Structure and properties of porous ceramic preforms made of α-alumina particles

J. W. Kaczmar, A. Kurzawa
Institute of Mechanical Engineering and Automation, Wrocław University of Technology, ul. Łukasiewicza 5, PL-50-371 Wrocław, Poland

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Abstract

Properties of ceramic preforms made of α-alumina particles for strengthening of metal based composite materials manufactured by squeeze casting are discussed in the paper. Composite materials strengthened with ceramic particles can be then plastically worked in order to remove the residual porosity and give the final net shape. There was investigated the permeability of porous preforms as the measure of the open porosity making possible the infiltration process with the molten metal, compression strength, bending strength and shear strength determining the stability of porous ceramic preforms during infiltration. Performed investigations allow to ascertain the occurrence of the open porosity in the porous preforms and strong effect of porosity on permeability. The largest permeability of 19 m²/Pa·s showed porous preforms characterized by porosity of 90%. The strength properties increased with the decrease of preforms porosity and the preforms characterized by 60% of porosity were characterized by the largest strength properties (bending strength $R_b = 7$ MPa, compression strength $R_c = 11.5$ MPa, shear strength $R_t = 4.0$ MPa). The microscopic observations of fractures surfaces allow to evaluate quality and forming phenomena of the binder bridges connecting the ceramic particles.

Keywords: cast composites, ceramic preforms, strengthening of metal alloys, squeeze casting.

1. Introduction

From among applied manufacturing methods of composite materials the squeeze casting method of infiltration of porous preforms made of fibres [1-3] and particles [4-6] is widely applied. This is connected with the advantages of this method, making possible the manufacturing of machine and devices elements strengthened locally at the given place where the expected loads are the largest or intensive wear can take place. Besides the technical advantages, the production costs of such elements can be lowered, according to the fact that amount of ceramic material applied as the ceramic preforms made of particles or fibres can be limited. In order to manufacture the element with the local strengthening, the ceramic preform is placed in the desired place in the casting mould, molten metal alloy is poured into the mould and then the punch exerts the pressure on the molten metal which penetrates into the open porosity of the porous ceramic preform. Application of squeeze casting method makes possible the net shape manufacturing of elements locally strengthened with ceramic preforms, what takes place in the case of metal alloy strengthening with the preforms made of ceramic fibres. On the other hand elements strengthened with the ceramic preforms made of particles can be further processed by plastic working methods like forging or rolling. During plastic working of such materials the bridges between particles are destroyed and the separate particles are transported in the relatively soft matrix, what effects in the increasing of the distribution homogeneity of particles in the matrix and decreasing of the residual porosity after infiltration process. The ceramic preforms should be characterized by the structure of the open porosity and good strength, making possible the infiltration with the molten metal alloys and maintaining the desired shape of ceramic preform during infiltration.

The manufacturing process of porous preforms consisted of preparation of mixture composed of binder water solution, alumina particles and porophor (pores forming agent). In order
to get the desired dimensions of preforms, the slurry was poured into the mould and then heated. During heating the excess of water evaporated, binder hardened and formed bridges between ceramic particles. Porophor transformed into gas forming the open porosity in the ceramic preform. The parameters of the heating process should be kept very strictly and application of improper heating parameters effects on the formation of defects in the preforms. The defects can occur as the gas cavities, cracks or delaminations. The flow sheet of the manufacturing process is schematically shown at Fig.1.

2. Range of investigations

The investigations of physical and mechanical properties were performed at the ceramic preforms made of α-alumina particles shown at the Fig.2. Particles are characterized by irregular shape with the particle size of 3-5 µm. Manufactured preforms are characterized by porosities of 90%, 80%, 70% and 60% and they will be further applied for the strengthening of aluminium alloys. In the paper the physical and mechanical properties of ceramic preforms: permeability, bending strength, shear strength and compression strength in the function of preforms porosity were studied.

3. Physical and mechanical properties of preforms

Investigations of permeability were performed basing on permeability standard measurement method during investigations of the moulding sands. The measurements were performed applying the LPiR-2e device on the samples in the form of cylinder of diameter of 20 mm and height of 10 mm. The permeability was interpreted as the measure of open porosity making possible the infiltration of preforms with the molten metal alloys. The results of permeability investigations are shown at Fig.3.

![Flow sheet of the manufacturing process of porous ceramic preforms made of α-alumina particles](image1)

![Permeability of ceramic preforms characterized by porosities of 60-90%](image2)

Preforms with the smallest porosity of 60% were characterized by the smallest permeability of 3 m²/Pa·s and on the base of this information it can be drawn the conclusion on the limited ability for the infiltration process of such preforms. In this case the flow resistance for the liquid metal front during squeeze casting process is relatively large and significant forces act on the porous preform. Then in such preforms defects like deformations of preform or cracks can occur.

Preforms with larger porosity are characterized by larger amount of pores and small channels, what effects on their higher permeability. In preforms with 70-90% of porosity there is observed the rapid growth of permeability which can be evaluated for 6 m²/Pa·s for the 10% increase of porosity. The preforms with the largest porosity were characterized by the permeability of P = 19 m²/Pa·s. Such permeabilities, especially in preforms characterized by 80% and 90% of porosity are sufficient for the infiltration with the molten metal alloys.

During infiltration of porous ceramic preforms with liquid metal alloys large forces caused by metal front and the relatively high infiltration pressure act on the preform. The forces can destroy the binder bridges connecting particles, deform the ceramic preform, what can be the reason of preform deformation and finally generation of cracks and delaminations. Investigations of mechanical properties give informations about the dimensional stability of preforms during pressure infiltration. Investigations of mechanical properties of ceramic...
preforms were performed applying the device of LRu type normally applied for strength investigations of moulding and core sands. There are presented results of bending strength $R_g$, shear strength $R_t$, and compression strength $R_c$ of preforms characterized by porosities in the range of 60 - 90%.

The investigations of bending strength $R_g$ were performed applying the rectangular samples of 60 x 20 x 10 mm. There was applied one point force loading and distance between supports of 50 mm. The results of investigations are shown at Fig. 4 and they revealed that there is strong relationship of bending strength from porosity of preforms. The smallest bending strength of 1.5 MPa shows samples characterized by porosity of 90% and this strength is almost 80% lower than the bending strength of samples characterized by 60% of porosity which are characterized by the bending strength of 7 MPa. The significant increase of bending strength with decreasing porosity is connected with larger number of bridges between ceramic particles in such preforms.

There is strong effect of porosity on shear strength. Samples characterized by the largest porosity of 90% show the smallest shear strength of 0.3 MPa. Decreasing of porosity to 80% slightly affects on the improvement of shear strength (increase to 0.6 MPa), but the considerable increase of shear strength is noticed for samples characterized by porosities of 70 and 60%. The maximum shear strength of 4 MPa was reached for samples characterized by 60% of porosity.

Compression strength reflects the ability of porous preforms to keep their shape and dimensions during infiltration with molten metal. Taking into account that applied pressures of squeeze casting are in the range 80-120 MPa, then large forces act on preform during squeeze casting with liquid metal alloys. The largest compression strength of $R_c = 11.5$ MPa shows samples characterized by 60% of porosity and the compression strengths in the function of porosity are shown at Fig. 6. Samples of 60% of porosity show 60% larger compression strength than samples of 70% porosity.

The shear strength of preforms was determined applying the cylindrical samples of diameter 20 mm and height of 10 mm and results are shown at Fig. 5. Observations of macrostructure fractures of preforms showed that applying of improper process parameters of porophor gasification leads to the generation of macro- and microcracks in the ceramic preforms (Fig.7), effecting on the lowering of the compression strength.
mechanical and development properties of composite materials after squeeze casting process. Although the infiltration of such preforms is easier, later in the service of such composite parts it can lead to the cracks resulting in the damages of the composite elements.

![Fig. 7. Fracture of porous preform of 80% porosity with visible cracks.](image)

Such problems do not occur in manufacturing process of ceramic preforms made of ceramic fibres, according to the fact that in such preforms larger pores of open character are mainly formed during production. On the other hand such preforms can be manufactured with the minimum porosity of 80% and contrary, the preforms made of ceramic powders can be manufactured with 60% of porosity.

On the other hand on the base of the performed experiments it was ascertained that proper firing and gasification parameters of porophor makes possible manufacturing of porous preforms from ceramic particles without cracks, suitable for the infiltration process (Fig.8). The alumina particles are connected with the bridges of the binder and are homogeneously distributed in the ceramic preform (Fig.9).

![Fig. 8. Fracture of ceramic preform of 80% porosity.](image)

Observations of preforms applying SEM at larger magnifications reveals the relatively homogeneous distribution of ceramic particles in the preform (Fig.10a) and binder bridges between ceramic particles (Fig.10b).

![Fig. 10. Fractures of ceramic preform of 80% porosity: a) structure of preform  b) bridges between ceramic particles](image)
In order to investigate the chemical composition of bridges connecting particles and layer covering particles the EDS analysis was applied. The places of EDS analysis at the surface of ceramic particles are designated as Spectrum 1 and at binder bridges between particles formed by the binder are designated as Spectrum 2 at Fig. 10b. The results of EDS analysis are shown at the Fig. 11 and the qualitative results of this analysis in the Table 1.

![EDS analysis of ceramic preform made of α-alumina particles](image)

**Fig.11.** EDS analysis of ceramic preform made of α-alumina particles shown at FIG. 10 b; a) EDS analysis of the particle surface designated as Spectrum1 and b) analysis of the bridge at the place designated as Spectrum 2

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
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<td>58.00</td>
<td>O</td>
<td>33.37</td>
<td>45.86</td>
</tr>
<tr>
<td>Na</td>
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<td>Na</td>
<td>0.70</td>
<td>0.67</td>
</tr>
<tr>
<td>Al</td>
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<td>39.81</td>
<td>Al</td>
<td>58.32</td>
<td>47.52</td>
</tr>
<tr>
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<td>1.62</td>
<td>Si</td>
<td>7.61</td>
<td>5.95</td>
</tr>
<tr>
<td>Total</td>
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Preliminary infiltration experiments with the liquid aluminium alloys were performed in order to investigate the possibility of liquid metal penetration into the ceramic preforms. Additionally plastic working of manufactured composite materials was performed and this process was realized by rolling. Plastic working can reduce the residual porosity and on the other hand the porosity can be reduced by alloying methods [7]. Both plastic working and casting methods can be good methods for net shape or near net shape manufacturing of machine elements [8, 9], although plastic working ensures better dimensional accuracy of elements.

On the base of EDS analysis taking additionally into account the EDS qualitative analysis shown in Table 1 it is evident that the bridges between particles and layer covering particles are characterized by the same chemical composition. Peaks of sodium (Na) and silicon (Si) reflect the chemical composition of the applied binder and the peaks of aluminium (Al) and oxygen (O) reflect the chemical composition of alumina. This kind of binder is widely applied for the manufacturing of porous preforms based on SiC particles as well [10]. The presence of the binder at the surface of particles indicates good wettability of α-alumina particles by the applied binder, what effects on the relatively good strength of the ceramic porous preforms.

5. Conclusions

On the base of performed investigations it was ascertained that is possible manufacturing of porous preforms characterized by the open porosity from the α-alumina powders suitable for pressure infiltration with molten metal alloys. The following conclusions can be drawn:

1. Correct production process of ceramic preforms from α-alumina particles consists of mixing of components, preparation of the slurry and forming of the slurry by pouring into the form. After drying of preform and gasification of porophor the firing takes place making possible manufacturing of preforms characterized by the relatively good strength. Achieved strength make possible the handling of preforms.

2. Elaborated technology makes possible manufacturing of preforms from ceramic α-alumina powders of particle size 3-5µm characterized by the open porosity in the range of 60 - 90 %.

3. The smallest porosity of 60 % results in the relatively good permeability of 3 m²/Pa·s. The preliminary infiltration (squeeze casting) with liquid aluminium alloys of such preforms was performed and showed that fully infiltrated composite material was manufactured. The shape of porous preform was kept during infiltration and the mechanical strength of preforms is sufficient to keep their initial shape and dimensions during squeeze casting process.

4. Possibility of placing of porous preform at the determined place of the cast die makes possible the strengthening of cast materials at the places where maximum loads are concentrated or large wear takes place.

5. The composite materials strengthened with ceramic particles contrary to the composite materials strengthened with...
ceramic fibres can be plastically worked by forging or rolling. In this case the net shape manufacturing with large accuracy can be achieved. On the other hand the maximum contents of alumina powders enabling deformation process without cracks should be further investigated.

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Literature