

The structure and properties of hybrid preforms for composites

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Received 14.03.2007; published in revised form 01.06.2007

Materials

ABSTRACT

Purpose: Article describes production technology of hybrid preform for reinforcing of composite materials. Some important properties such as shear strength and permeability were investigated in order to evaluate the usability of the preform for squeeze casting process.

Design/methodology/approach: The preforms produced by mixing of alumina „Saffil” fibers with graphite in form of flakes or fibers have open porosity. An inorganic binder used for production of preform ensures sufficient strength needed during high pressure infiltration process. The investigations of the structure of ceramic hybrid preforms on the scanning electron microscope (SEM) have been made.

Findings: The produced preforms in spite of worse permeability reveals open porosity and are suitable for infiltration process. Graphite with alumina fibers forms skeletal structure and can be easily incorporated into alumina matrix.

Research limitations/implications: Proposed method can be used for manufacturing of hybrid preforms with graphite fibers less than 10 vol. % due to its nonwettability by inorganic binders.

Practical implications: Obtained preform can be widely used as the reinforcement to produce hybrid composite materials by the infiltration method. Aluminum casting alloys can be locally reinforced to improve mainly strength at high temperature and wear resistance.

Originality/value: Article is valuable for persons, that are interesting in production of casting composite materials reinforced with ceramic preform. Proposed method allows incorporate graphite into preform with about 6,5 to 15,0% of Al_2O_3 fibers (Saffil).

Keywords: Composites; Ceramic preform; Alumina; Strength; Permeability

1. Introduction

The wide application of high-quality metal materials, meeting economic profitability and more and more severe ecological norm requirements is necessitated by both the tendencies to continuously upgrade the production and the requirements of everyday life. Recently the biggest group of such materials has been made up by composites, obtained by means of powder metallurgy or casting methods, which have been subject to extremely dynamic development. These materials, typically reinforced with fibres of ceramic particles, exhibit excellent strength properties in a wide temperature range [1,2], high

resistance to friction and fatigue wear [3,4,5,6], size stability and resistance to corrosion.

Presently the most prospective methods of producing cast composite materials include squeeze casting process, where a liquid alloy infiltrates a porous ceramic preform, which exhibits a certain intrinsic structure and planned porosity. The preforms are typically made on the basis of ceramic fibers of aluminium oxide, aluminosilicate of silicon carbide, calcium oxide and titanium. In the process of producing the preform a silicon or phosphor binder is usually used [7,8], which, after burning, should form small yet strong bridges between the fibers. A properly produced preform will allow for obtaining a composite exhibiting a certain content

Table 1.

Composition and properties of Saffil alumina short fibres and graphite components

Materials	Composition (wt.%)	Density (g/cm ³)	Tensile strength (MPa)	Young's modulus (GPa)	Diameter (μm)	Length (mm)
Saffil fibre	Al ₂ O ₃ -δ: 96-97 SiO ₂ : 2-4	3,3	2000	300	2-4	0,1-0,3
Graphite fibre	C:>99,9	1,8-2,0	1960	340	9-10	1-2
Graphite flakes	C:>99	2,26		12		

of the reinforcing fibers, at the same time not being deformed during the infiltration process when the head of the liquid metal stream might exert a considerable pressure on it [9]. Also the appropriate infiltration pressure and the matrix temperature should be adjusted to eventually obtain the correct link between the matrix and the reinforcing phase [10,11].

For hybrid ceramic preform other serious problem may occur during manufacturing. Very important is the uniform arrangement of all components inside the preform structure and its open porosity. In some cases different thermal properties of these component need a special operations. In the case of graphite, nonwetable and reactive at higher temperature [12,13], frying and sometimes infiltration must be performed under inert atmosphere. The graphite form of carbon due to unusual features can considerably change some properties of the composite. Its unique sort of texture consists of two-dimensional network structure. String covalent bonds hold atoms together in each layer, but the layers are bonded only by weak van der Waals forces, so the layer slide across one another readily. This property account for the widespread use of graphite as a lubricant. Graphite also has delocalized electrons, so it is a good electrical conductor in the direction of its layers.

2. Experimental procedure

The porous ceramic performs were made of Saffil ceramic fibers with an addition of graphite flake or graphite fibres, the chemical composition and properties of which are listed in Table 1. Saffil fibers contain ca. 3-4% of silica, which prevents aluminium oxide grain over-growth at higher temperatures and increases the resistance of the fibers to chemical corrosion [14]. The graphite flakes Els-395, fraction 0.062 and 0.37, manufactured by SGL Carbon Company, Nowy Sącz were of fractional purity. The graphite fibres made on the polyacrylonitrile precursor basis, produced by laboratory of ZEW Raciborz, contained at least 99.99 % C.

To make the performs wet forming process was used, incorporating a few stages [15]. First, an aqueous solution of the inorganic binder with an addition of an organic component which facilitates separation of the ceramic fibers, susceptible to segregation due to static charges was prepared. Then, after adding the respective amount of the fibers and graphite, the liquid was mixed, dried out, and, by squeezing, the perform was given the assumed form and porosity. The final stage of making the perform is its drying and burning at a proper temperature. To convert silica binder into its irreversible phase tridimite, preform was burned at 950°C temperature. Because graphite oxidizes above 450°C [16] so inert atmosphere of argon was applied. The performs exhibit a

skeleton structure, with a disorderly arrangement of the fibers and graphite in the horizontal plane, whereas the fibers are partially ordered in the vertical planes. Therefore, if performs are applied to reinforce elements of machinery carrying tensile loads, this fact should be made a not of.

The samples for the measurement of shear strength and permeability had a cylindrical shape of 6 mm high and 20 mm in diameter. The shear strength was measured using a universal device of LRu type for moulding sand. In similar way the permeability with use of special instrument to hold the samples was performed.

3. Discussion of the test results

Microscopic investigation were performed by means of scanning electron microscopy with X-ray analyzer. Observation of fracture surface after shear test revealed random distribution of alumina fibers with relatively uniform arrangement of graphite flakes (Fig.1). Mixing of graphite flakes with alumina fibers accomplished much easier then mixing with graphite fibers. Selection of solution concentration of the binder facilitates this process and allows for obtaining better structure of the performs and finally of the composites.

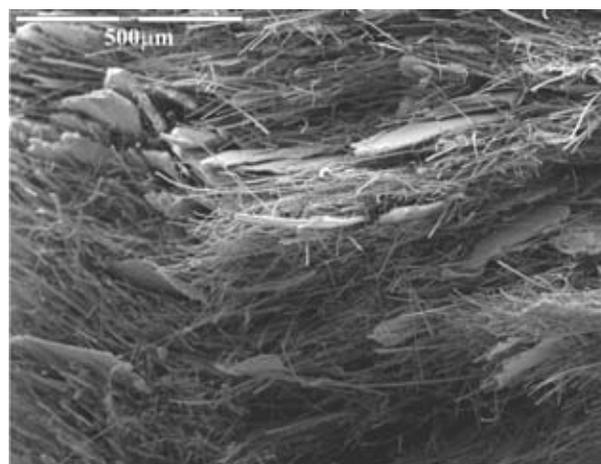


Fig. 1. The microstructure of ceramic preform based on Al₂O₃ fibers with addition of graphite flakes

The manufactured perform used in the infiltration processes in order to resist high pressure exerted by flowing liquid metal front should possess good strength. This condition can be performed when bonds between component of the perform are strong at high

temperature. In the case of graphite, which is usually nonwettable it is very difficult to form such connections. For the fabrication of our preform used silica binder readily forms small bonds between separate alumina fibers but only some kind of defect of the graphite, such as gaps, surface roughness or undulation allows to form connection and bond all components together.

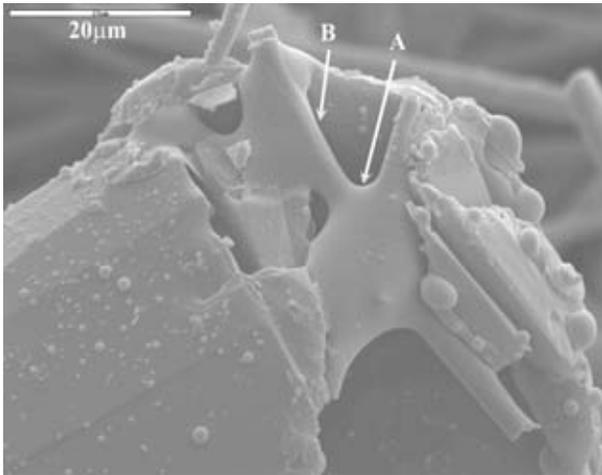


Fig. 2. The bond of alumina fibers between broken graphite flakes

Local X-ray analysis of a bonds between alumina fibers (Fig. 3(a)) and of the fibers surface (Fig. 3(b)) shown in Fig.2 at points A and B allow the ascertaining, that after firing the bond is composed mainly of applied inorganic binder, but on the other hand this binder is almost absent at the surface of the fibers. Graphite flake bonds to alumina fiber due to the local gaps and porosity, which are often in association with cracks. This is possibly only occasion to bond graphite and receive good strength, necessary to prevent preforms from deformation. During squeeze casting of the preform large forces caused by the infiltrating medium acts on fibers. On account of this, porous preform should posses proper strength in order to maintain the original shape and fiber distribution.

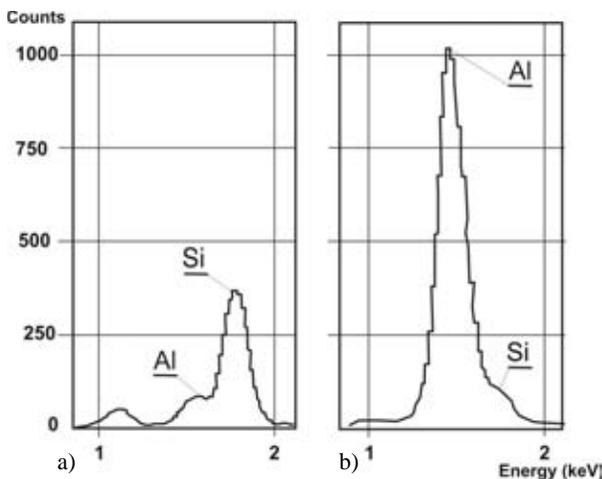


Fig. 3. Presenting X-ray analysis of: (a) the bond between alumina fibers at point A in Fig. 2 and (b) fiber shown at point B in Fig. 2

The shear strength of the preform, except other parameter such as the binder content and porosity depends strongly on the graphite content (Fig. 4). Greater strength is noticed for samples formed with less graphite content (3,3 vol.%). With increase of the graphite content we can observe nearly linear decreasing of the shear strength, which reaches its minimum value of 0,3 MPa at about 7,5 vol.% of graphite. Nonwettable graphite flakes act as a empty spaces and efficiently weaken the structure of preform.

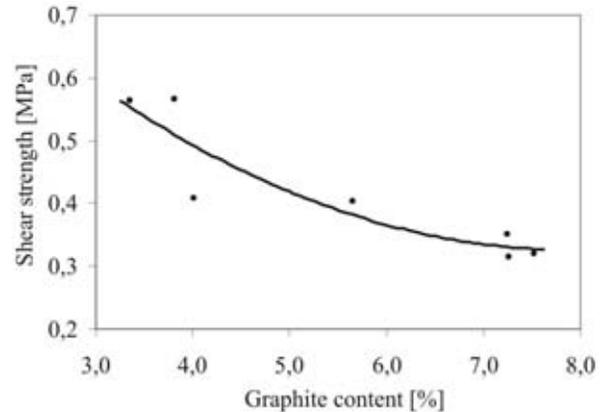


Fig. 4. The shear strength of the preform containing 8,4 vol. % of Al₂O₃ as a function of the graphite content

Next property significantly effects the infiltration qualify is permeability, frequently used to describe green sand moulding. It is defined as a measure of the ease with which air can pass through the sand aggregate, a property which is very sensitive to the sand size distribution, moisture and clay content and degree of compaction. Preform should be also characterised by open porosity making possible their infiltration by liquid metal alloy. Obtained data for permeability vs. % the carbon content have been calculated with formula:

$$P = \frac{V \cdot h}{p \cdot A \cdot t}$$

Where: P- permeability, p-pressure , A-cross section of preform, h - height of preform, V-volume of air, t-time.

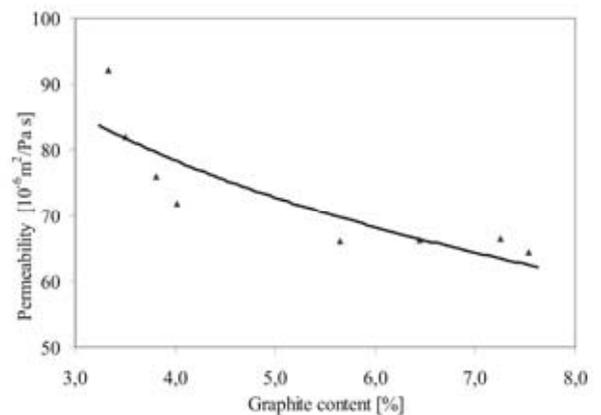


Fig. 5. The Permeability of the preform containing 8,4 vol. % of Al₂O₃ as a function of the graphite content

Figure 5 shows the effect of graphite content on the permeability of preform containing 8,4 vol.% Al_2O_3 and characterised by medium porosity within the range 82-87 %.

The value of the permeability was found to be very sensitive to graphite content especially when graphite is in form of flakes. An increase of the graphite content from 3,3% to 7,5% results in permeability decrease of ca. 50%. Even small amount of graphite can lower it drastically. But this minimum value for the preform with 7-8vol.% of graphite fibers allows to infiltrate and produce composite without any visible defect in the microstructure. Figure 6 shows the optical microstructures of as-cast Saffil short fiber (thin long shape) with graphite fiber (thicker dark shape) reinforced composite.

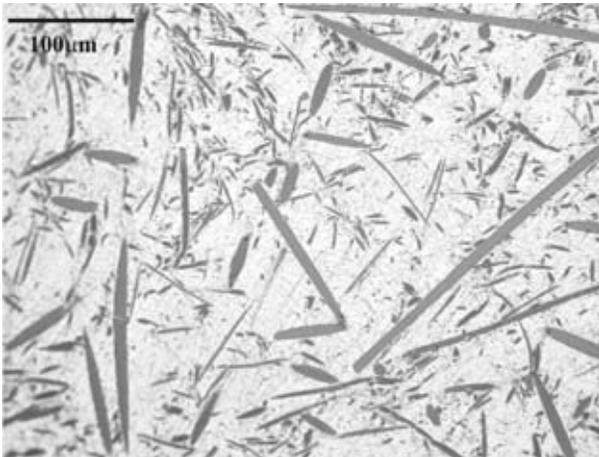


Fig. 6. Optical microstructure as cast Saffil/graphite fiber reinforced EN-AC AlSi7Mg composite in longitudinal cross section

4. Conclusions

The manufacturing process results in the production of porous fiber preform from Saffil Al_2O_3 fibers and graphite with open porosity, suitable for infiltration by liquid metal alloys. The performed tests allowed to determine the strength properties and the permeability of preform with addition of 3-8vol.% of graphite flakes. The result of this study confirm that for the $\text{Al}_2\text{O}_3/\text{C}$ system there is a relationship between the graphite content and the applicability of the preform for squeeze casting process. It was found out that graphite considerably decreases all properties especially the shear strength. A strength decreases in relation to the preform without graphite 50% for a preform containing 8% vol. of the graphite, and permeability decrease was 30%. In spite of such trends preforms possess open porosity and allows to produce sound composite materials.

In order to identify the bonding mechanism detailed structural investigations were performed using optical and electron microscopy and establishing relations between the results of such investigations and the physical properties of the preform. The microscopic observations confirmed obtaining a proper link between Saffil fibers. Graphite was nearly nonwetttable by inorganic binder. The good shear strength attained by the preform is due only to the strong bonds between Saffil fibers. During infiltration the load is mainly transferred by the fibers and at

relatively small amount of graphite liquid metal can easily flow and fill entire volume of preform.

References

- [1] Y.D. Huang, N. Hort, K.U. Kainer, Thermal behavior of short fiber reinforced AlSi12CuMgNi piston alloy, *Composites A35* (2004) 249-263.
- [2] C. Badini, P. Fino, M. Musso, P. Dinardo, Thermal fatigue behaviour of a 2014/Al₂O₃-SiO₂ (Saffil® fibers) composite processed by squeeze casting, *Materials Chemistry and Physics* 64 (2000) 247-255.
- [3] J.B. Yang, C.B. Lin, T.C. Wang, H.Y. Chu, The tribological characteristics of A356.2Al alloy/Gr composites, *Wear* 257 (2004) 941-952.
- [4] A. Daoud, Wear performance of 2014 Al Alloy reinforced with continuous carbon fibres manufactured by gas pressure infiltration, *Materials Letters* 58 (2004) 3206-3213.
- [5] M. Kok, Production and mechanical properties of Al₂O₃ particle-reinforced 2024 aluminium alloy composites, *Journal of Materials Processing Technology* 161 (2005) 381-387.
- [6] J. Myalski, J. Wiczorek, A. Dolata-Grosz, Tribological properties of heterophase composites with an aluminium matrix, *Journal of Achievements in Materials and Manufacturing Engineering* 15 (2006) 53-57.
- [7] J.M. Chiou, B.Y. Wei, C.M. Chen, The effects of binders and heating temperatures on the properties of preforms, *Journal of Materials Engineering and Performance* 2 (1993) 383-392.
- [8] D.D.L. Chiou Jeng-Maw, D.D.L. Chung, Improvement of the temperature resistance of aluminium-matrix composites using an acid phosphate binder, Part I Binders, *Journal of Materials Science* 28 (1993) 1435-1446.
- [9] C.G. Kang, Y.H. Seo, The influence of fabrication parameters on the deformation behaviour of the preform of metal-matrix composites during the squeeze-casting processes, *Journal of Materials Processing Technology* 61 (1996) 241-249.
- [10] J.M. Chiou, B.Y. Wei, C.M. Chen, The effects of binders and heating temperatures on the properties of preforms, *Journal of Materials Engineering and Performance* 2 (1993) 383-392.
- [11] S. Cardinal, M. R'Mili, P. Merle, Improvement of high pressure infiltration behaviour of alumina preforms: manufacture and characterization of hybrid preforms, *Composites Part A* 29A (1998) 1433-1441.
- [12] K. Landry, S. Kalogeropoulou, N. Eustathopoulos, Wettability of carbon by aluminum and aluminum alloys, *Materials Science and Engineering A254* (1998) 99-111.
- [13] O. Dezellus, N. Eustathopoulos, The role of Van der Waals interactions on wetting and adhesion in metal/carbon systems, *Scripta Materialia* 40/11 (1999) 1283-1288.
- [14] Imperial Chemical Industries, Company's Prospect „SAFFIL Alumina Fibre” 1996.
- [15] K. Naplocha, A. Janus, J. Kaczmar, Z. Samsonowicz, Technology and mechanical properties of ceramic preforms for composite materials. *Journal of Materials Processing Technology* 106/1-3 (2000) 119-122.
- [16] L.A. Dobrzański, M. Kremzer, A. Nagel, B. Huchler, Fabrication of ceramic preforms based on Al₂O₃ CL 2500 powder, *Journal of Achievements in Materials and Manufacturing Engineering* 18 (2006) 71-74.