Porous ceramic preforms dedicated for local reinforcement subjected to the squeeze casting infiltration process

Key words
Metal matrix composites, squeeze casting.

Abstract
An attempt was made to obtain ceramic preforms with different contributions of porosity. Compressive strength tests were carried out to estimate the ability of ceramic preforms to be utilised in the process of squeeze casting for the fabricating of metal matrix composites. The achieved ceramic preforms were subjected to the squeeze infiltration process, and materials with much higher mechanical properties relative to monolithic materials were obtained.

Introduction
Metal matrix composites are widely used in many branches of the industry due to an interesting set of features that better or different in relation to monolithic materials. It allows the application of metal matrix composites where other materials do not meet the assumed parameters, e.g., high strength, abrasion resistance, creep resistance, etc., in the whole volume of a casting or its fragment [1–2]. A few basic methods used for fabricating metal matrix composites can be distinguished, although casting methods are most widely applied.
Depending on the type or the reinforcing phase and liquid metal, the following variations of the squeeze casting process can be distinguished [3]:

1. Squeeze infiltration (compression impregnation, high-pressure infiltration casting) of a porous ceramic preform (fibrous, skeletal, or dispersive) which results in a ready casting, a preshaped product, or semi-finished composite material (reinforcement insert);

2. The fabrication of a casting with local reinforcement – by a composite semi-finished product or a porous ceramic preform with its simultaneous high-pressure infiltration;

3. The fabrication of a dispersion particulate reinforced casting, (the reinforcing phase is present in the form of particles, whiskers, or short fibres); or,

4. The fabrication of a hybrid composite casting, reinforced by a preform (sometimes with simultaneous infiltration), and a dispersion phase.

From the utilitarian point of view, the method of high-pressure infiltration is the most useful, which is presently one of the most perspective ways of fabricating particular materials. With the use of this process, a wide scope of materials can be achieved with relatively low outlays [4]. The short time for the solidification of the metal matrix limits its reactiveness with the reinforcing phase, and the high pressure of infiltration guarantees both adequate quality of fabricated heterogeneous materials – practically without participation of porosity, and considerably improves wettability of the reinforcing phase by the liquid matrix. The short time for the solidification of the matrix also limits unfavourable reactions on the boundary of the matrix-reinforcement.

The final properties of metal matrix composites are mainly determined by a proper distribution of the reinforcing phase in the structure of the heterogeneous material. That is why a favourable solution is the introduction of porous preforms to liquid metal that include particles or fibres with a given spatial distribution (homogeneous) of the reinforcing phase [5].

The article describes methods for the fabrication of ceramic preforms designated for local reinforcement of castings achieved with the high-pressure infiltration process. The produced material must be characteristic of the high participation of open porosity and adequately high compressive strength.

The fabrication of a ceramic preform is a very difficult task due to a number of problems that occur. The most important problems are the following [6]:

- Difficulties in obtaining a homogeneous distribution of porosity and sizes of pores, and
- The low repeatability of the process.

1. The course of the research

In the initial stage of research, the input materials were selected for the fabrication of ceramic preforms, of which sample mixtures of ceramic powders with different contributions of pore-creating factors were prepared. Then
mixtures of powders were pressed and sintered at different parameters of processes. The obtained preforms were tested for total and open porosity, as well as compressive strength. Preforms with satisfactory parameters underwent scanning microscopy tests in order to reveal their internal structure. After that, a series of sample composite castings were made infiltrating preforms with liquid 7075 aluminium alloy.

2. The selection of reinforcing materials

A proper selection of ceramic materials for the process of squeeze infiltration constitutes a highly important stage, since physical-chemical properties of the reinforcing phase have a significant impact on the final properties of the heterogeneous material. Based on the analysis of topics in the literature on reinforcing materials and technical possibilities of their later processing, the following reinforcing phases were selected for further tests (attempts to fabricate porous preforms):
- Saffil® continuous fibres
- α – aluminium oxide particles with two diameters of grain (NABOND 0.1 μm + ALCOA 23–48 μm).

3. Fabrication of porous preforms

In order to fabricate preforms with a high participation of open porosity, the following methods were applied:
- Introducing different sizes of particles,
- The addition of organic substances, and
- The addition of removable additions.

Mixtures of powders of aluminium oxide with different sizes of grain (submicron and approx. 35 μm) 1:1 ratio and cut Saffil® fibres were mixed in a high-energy ball mill for 5 minutes with an addition of distilled water and polyvinyl alcohol as a separator, which prevents lumping of ceramic material at 1% by weight. Next, colloidal silica (LUDOX® AS 40) was added to the aforementioned mixture – binding at approx. 5% by weight and the following substances that increase porosity (5–40% by weight):
- Metacellulose (cz.),
- Sawdust,
- Triethanolamine (POCH, Ph.Eur.), and
- Polyvinyl alcohol (POCH).

The material prepared in this manner was mixed once again in the ball mill for 5 minutes. Dried mixtures were introduced into the casting mould installed in a hydraulic press, after which, with the use of a punch, they obtained the final shape (squeeze casting at different pressures 30, 50 and 100 MPa). A cylindrical
mould was used with a diameter of 48 mm. Preforms underwent the process of sintering at changeable temperatures: 900, 1100 and 1300°C for 2 hours in order to give them proper mechanical properties.

4. Results and discussion

Preforms with the range of porosity from 0% (solid ceramics) to 75% (sawdust) as the pore-creating agents were obtained. The participation of porosity achieved in given variants was measured in three ceramic samples. Significant scatter of porosity was observed in the examined material.

Table 1 presents process parameters for the fabrication of ceramic preforms and the obtained total and open porosity.

<table>
<thead>
<tr>
<th>No.</th>
<th>Initial material</th>
<th>Pore-creating agent</th>
<th>Content [% by weight]</th>
<th>Squeeze pressure [MPa]</th>
<th>Sintering temperature [°C]</th>
<th>Porosity [%]</th>
<th>Open porosity [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Al₂O₃ (0.1 ±35 µ)</td>
<td>-</td>
<td>-</td>
<td>30</td>
<td>1100</td>
<td>18.3</td>
<td>7.5</td>
</tr>
<tr>
<td>1.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>50</td>
<td>1100</td>
<td>5.9</td>
<td>0</td>
</tr>
<tr>
<td>1.3</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>100</td>
<td>1100</td>
<td>2.1</td>
<td>0</td>
</tr>
<tr>
<td>2.1</td>
<td>methylocellulose</td>
<td>10</td>
<td>30</td>
<td>1100</td>
<td>31.2</td>
<td>28.6</td>
<td></td>
</tr>
<tr>
<td>2.2</td>
<td>methylocellulose</td>
<td>20</td>
<td>30</td>
<td>1100</td>
<td>47.7</td>
<td>43.9</td>
<td></td>
</tr>
<tr>
<td>2.3</td>
<td>methylocellulose</td>
<td>40</td>
<td>30</td>
<td>1100</td>
<td>57.3</td>
<td>55.9</td>
<td></td>
</tr>
<tr>
<td>3.1</td>
<td>sawdust</td>
<td>10</td>
<td>30</td>
<td>1100</td>
<td>26.2</td>
<td>24.3</td>
<td></td>
</tr>
<tr>
<td>3.2</td>
<td>sawdust</td>
<td>30</td>
<td>30</td>
<td>1100</td>
<td>63.5</td>
<td>62.5</td>
<td></td>
</tr>
<tr>
<td>3.3</td>
<td>sawdust</td>
<td>40</td>
<td>30</td>
<td>1100</td>
<td>76.1</td>
<td>75.8</td>
<td></td>
</tr>
<tr>
<td>4.1</td>
<td>triethanolamine</td>
<td>5</td>
<td>30</td>
<td>1100</td>
<td>23.7</td>
<td>20.9</td>
<td></td>
</tr>
<tr>
<td>4.2</td>
<td>triethanolamine</td>
<td>10</td>
<td>30</td>
<td>1100</td>
<td>29.5</td>
<td>25.2</td>
<td></td>
</tr>
<tr>
<td>4.3</td>
<td>triethanolamine</td>
<td>20</td>
<td>30</td>
<td>1100</td>
<td>Spalling</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.1</td>
<td>polyvinyl alcohol</td>
<td>10</td>
<td>30</td>
<td>1100</td>
<td>After removing from the furnace preforms disintegrated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.2</td>
<td>polyvinyl alcohol</td>
<td>20</td>
<td>30</td>
<td>1100</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.3</td>
<td>polyvinyl alcohol</td>
<td>40</td>
<td>30</td>
<td>1100</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Saffil®</td>
<td>-</td>
<td>-</td>
<td>1100</td>
<td>69.3</td>
<td>69.1</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Al₂O₃ + Saffil®</td>
<td>-</td>
<td>-</td>
<td>1100</td>
<td>47.4</td>
<td>46.8</td>
<td></td>
</tr>
</tbody>
</table>
The highest quality of preforms was obtained with the application of sawdust as the addition (variation No. 3). The result is surprising, because burning sawdust produces gases and releases contamination, which may cause the degradation of the structure of preforms. Due to this fact, it was believed that methylcellulose, which is a purer material, would be better in the role of a pore-creating agent. Ceramic preforms in variation No. 3 were characteristic of open porosity contribution within the range from 24 to 75%. Figure 1 presents selected examples of achieved ceramic preforms. Both variation No. 1 (solid structure – porosity 0%), and variation No. 2 (big, lumped Al₂O₃ particles) disqualify the fabricated material to be infiltrated with liquid metal.

![Selected examples of fabricated ceramic preforms](image)

**Fig. 1.** Selected examples of fabricated ceramic preforms: a) preform made of solid ceramics, b) macrostructure, c) preform’s microstructure, d) preform with 56% porosity contribution together with its: e) macro- and f) microstructure, g) variation 3.3 with 75% porosity contribution, h) macrostructure, i) preform’s microstructure

Excellent effects were obtained with the use of continuous Saffil® fibres as ceramic material, which were cut and ground in the ball mill in order to reduce
their dimensions. The photograph of the preform, its macro- and microstructure is presented in Fig. 2.

Fabricated ceramic preforms in variation No. 6 are characteristic of an open porosity of 70%, with insignificant closed porosity (0.2%), which predestines them for squeeze infiltration attempts. Compressive strength tests were carried out for the fabricated preforms. The achieved results are presented in Table 2.

![Fig. 2. A research variation of a fabricated ceramic preform made of cut Saffil®: a) a photograph of a solid ceramic preform, b) macrostructure, c) preform microstructure](image)

<table>
<thead>
<tr>
<th>Initial material</th>
<th>Pore-creating agent</th>
<th>Content [% by weight]</th>
<th>Open porosity [%]</th>
<th>Compressive strength [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃ (0.1 +35 µm)</td>
<td>methylcellulose</td>
<td>20</td>
<td>43.9</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>methylcellulose</td>
<td>40</td>
<td>55.9</td>
<td>7.5</td>
</tr>
<tr>
<td></td>
<td>sawdust</td>
<td>30</td>
<td>62.5</td>
<td>13.5</td>
</tr>
<tr>
<td>Saffil®</td>
<td>–</td>
<td>–</td>
<td>69.1</td>
<td>8.2</td>
</tr>
<tr>
<td>Al₂O₃ (0.1 +35 µm)</td>
<td>sawdust</td>
<td>40</td>
<td>75.8</td>
<td>2.4</td>
</tr>
</tbody>
</table>

5. Squeeze infiltration of fabricated ceramic preforms

For infiltration attempts preforms were selected with an open porosity contribution above 50%. The selected preforms underwent infiltration with liquid 7075 aluminium alloy. The preheated preforms were infiltrated with liquid metal with the use of “bottom” infiltration. The tests were made in a UBE VSC 500 hydraulic press. The external pressure was imposed directly on the liquid metal surface (direct squeeze casting) with the use of a piston. The pressure equalled 150 MPa, and time was 60 seconds. Figure 3 presents the microstructure of a composite casting fabricated with the use of the squeeze infiltration method of Saffil® fibres with aluminium alloy (7075) for mechanical working.
Conclusions

1. Preforms made of powder ceramics (Al₂O₃) and Saffil® fibres were fabricated.
2. The achieved preforms were characteristic of open porosity within the range from 0 to 75%.
3. A significant impact of additions of pore-creating substances on the content of ceramic preforms was observed.
4. The best effects (the highest porosity and compressive strength) were achieved with the application of sawdust as the pore-creating substance.
5. The fabricated ceramic preforms are characteristic of differentiated compressive strength (from 2.4 to 55 MPa in variations with open porosity above 50%).
6. For the attempts of squeeze infiltration, preforms were used fabricated from Al₂O₃ powders with an addition of sawdust and methylcellulose (open porosity > 50%) and Saffil® fibres (70%).
7. The high efficiency of infiltrating preforms with liquid metal was proved.

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References


Porowate kształtki ceramiczne przeznaczone do lokalnego zbrojenia odlewów sposobem infiltracji ciśnieniowej

Słowa kluczowe
Kompozyty metalowo-ceramiczne, prasowanie w stanie ciekłym.

Streszczenie

Podjęto próbę uzyskania preform ceramicznych o różnym stopniu porowatości.

Przeprowadzono badania ich wytrzymałości na ściskanie w celu określenia możliwości ich zastosowania w procesie otrzymywania kompozytów metalowo-ceramicznych technologią prasowania w stanie ciekłym. Otrzymane preformy poddano infiltracji ciśnieniowej i otrzymano kompozyty o znacząco wyższych właściwościach wytrzymałościowych w stosunku do odlewów monolitycznych.