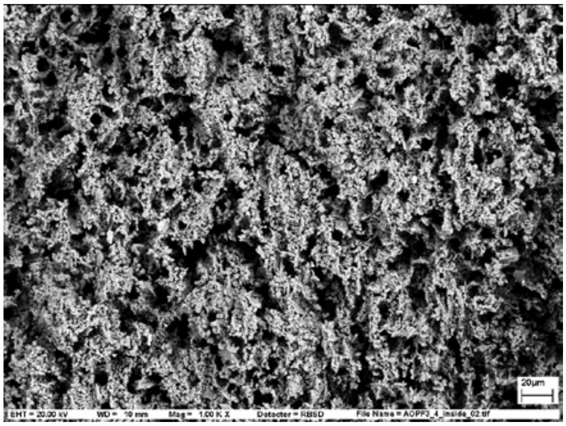
Table 2.
Ceramic phase volume fraction in preforms obtained from different powders of different levels of carbon fibres addition, pressed under different pressure

Carbon fibres content, %	Pressing pressure, MPa		
	25	50	100
30	26.10	28.18	31.24
40	19.67	23.04	24.4
50	14.73	16.76	19.2

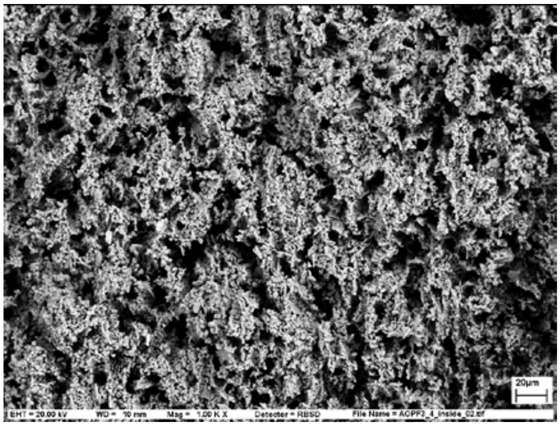
Permeability measurements of ceramic preforms manufactured from powder of carbon fibres with mass fraction of 30, 40 and 50% pressed under pressure of 100 MPa showed the increase of permeability together with the increase of porosity in accordance to: $2.45 \cdot 10^{-13}$ m^2 for preforms with 69% of porosity, $5.2 \cdot 10^{-13}$ m^2 for materials with 76%of porosity and $1.05 \cdot 10^{-12}$ m^2 for preforms with 81%.of porosity.

The observations of preforms fractures manufactured by the sintering of Al_2O_3 Condea Cl 2500 powder carried out using the scanning electron microscope (Fig. 2) allowed revealing two main types of pores. The first are bigger and have been formed because of carbon fibres degradation, the second are smaller and appear around singular ceramic particles and result from intentional lack of condensation (caused by using the greater pressure and higher sintering temperature). Moreover the study proved no presence of carbon fibre aglomerations, what resulted from the proper work of the used addition of preperation Dolapix CE 64 eliminating electrostatic effects.

a)



b)



c)

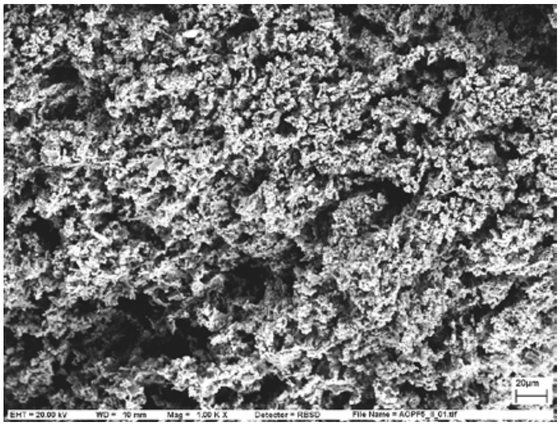


Fig. 2. The microstructure of ceramic preforms fracture based on Al_2O_3 powder with addition: a) 30%, b) 40% and c) 50% of carbon fibres

4. Conclusions

Geometrical dimensions and masses of the obtained ceramic materials measurements can tell that there is a possibility of manufacturing with the presented method preforms with 85% of porosity of material volume. The findings of permeability measurements claim that pores and micro-canals in the material are connected (open porosity) and allow easy penetration with liquid metal alloy during the infiltration process.

The examinations of the structure carried out using scanning electron microscope show the proportionate arrangement of canals have arisen after the oxidized carbon fibres and the presence of micro-pores around the ceramic particles.

It have been proved that the developed technology for obtaining porous ceramic preforms with the use of sintering method of Al_2O_3 Condea Cl 2500 powder with the addition of carbon fibres Sigrafil C10 M250 UNS as the pores forming agent provides the required structure and properties and for this reason it can find the practical application

Moreover the obtained materials can be a cheaper alternative for widely used preforms based on ceramic fibres. Some researches are taking place over the manufacturing of the composite material based on the developed semi-finished product.

References

[1] L.A. Dobrzanski, A. Wlodarczyk, M. Adamiak: "Structure, properties and corrosion resistance of PM composite materials based on EN AW-2124 aluminum alloy reinforced with the Al_2O_3 ceramic particles", Journal of Materials Processing Technology 162–163 (2005) 27–32.

[2] L.A. Dobrzanski, A. Wlodarczyk-Fligier, 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 11th International Scientific Conference CAM^{3}S 2005, 289-294.

[3] L.A. Dobrzanski, A. Wlodarczyk, M. Adamiak: "Aluminum alloy AlCu4Mg1 matrix composite materials reinforced with ceramic particles", Proceedings of the 12th International Scientific Conference AMME 2003, 297-300 (in Polish).

[4] L.A. Dobrzanski, M. Kremzer, A. Nagel, B. Huchler: "Composite materials based on the porous Al_2O_3 ceramics infiltrated with the EN AC – AlSi12 alloy", Kompozyty (Composites), 4a (2005) 35-41 (in Polish).

[5] L.A. Dobrzanski, M. Piec: "Structure and properties of aluminium alloys reinforced with the Al_2O_3 particles", Proceedings of the 12th International Scientific Conference AMME 2003, 271-276.

[6] V.M. Kevorkijan: "The reactive infiltration of porous ceramic media by a molten aluminum alloy", Composites Science and Technology 59 (1999) 683-686.

[7] W.S. Sheng, S.J. Lin: "Ni-coated SiC_p reinforced aluminum composites processed by vacuum infiltration", Materials Research Bulletin 31/12 (1996) 1437-1447.

[8] G.W. Han, D. Feng, M. Yin, W.J. Ye: "Ceramic/aluminum co-continuous composite synthesized by reaction accelerated melt infiltration", Materials Science and Engineering A225 (1997) 204-207.

[9] Abd-Elwahed M. Assar: "Fabrication of metal matrix composite by infiltration process-part 2: experimental study", Journal of Materials Processing Technology 86 (1999) 152-158.

[10] N. Nagendra, B.S. Rao, V. Jayaram: "Microstructures and properties of Al_2O_3/Al-AlN composites by pressurless infiltration of Al-alloys", Materials Science and Engineering, A269 (1999) 26-37.

[11] M. Szafran, G. Rokicki, W. Lipiec, K. Konopka, K. Kurzydłowski: "Porous ceramic infiltrated by metals and polymers", Kompozyty (Composites) 2 (2002) 313-316 (in Polish).

[12] A. Mattern, B. Huchler, D. Staudenecker, R. Oberacker, A. Nagel, M.J. Hofmann: "Preparation of interpenetrating ceramic-metal composites", Journal of the European Ceramic Society, 24 (2004) 3399-3408.

[13] G.G. Kang, Y.H. Seo: "The influence of fabrication parameters on the deformation behavior of the preform of metal-matrix composites during the squeeze-casting processes", Journal of Materials Processing Technology 61 (1996) 241-249.

[14] K. Naplocha, A. Janus, J.W. Kaczmar, Z. Samsonowicz: "Technology and mechanical properties of ceramic preforms for composite materials", Journal of Materials Processing Technology 106 (2000) 119-122.

[15] L.M. Peng, J.W. Cao, K. Noda, K.S. Han: "Mechanical properties of ceramic-metal composites by pressure infiltration of metal into porous ceramics", Materials Science and Engineering A374 (2004) 1-9.

[16] M.P. Dariel, L. Levin, N. Frage: "Graded ceramic preforms: various processingapproaches", Materials Chemistry and Physics 67 (2001) 192-198.

[17] N. Altinkok, A. Demir, I. Ozsert: "Processing of Al_2O_3/SiC ceramic cake preforms and their liquid metal infiltration", Composites, 34 (2003) 577-582.