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Structure and properties of ceramic preforms based on Al_2O_3 particles

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Materials

ABSTRACT

Purpose: The main goal of this project is to elaborate and optimize 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 composites by pressure infiltration method with liquid metal alloys.
Design/methodology/approach: Ceramic preforms were manufactured by the sintering method of Al_2O_3 powder with addition of pore forming agent. The preform material consists of powder Alcoa 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 addition of carbon fibres was 30, 40 and 50% of weight. The TGA analysis of carbon fibres has been made. The investigations of the structure of powder Al_2O_3 Alcoa 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 characterized by volumetric participation of ceramic phase of 15-31%, what is the result of differential addition of the pores forming agent, 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 infiltration method. 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; TGA analysis; Permeability

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1. Introduction

Modern composite materials based on aluminum and magnesium alloys reinforced by fibres and ceramic particles are characteristic of very good mechanical properties, stiffness, 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.

Several development directions of manufacturing of the composite materials with aluminium alloys matrix are observed in the last dozen years. The basic technologies used for manufacturing of these composites are powder metallurgy [1,5,6] and casting techniques [9,10] 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 [8,11,12,14,16,19,21]. The usage of infiltration process as the high-profitable technology is a base of getting the wide range of composite materials and allows to obtain the following technological - organizational profits [20]:

- the possibility of obtaining the composite products of precise shape mapping and the high-quality surface (near net shape),
- the possibility of local reinforcement of the product,
- 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 porous preforms, containing ceramic particles, fibres or combination of both 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 no infiltrated by liquid metal. Several fundamental development ways of manufacturing of the porous ceramic material are observed [2,3,4,13,15,17,18]:

- 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.:

$$Ca\,CO_3 \rightarrow CaO + CO_2 \quad (1)$$

  That way enables to obtain 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.

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 Alcoa 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 are shown in Table 1 and the chemical composition mean diameter and density of Al_2O_3 Alcoa CL 2500 powder are shown in Table 2.

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 wt. %
8	135	1.75	2.5	26	>95

The observation of the used carbon fibres Sigrafil C10 M250 UNS of Company SGL Carbon Group and powder Al_2O_3 Alcoa CL 2500 was made in scanning electron microscope Zeiss Supra 25. The manufacturing of the ceramic preforms involved: the preparation of powders, their pressing and sintering. The powder Al_2O_3 Alcoa CL 2500 was wet - grinded with the use of distilled 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 set of carbon fibres Dolapix CE 64 of Company 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 distilled water, packed into the plastic bags and put away for 24h.

Table 2.
Properties of Al_2O_3 Alcoa CL 2500 powder

Density, g/cm^3	Particle diameter D50, µm	Mean mass concentration of elements, wt.%						
		Al_2O_3	Na_2O	Fe_2O_3	SiO_2	CaO	B_2O_3	The others
3.98	1.80	99.80	0.05	0.02	0.01	0.01	0.01	0.10

The powders were uniaxially pressed in the hydraulic press "Nelke" in steel mold with the inside diameter of 30mm. The picture of the used press is presented in Figure 1. 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 (Fig. 2). On the base of the thermogravimetric analysis (TGA) there was evaluated the thermical stability of carbon fibres C10 M250 UNS used as the pore forming factor in the ceramic performs manufactured from Al_2O_3 Alcoa CL 2500 powder. The temperature of total thermical degradation is about 700°C (Fig. 3) while under the temperature 400°C sample shows thermical stability.

Fig. 1. The laboratory hydraulic press "Nelke" equipped with pressing form of inside diameter 30 mm

Obtained results allows to optimize the program of sintering ceramic performs shown on Figure 4. There was applied slow heating 20°C/h up to 800°C remaining at that temperature by 10h. It was not decided to applying higher heating rate under the temperature of carbon fibres thermical stability (400°C) because compact includes the forming factor – polyvinyl alcohol (1%), activating by the distillate water (3%). Their rapid draining could be the reason of the preform's crackig.

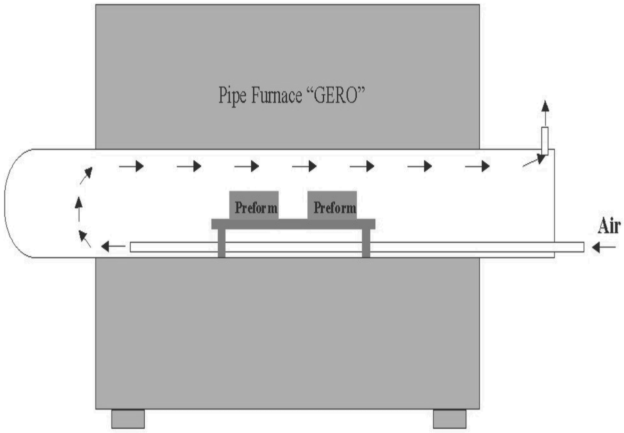


Fig. 2. Scheme of pipe furnace "GERO" used in sintering process

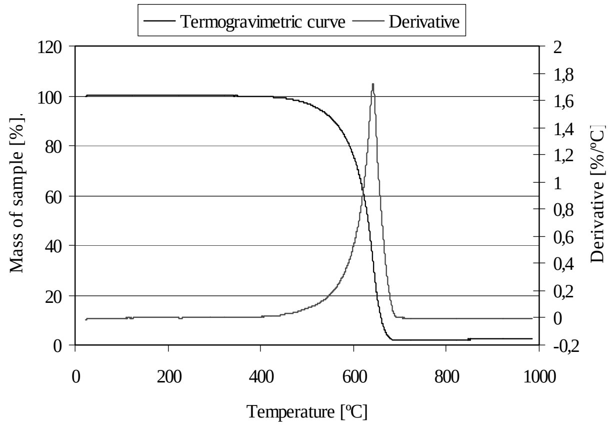


Fig. 3. Thermogravimetric curve of carcon fbibres Sigrafil C10 M250 UNS used as pores forming agent in ceramic performs

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 (Figure 5) for pumping over the liquid through the porous materials.

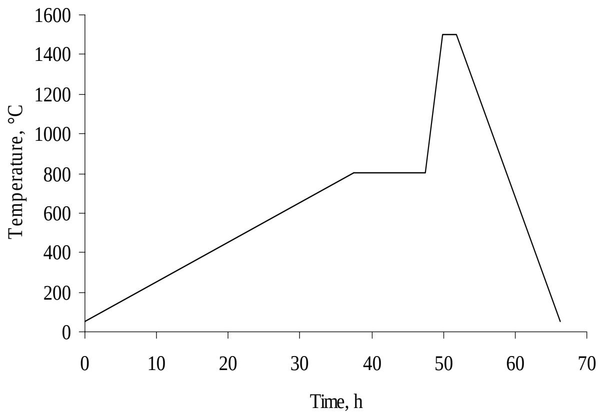


Fig. 4. Temperature performance during the sintering process

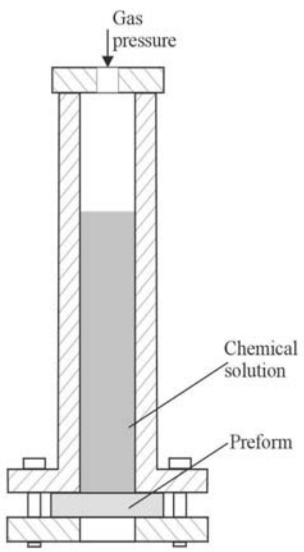


Fig. 5. The scheme of a permeability measure device of ceramic performs

During the test, the flow time of 350 ml 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:

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

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

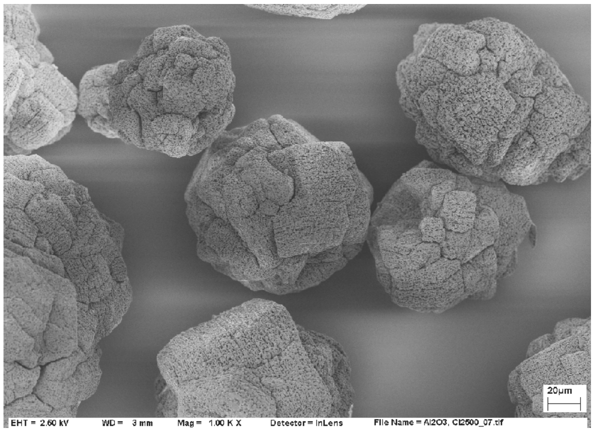
The observation of the structure of ceramic preforms fractures, manufactured by the sintering of Al_2O_3 Alcoa CL 2500 powder method was carried out using the Zeiss Supra 25 scanning electron microscope.

3. Experimental results and their discussion

The examination of structure of Al_2O_3 Alcoa CL 2500 powder when delivered and after the grinding process was carried out using scanning electron microscope (Figure 6). It ascertains that the agglomerations of ceramic particles were broken down during the grinding process, moreover the observation results helped to define the particles' diameter oscillating between 0.5 – 2 μm.

Carbon fibres Sigrafil C10 M250 UNS, manufactured by SGL Carbon Group observations carried out using scanning electron microscope confirmed their geometrical dimensions presented by the producer (diameter: 8μm and average length: 135 μm) (Figure 7)

a)



b)

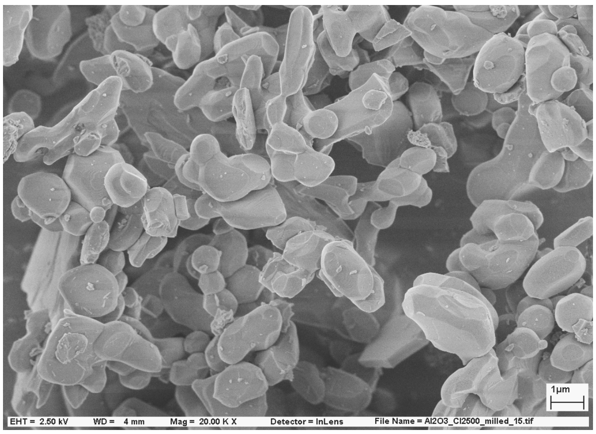


Fig. 6. The Al_2O_3 Alcoa CL 2500 powder structure: a) as delivered state magnification 1000x and b) after the grinding process, magnification 20.000x

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 3 and in Figure 8. Regarding to the optimal mechanical properties in the next researches the materials pressed only under 100MPa were used.

a)

b)

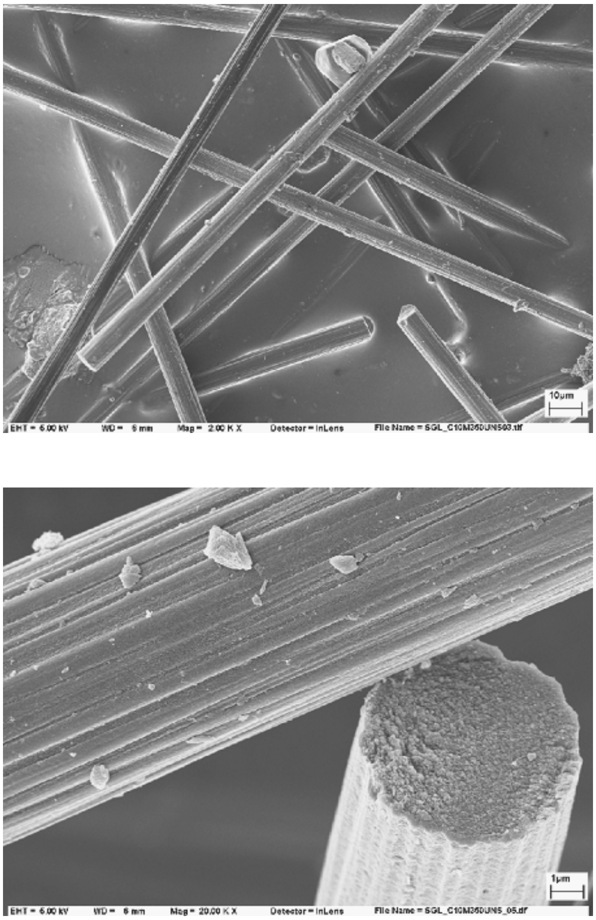


Fig. 7. Carbon fibres Sigrafil C10 M250 UNS, made by SGL Carbon Group a) magnification 2000x and b) magnification 20.000x

Measurements of permeability of ceramic preforms manufactured from powder of carbon fibres with mass fraction of 30, 40 and 50% pressed under weight of 100MPa 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 fractures of preforms manufactured by the sintering of Al_2O_3 Alcoa CL 2500 powder carried out using the scanning electron microscope (Fig. 9) 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 eliminatinge electrostatic effects.

Table 3.
Ceramic phase volume fraction in preforms obtained from different powders of different levels of carbon fibres addition, pressed under different pressure

Carbon fibres content, wt. %	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

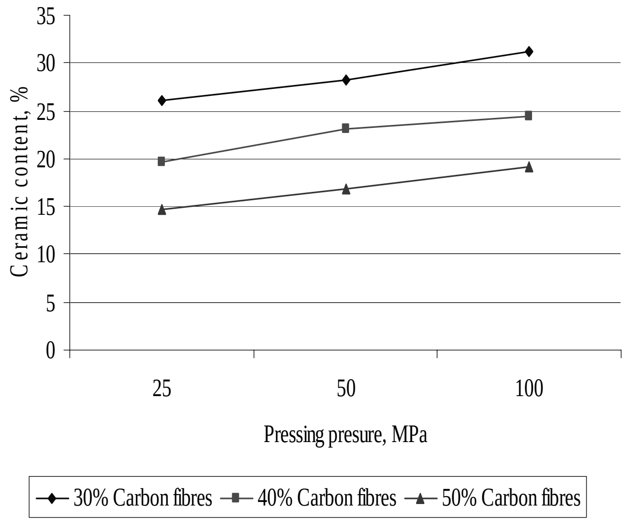


Fig. 8. Carbon fibres content and pressing pressure influence on ceramic phase volume fraction in preforms

4. Conclusions

The researches of Al_2O_3 Alcoa CL 2500 powder carried out using the scanning electron microscope allowed to define its particles diameter oscillating between 0.5 – 2μm. They also showed that the agglomerations of the particles during the grinding process where broken down, what was the main purpose of these operation.

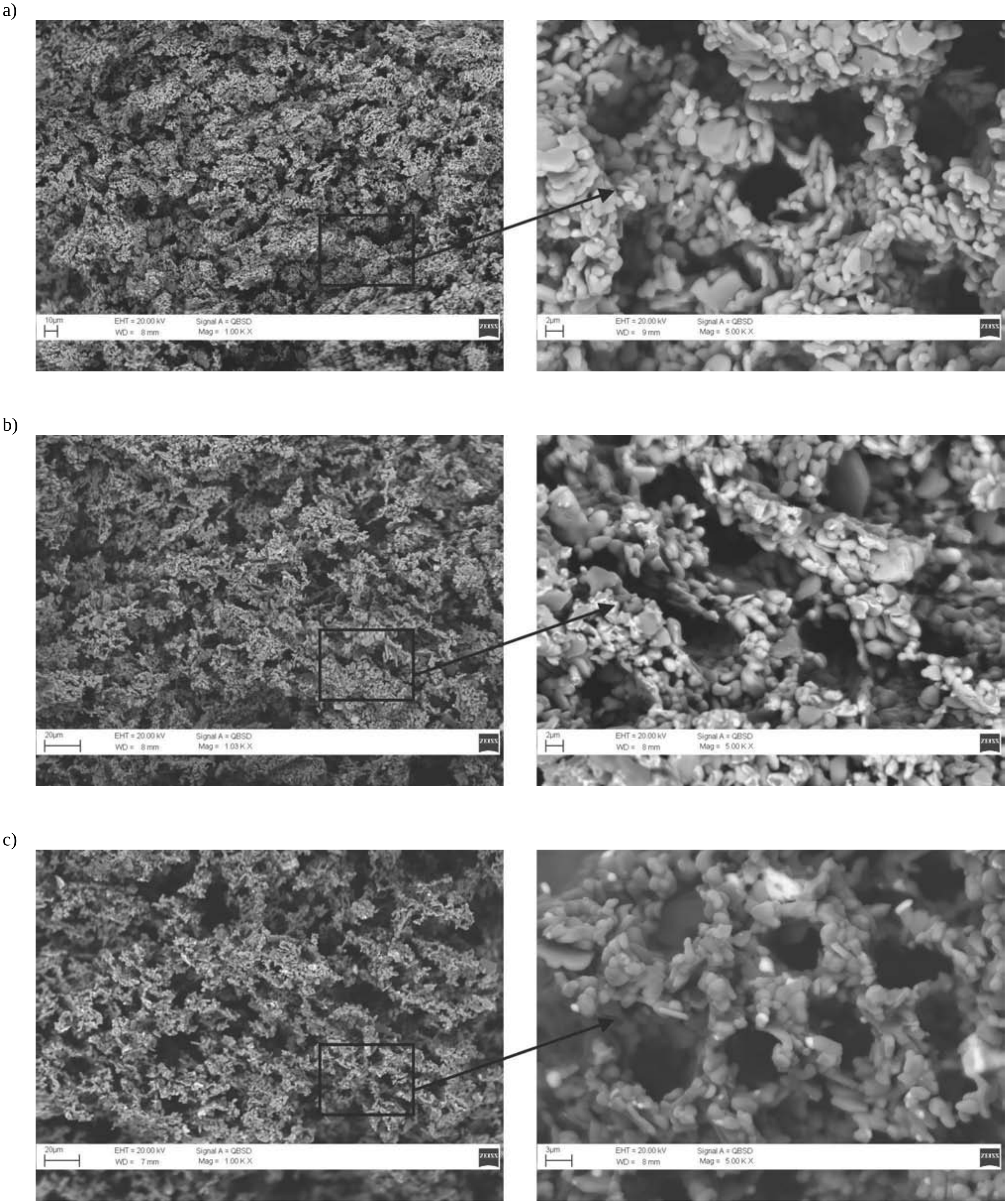


Fig. 9. The microstructure of ceramic preforms fracture based on Al_2O_3 powder with addition: 30 wt.%, b) 40 wt.% and c) 50 wt.% of carbon fibres

Carbon fibres Sigrafil C10 M250 UNS observations, carried out using scanning electron microscope confirm their geometrical dimensions declared by the producer – the SGL Carbon Group Company, presented in Table 1.

Geometrical dimensions and masses of the obtained ceramic materials measurements shows 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 Alcoa 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 semi finished products 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 porous preforms.

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