Wax-based binder for low-pressure injection molding and the robust production of ceramic parts

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Abstract

In this paper, a wax-based binder was developed for low-pressure injection molding of very fine (0.4 μm) average particle size alumina ceramic bodies. The major component used in the binder mixture was paraffin wax. The process of binder removal from the green ceramics presented several difficulties especially when the ceramic powder was very fine, and the pieces had large cross-section.

The process used for debinding was wicking in an alumina powder bed of the same powder. With the binder formulation presented here, and the optimization of the ramps and dwells furnace programmation for the debinding, it was possible to produce sintered alumina bodies with large cross-section and 98.5% of the theoretical density, free of defects.

Keywords: low pressure injection molding, very fine alumina, large cross-section parts.

1. Introduction

Low-pressure injection molding (LPIM), or hot molding of ceramics, has been continuously developed since about 50 years ago [1]. This process presents important advantages for complex ceramic parts production, in comparison with traditional high-pressure injection molding. These includes the lower cost of dies and less die wear, thanks to the use of lower molding pressures and temperatures, and a less expensive...
injection equipment [1,2,3]. However, the use of lower injection pressures is only made possible by the use of a relatively large proportion of low viscosity binders in the injection mixture. In consequence of that, LPIM usage has been limited mainly by the difficulties associated to the process of binder removal. Moreover, even nowadays several aspects important for LPIM are only poorly understood [4].

The difficulties encountered during binder removal are greater when the parts have large cross-section, and are made with submicrometer-sized ceramic powder [2,3].

In LPIM, low viscosity paraffins and waxes are usually employed as binders [1]. Paraffin wax is commonly employed as the main binder component for LPIM because of its wide range of molecular size and low viscosity at moderate temperatures [5]. Other waxes that can be added to the binder mixture include polyethylene wax, carnauba wax, bee wax, etc. Carnauba is a brittle and hard vegetable wax and acts as both a lubricant and an internal mold releaser [5,6].

Other components are commonly added to the binder mixture to promote modification of the ceramic powder surface [2]. A mixture with very low viscosity can be obtained with the addition of surfactants, which help to stabilize the dispersion of fine ceramic particles in the binder [1]. These surfactants (in our case, stearic and oleic acids) react with the ceramic oxide surface (more specifically with hydroxyl groups) thus enhancing the wetting of the binder on the particle surface [1,2].

Usually a polymer is added in minor proportion to the binder mixture in order to confers a greater rigidity to the green ceramics, especially during debinding [4]. Because polymers only begin to degrade at higher temperatures, they can help to hinder the plastic flow of ceramic parts during thermal debinding [7]. On the other
hand, polymers with high molecular weight are difficult to remove from large ceramic parts, mainly those made with fine powder, and render the debinding process very slow.

The ceramic powder plus binder mixture used for LPIM should be optimized for high fluidity while keeping the ceramic content as high as possible. For the successful production of ceramic parts by LPIM, it is thus necessary to develop an adequate binder formulation. This is a difficult task, especially for fine particles, as it involves a large number of variables related to physical and chemical properties of binder components, the interactions between them and also with the powder surface.

In this paper we present a wax-based formulation for LPIM, suitable for the production of fine particle size and large cross-section ceramic parts. We present also procedure developed us for the injection molding, debinding and sintering of those parts. The binder used in the formulation described here includes only low molecular weight waxes. The quality of the sintered ceramic parts obtained according to the process described in this letter was assessed by density analysis, as well as by tests under working conditions.

2. Experimental

The ceramic powder used was submicrometer-sized alumina (Al₂O₃) A-1000SG (Alcoa), with specific surface area of 9 m²/g, 99.9% purity and particle size of about 0.4 µm. The machine used for LPIM was a Pelsman MIGL-33. The mixture was prepared directly in the LPIM machine, and consisted of 86 wt% of alumina (dried for 3 hours at 150°C before used) and 14 wt% of binder (~45 vol%). The major binder component was paraffin wax, which represents 75 wt% of the binder. Others
components were added in minor proportion to the binder mixture, including polyethylene wax, carnauba wax, stearic and oleic acids (see Table 1).

Insert Table 1

These components were mixed for 20 hours at 90°C before injection. The powder plus binder mixture was injected into brass molds at 90°C and 400 kPa of pressure for 12 s. A large portion of the ceramic parts molded with this mixture were thread-guides for textile industry. The ceramic parts injected were subjected to debinding immersed into alumina powder (wicking). The powder bed consisted of the same alumina employed to make the ceramic parts, providing a physical support for the ceramic bodies and preventing major distortions during debinding [2,3].

3. Results and Discussion

Figure 1 illustrates some of the furnace ramps and dwells times used for wick debinding. After completion of the furnace programmation, the parts were cooled to room temperature over a time defined by the furnace thermal inertia.

Insert Fig. 1

For the debinding of thin ceramic parts, made with submicrometer-sized powder, with cross-section up to 7 mm (see Fig. 2), a very fast ramp with wicking showed to be adequate. On the other hand, for large section ceramic parts with more than 10 mm of cross-section (Fig. 3a), the fast ramp up to 250°C resulted unfeasible.
Indeed, at this temperature, a hard-skin is formed on the ceramic surface of parts fired under oxidative conditions [2]. This hard-skin makes the debinding process difficult and should be avoided. Accordingly, for these large section ceramic parts it was necessary to employ a long dwell time at 170°C [2,3]. Very large pieces, with cross-section greater than 20 mm (Fig. 3b), needed a very long dwell at 170°C, in order to obtain ceramic bodies free of defects.

*Insert Fig. 2*

For many reasons, the production of ceramic bodies with large cross-section, represents a technological challenge, especially when these ceramics are made with submicrometer-sized powder. The use of the binder formulation described in this work, together with the optimization of the furnace programmation for the debinding, permitted us to obtain large cylinders of submicrometer-sized alumina free of defects, as that showed in Fig. 3b.

*Insert Fig. 3*

The density of the ceramic parts sintered at 1600°C/2 hours, as measured by the Archimedes method, varies from 96% to 98.5% of the theoretical density of alumina. After sintering, the particle size increases by more than one order of magnitude, forming a ceramic body of high density and excellent mechanical properties, with relatively low distortion in shape as illustrated in micrography of the Fig. 4.
4. Conclusion

The process described in this work allowed the production of high quality ceramic parts, with very good reproducibility. The ceramic parts produced in this work had very good performance in tests conducted under working conditions. The overall process resulted robust, reliable and adequate for the production of complex shape ceramic parts in the range of 100 - 10000 units, being well suited for the production of ceramic components used in the textile industry.

Acknowledgments

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References


Table Caption

Table 1

Binder formulation. Components and its weight percentage in the binder mixture employed in this work.
<table>
<thead>
<tr>
<th>Constituent</th>
<th>Source</th>
<th>wt %</th>
<th>melting point (°C)</th>
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<tbody>
<tr>
<td>paraffin wax</td>
<td>120/125-3 Petrobras-BR</td>
<td>75</td>
<td>49-52</td>
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<td>polyethylene wax</td>
<td>Ipiranga - BR</td>
<td>10</td>
<td>80-90</td>
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<tr>
<td>carnauba wax</td>
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<td>80-87</td>
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<tr>
<td>oleic acid</td>
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<tr>
<td>stearic acid</td>
<td>Synth - BR</td>
<td>2</td>
<td>75</td>
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</tbody>
</table>

J. E. ZORZI, C.A. PEROTTONI and J.A.H. da JORNADA, Table 1.
**Figure Captions**

Fig. 1. Temperature-time diagrams for binder removal from (●) thin ceramic parts, (▲) large ceramic parts and (∇) very large ceramic parts.

Fig. 2. Sintered thread-guides with thin cross-section.

Fig. 3. Sintered alumina parts with (a) large and (b) very large cross-section.

Fig. 4. SEM micrography of a fracture section of an alumina ceramic part sintered at 1600°C.
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J. E. ZORZI, C.A. PEROTTONI and J.A.H. da JORNADA, Fig. 3.
J. E. ZORZI, C.A. PEROTTONI and J.A.H. da JORNADA, Fig. 4.