Abstract

The paper presents a comparison of properties of advanced ceramics formed by uniaxial hydraulic pressing and pressure casting and gives some indication for proper selection of the best shaping technique regarding product quality, throughput capacity, and economic aspects. Adequate firing technologies are also discussed. This helps to design new plants as well as to optimize existing production facilities.

Introduction

For the production of advanced ceramics, many shaping technologies are used, e.g. cold and hot isostatic pressing, injection moulding, slip casting etc. [1-5]. Uniaxial hydraulic pressing is regarded traditionally as a possible shaping technology only for few special applications, mainly limited to smaller product sizes with additional restrictions regarding the complexity of the specimen and their aspect ratio [1, 6-8]. However, during the last decade hydraulic pressing technology was improved in many aspects, like sophisticated hydraulic and electric control systems, vacuum technology and others [9-12]. These improvements open up new fields for application of uniaxial hydraulic pressing, also for the production of advanced oxide and non-oxide ceramics and composites [13]. Also slip pressure casting technology, originally mainly used for tableware and sanitaryware ceramics, has been improved to become a suitable production technology for technical ceramics, even for larger dimensions and especially for complex shapes [14].

In order to demonstrate the possibilities of hydraulic pressing and pressure casting, a series of similar sample specimens have been produced from the same raw material using both technologies.

Experimental

As raw material a reactive alumina powder (CT3000, source: Almatis, Frankfurt) with 99.7 % Al₂O₃ was used which was prepared for the tests at the LAEIS test center ALPHA CERAMICS in Aachen. For the hydraulic pressing tests the powder was spray dried and for the pressure casting tests a suitable slip has been developed.

The binder system for pressing powder with the main components acrylate and wax has been developed for other applications and was not optimized for this special task. The binder content was in the range of 2 mass %. The bulk density of the spray dried powder was about 1200 g/dm³.

The slip for pressure casting was prepared with a solid content of 70 mass-% (density about 2100 g/dm³). A complex mixture of organic additives had to be used to achieve a stable slip, but it was possible to get a useful slip without any temporary binder, therefore the total content of organics was less than in the pressing powder (see table 1).

<table>
<thead>
<tr>
<th></th>
<th>spray dried powder for hydraulic pressing tests</th>
<th>slip for pressure casting tests</th>
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</thead>
<tbody>
<tr>
<td>binder main components</td>
<td>acrylate; wax; liquifier</td>
<td>antifoaming agent; deflocculant; liquifier; wetting agent; plasticizer</td>
</tr>
<tr>
<td>content of organics (mass-%)</td>
<td>2 %</td>
<td>1 %</td>
</tr>
<tr>
<td>solvent content (mass-%)</td>
<td>0 % (dry powder)</td>
<td>30 % (water)</td>
</tr>
<tr>
<td>density (g/dm³)</td>
<td>1200</td>
<td>2100</td>
</tr>
</tbody>
</table>

Table 1: Binder and additive systems for pressing and casting tests

Uniaxial Hydraulic Pressing

The spray dried powder was dry pressed on a LAEIS press type ALPHA 800 (figure 1) to round discs in a single cavity steel mould with a diameter of 193 mm.

The press was equipped with a vacuum system. No specific measures were taken with respects to surface treatment (polishing etc.) of the dies. Discs with various thicknesses were pressed under various pressing forces from 5 to 15 kN/cm² (50-150 MPa), using the same mould with various filling heights. The mould was filled volumetrically. Densification took place after evacuation of the mould to a residual atmospheric pressure < 50 mbar. The total pressing cycle time including evacuation was about 20 s. After pressing, the
discs were automatically ejected and manually removed from the mould.

Fig. 1: Hydraulic press type ALPHA 800

**Slip Pressure Casting**

The slip pressure casting tests were made on a SAMA pressure casting machine Type PCM 100 (closing force: 1000 kN).

The slip was stirred and pumped into a two-partite macro porous resin mould (material: SAMA-por Standard; $d_{50} = 25\mu m$) with a central sprue. The mould diameter was the same than for the hydraulic pressing tests (193 mm) with a mould height (defining the thickness of the green sample) of 8 mm. A feed pressure between 1 and 5 bar was applied. After a filling time of about 15 s a slip pressure of 15 to 30 bar (1.5-3 MPa) was built up in about 20 s and the final pressure was maintained for 60-80 s. After opening of the mould the plate was removed manually. The residual moisture of the samples was about 16.4 %.

Fig. 2: High pressure casting machine type PCM 100

**Drying and Firing**

For the pressed samples no drying was necessary. The pressure cast pieces were pre-dried at 40 °C over night and then dried to constant weight at 100 °C. The samples were fired under normal atmosphere conditions in an electrically heated kiln according to the firing curve shown in figure 3. A holding time of 3 h at 120 °C was inserted in order to finish the drying process for the pressure cast parts carefully. The maximum temperature of 1550 °C was held for 8 h. The firing cycle has not yet been optimized for fast firing.

Fig. 3: Firing curve for Al2O3 plates

**Results and Discussion**

The tests showed that with both shaping technologies good samples could be prepared. Table 2 gives a survey about some characteristic data of the green samples obtained by hydraulic pressing and by pressure casting. Each line for pressed samples in the table represents a collective of up to 15 identical samples with up to 9 measuring spots each. For cast samples the line with a slip pressure of 2 MPa represents the same number of samples, whereas for the other pressures only 2 samples were available, which is not enough for a statistical evaluation. Therefore, in these cases no standard deviation values could be calculated. The pressure cast samples were characterized after drying, whereas the pressed samples were taken just as they came out of the press. With the hydraulic press, samples of various thicknesses between < 1 mm and 20 mm could be produced (fig 4), but in this comparison only plates of about 8 mm thickness have been included, prepared in both machines with different pressures.

Fig. 4: Plates of various thickness obtained by hydraulic pressing (after firing)
Both systems can be operated at very low binder content with surprisingly high green strength of the plates (see fig. 5). The moisture content of the pressure cast plates causes a linear shrinkage in the diameter of the plates during the drying process. This shrinkage increases with decreasing slip pressure from about 1.3 % (1.5 MPa) to about 0.9 % (3 MPa). In case of hydraulic pressing, there is a slight springback effect after ejection, which leads to an increase of the plate diameter in the range of 0.1 to 0.3 %, increasing with increasing thickness of the plate (0.17 % at 8 mm thickness).

The average thickness of the green cast plates shows practically no variation with different slip pressures, due to the fixed mould geometry. With hydraulic pressing, however, an increasing pressure leads to a reduction in pressed sample thickness. Both variations result in higher green density with increasing pressure.

The thickness variation of the green plates were quite low, from plate to plate (when shaped under identical conditions) as well as at different spots of one plate. In case of pressed plates, the thickness variations were mainly caused by the manual filling of the mould, the variations of the cast plates mainly came from the central sprue. Both systems still can be optimized depending on the geometry of the specimen to be produced.

The green density of the pressed plates was somewhat higher than that of cast plates, due to the higher residual porosity after drying out of the water. As to be expected, the green density could be increased with increasing specific pressure. Interestingly, doubling of the pressure led to an increase of 0.04 g/cm³ in green density in both systems. The green density variations of the plates again showed very satisfying results. With standard deviations of less than 0.01 g/cm³ (pressed plates) and 0.016 g/cm³ (cast plates) both technologies can be compared easily with other “advanced” shaping technologies. The somewhat higher density fluctuation of the cast plates again are mainly caused by the difference between the central sprue area and the peripheral areas of the discs. The density variations of the pressed plates can be reduced further by increasing the homogeneity of the plate thickness with improved filling technology.

The linear shrinkage in diameter during the firing process was about 17.3 % for the plates made with both technologies. The fired densities were very similar for both systems (about 3.85 to 3.88 g/cm³). It has to be mentioned, that it was not the purpose of these investigations to optimize the product density. The fired densities were practically independent of the green densities.

The smoothness of surface as well as the sharpness of edges are clearly better at the pressed plates, due to the porosity of the pressure casting mould. The pressed plates showed a surface roughness Rₐ of < 0.6 µm after firing without any machining, which is a similar quality as can be obtained by tape casting and still could be improved by polishing of the mould dies, if required.

<table>
<thead>
<tr>
<th>shaping technique</th>
<th>specific pressure</th>
<th>green plate thickness</th>
<th>green density</th>
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<tr>
<td></td>
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<td>average</td>
<td>min</td>
</tr>
<tr>
<td>mm</td>
<td>MPa</td>
<td>mm</td>
<td>mm</td>
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<tr>
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<tr>
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<tr>
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<tr>
<td>casting</td>
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</tr>
<tr>
<td>casting</td>
<td>3</td>
<td>8.11</td>
<td>8.06</td>
</tr>
</tbody>
</table>

n.d. = not determined (number of samples not enough for statistical evaluation)

Table 2: Comparison of green sample properties
Comparison of uniaxial hydraulic pressing and slip pressure casting technology

Uniaxial hydraulic pressing and slip pressure casting are mainly used up to now for the production of "traditional" ceramic products like refractories, tiles (pressing) or tableware and sanitaryware (pressure casting). The results of these investigations show, that both technologies are very useful also for shaping of advanced ceramics. The selection of the most adequate system for a given task has to consider various aspects regarding technological as well as economic factors:

- Machine and mould investment costs are higher for hydraulic pressing technology, but also the production capacity (e.g. in terms of m² production per hour) is much higher.
- Pressure casting machines have a typical annual production capacity of 5000 to 50000 pieces, which can be produced in an adequate hydraulic press in a couple of hours or in a couple of days. Typical cycle times are 60 - 150 s for pressure casting machines, 5-20 s for hydraulic presses.
- Hydraulic pressing requires dry materials with a suitable flowability for an even mould filling, preferably spray dried powder; for pressure casting a slip has to be prepared.
- Both systems can produce near net shape articles, but due to the mould material hydraulic pressing can provide better surface quality and sharpness of edges.
- Pressure casting machines mostly work with single moulds, while hydraulic presses -depending on product dimensions and size of the press- often are using multi-cavity moulds.
- Two-partite moulds with one closing direction are standard for pressure casting machines, but also 3-, 4- or 5-partite moulds for 2 or 3 closing directions are possible. Hydraulic presses generally have only one closing direction, but also divided moulds can be used for articles with more complex shapes.
- Both systems are mainly used for fully dense product geometries like plates, blocks, cylinders etc. Hydraulic pressing allows also shaping of hollow cylinders with simple geometry, but no undercut is possible. In contrary, pressure casting can realize much more complex designs like curved hollow tubes and pipes.
- Pressure casting is more restricted in product thickness and wall thickness of hollow articles (typically 3 - 15 mm). Each variation in thickness requires a separate mould. Hydraulic pressing allows the variation of product thickness in a much wider range (< 1 mm up to 50 mm or more) without changing the mould (only adjustment of filling height necessary).
- Hydraulic presses are available in a big range of sizes (pressing force, filling height and useful pressing area). The available selection of pressure casting machines is smaller.

Firing technology

After shaping, the firing process is another key technology which determines the quality of the final product. Depending on the required temperature profile, required throughput capacity and other factors several solutions for heat treatment of these types of advanced ceramics are available:

- Pusher-type kiln: Continuous operation for medium-size capacities, heating possible with gas and/or electricity. Design for heavy load on the pusher slabs, product can be arranged in individually separated layers or in stacks. Slabs can be arranged up to three per width, which leads to a maximum useful width of about 1.3 m. Length of pusher type kilns is up to 30 m, with a maximum temperature up to 1600 °C. If advantageous, the kiln can be subdivided in a binder burnout zone and a sintering zone for special applications related to product requirements regarding kind of heating and atmospheric conditions. Also a gas-tight design is possible if further development shows the necessity.
- Roller kiln: Designed for continuous operation for low, medium and high throughput capacities. Heating is possible with gas and/or electricity. Fast cycles are possible due to low mass flow which lead to lower energy consumption compared to the pusher-type kiln application. Again, a gas-tight design is possible if necessary. Like the pusher-type kiln, the roller kiln can be subdivided in a binder burnout zone and a sintering zone. Width up to 1.5 m, depending on maximum temperature even wider, length from 6 m to 60 m depending on product requirements. Maximum temperature up to 1600 °C.
- Top-hat-kiln: Batch operation for different firing conditions due to different product requirements concerning temperature profile, atmosphere and cycle time, based on the fact that different shapes, product sizes and material properties require a flexible firing program. The products can be arranged in individual separated layers or in stacks. Top hat kilns for technical ceramic applications are designed gas-tight and electrically heated, with useful kiln volumes from 0.13 m³ up to 1.74 m³, which means from two stacks up to sixteen stacks in steps of two (2,4,6,...,16 stacks). The maximum temperature is 1600 °C.
- Shuttle kiln: Simpler design as top-hat kiln, however with the possibility of gas and electric heating. A shuttle kiln is the preferable option, if the product does not need any further special treatment, i.e. the temperature profile indicated is suitable and the binder burnout is sufficient. Useful kiln volume from 0.5 m³ up to 30 m³ depending on product and capacity requirements. Maximum temperature up to 1800 °C.

All described kiln types can be equipped with a thermal post combustion if required.
Summary

The results of the investigations show that with uniaxial hydraulic pressing as well as with slip pressure casting there are two useful technologies available for shaping of advanced ceramics. In combination with an adequate firing technology solutions can be offered which in many cases will provide optimal possibilities for an economical production of advanced ceramic products fulfilling utmost quality requirements.

References

3. I. Cremer: Metal injection moulding is mature. cfi/Ber.DKG 83 (2006), E22-23

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