Comparison of Different Shaping Technologies for Advanced Ceramics Production

Abstract
Many shaping technologies are considered for the production of advanced ceramics, like cold and hot isostatic pressing, uniaxial pressing, tape casting, etc. This paper presents a comparison of properties of advanced ceramics (using alumina as material for the comparison tests) formed by pressure casting, uniaxial hydraulic pressing and tape casting and gives some indication for proper selection of the most suitable shaping technique regarding requirements concerning dimensions and shape of products, product quality, throughput capacity, and economic aspects. Adequate firing technologies are also discussed. The results can help to design new plants as well as to optimize existing production facilities.

1 Introduction
For manufacturing of advanced ceramics or technical ceramics many different shaping technologies can be used (in the context of this paper "advanced" or "technical" ceramics will be referred to as ceramics produced mainly from well defined raw materials, often synthetic powders, rather than "classical" ceramics mainly made from natural raw materials). Such shaping technologies can be pressing (e.g. cold and hot isostatic pressing, uniaxial pressing), casting (pressureless casting, pressure casting, tape casting), extrusion, injection moulding, up to high sophisticated processes like rapid prototyping or manufacturing, freeze gelation, printing technologies and many others [1 – 11]. Some of the technologies are well introduced and available also for large scale production, others are under development or generally suitable only for laboratory scale or prototype manufacturing. All of them have specific advantages and restrictions, making them more or less useful for a given task. When new advanced ceramics products are developed, the activities very often are focused on material and recipe optimization and thermal processes, whereas for shaping any available laboratory equipment is used, without searching for better alternatives and especially without respect to a later industrial scale production. TEAM by Sacmi, an alliance of several companies belonging to the Sacmi group (the world’s biggest supplier of machinery for ceramics production) and active in the field of advanced ceramics, offers a wide range of various shaping machinery and also provides outstanding R&D facilities. In order to provide a sound background for our customers to choose the optimum forming technology for new production lines, investment in production capacity increase and other tasks, the capabilities of different shaping technologies offered by TEAM have been investigated, namely slip pressure casting, uniaxial hydraulic pressing and tape casting.

2 Experimental Details
2.1 Selection of Comparison Standard Material / Material Preparation
As standard raw material for these investigations, a reactive alumina powder type CT 3000 SG (source: TEAM by Sacmi, an alliance of several companies belonging to the Sacmi group) has been selected. The properties of this powder are presented in Fig. 1. The particle size distribution of the powder is shown in Fig. 1. The particle size distribution of the powder is shown in Fig. 1. The spray dried granules are shown in Fig. 2. The spray dried granules are shown in Fig. 2. The spray dried granules are shown in Fig. 2.

Tab. 1 Binder and additive systems for pressing and casting tests

<table>
<thead>
<tr>
<th></th>
<th>slip for pressure casting</th>
<th>spray dried powder for pressing</th>
<th>slip for tape casting</th>
</tr>
</thead>
<tbody>
<tr>
<td>main organic components</td>
<td>antifoaming agent; deflocculant; liqurier; wetting agent; plastitizer</td>
<td>acrylate; wax; liquifier</td>
<td>acrylate based polymer suspension; liquifier;</td>
</tr>
<tr>
<td>content of organics (mass-%)</td>
<td>≤ 1 %</td>
<td>≤ 2.5 %</td>
<td>18.7 % (including solvent)</td>
</tr>
<tr>
<td>solvent content (mass-%)</td>
<td>30 %</td>
<td>0 %</td>
<td>30 %</td>
</tr>
<tr>
<td>density (g/dm³)</td>
<td>2100</td>
<td>1200</td>
<td>1650</td>
</tr>
</tbody>
</table>

Fig. 1 Particle size distribution of CT 3000 SG powder and spray dried granules
Fig. 2 Spray dried alumina as used for pressing tests
Almatis, Frankfurt [12]) with 99,7 % \( \text{Al}_2\text{O}_3 \) was used. The CT 3000 was prepared as spray dried powder in an industrial scale spray dryer at our technical center Alpha Ceramics in Aachen. The particle size distribution of the CT 3000 SG powder and of the spray dried granules are shown in Fig. 1 and 2. The slips for pressure casting and tape casting were prepared in laboratory scale. A summary of binder and additive contents and properties of the materials prepared for the shaping tests is given in Tab. 1.

The binder system for the spray dried pressing powder with the main components acrylic 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). In order to get a good homogeneity and to avoid any agglomerates, the slip was stirred with a directed jet turbine mixer for two hours. 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.

For the tape casting slip a much higher organic content was necessary. Due to this high organic content, a ball mill was used for homogenization of the slip. Additionally to the acrylate based polymer which was used as binder, also a high content of liquefier had to be added to get a good castable slip. The final inorganic solids content was about 51 %, with a slip density of about 1650 g/dm³.

2.2 Shaping Machines

For the comparison tests of slip pressure casting and hydraulic pressing, production scale machines have been used which are available in our labs for process development and optimization. The tests for tape casting have been made to a much smaller extend on a pilot scale tape caster. They were made without detailed process optimization work, just to show the preparation aspects of thin ceramic plates, compared to hydraulic pressing technology.

Slip pressure casting

Slip pressure casting represents a successful combination of conventional casting and pressurised filtration, where the filter cake is formed to the respective article. This technology was investigated already 100 years ago, but could not be used in production lines due to the lack of suitable mould materials with sufficient strength and porosity, to provide high numbers of casting cycles at reasonable costs. Now fully porous resin moulds have been developed featuring the following characteristics:

- Application of slip pressures up to 40 bar without additional reinforcement
- Minimum surface wear, therefore exact and reproducible article contours
- Life span of up to several 10 000 moulding cycles
- Homogeneous pore structure allowing a reliable demoulding and effective backflushing
- Adjusted grade of flexibility serving for safe sealing and preventing leakage
- Simple production of working moulds at production site granting the highest production flexibility

At first, pressure casting was established in tableware production for shaping of non-rotation-symmetric articles in two-part working moulds. Then sanitaryware production followed with the task to control the shaping of very large and quite complex articles, such as siphon-jet toilet bowls needing up to four-part working moulds even with further inserted loose parts. Today also technical ceramic products are manufactured in multi-partite as well as multi-cavity moulds. Not only the shaping process, but also the downstream processes like demoulding, automatic and highly precise fettling and finishing, optimized drying and fully automatic transport and logistics are at a very high level of sophistication. For the slip pressure tests a SAMA PCM 100 was used (Fig. 3). Some general technical data of this machine are given in Tab. 2. The slip was stirred and pumped into a two-partite macro porous resin mould (material: SAMA-por Standard; \( d_{50} = 25 \) µm) with a central sprue, clamped to the closing unit. The mould diameter was 193 mm, with a mould height (defining the thickness of the green sample before drying) 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 40 bar (1,5 – 4 MPa) was built up in about 20 s and the final pressure was maintained for 60 – 80 s. During the shaping process the mould surface acts as a diffusion barrier, building the so-called “casting skin” as a spontaneous reaction when the slip is applied. This skin serves as self-filtration layer, on which the further filter cake forms up to the pre-determined body thickness. After opening of the mould, the filter cake is formed to the article. The body thickness is determined by the opening of the mould and releasing the plate from the mould by applying compressed air from the backside.
The plate was removed manually (Fig. 4). The residual moisture of the samples was about 16.4%.

Uniaxial hydraulic pressing

Uniaxial hydraulic presses of the types ALPHA and OMEGA are generally used for the production of ceramic tiles, slabs or plates, characterized by large pressing area and limited height. Applications include technical ceramic products like sputtering targets, ceramic armour plates, wear protection plates, substrates for electronic devices and many more. Such presses are available with pressing forces in the range from 600 t up to 4200 t and a useful pressing area up to 1600 x 1000 mm². Generally they have a maximum filling height of 60 mm, but some of them are available with a filling height of up to 120 mm. Products of the same shape with different thicknesses can be made in the same mould, simply by changing the filling height. With active mould elements also more complex shapes can be produced.

The hydraulic pressing tests described here were done using a LAEIS press ALPHA 1500-120 (Fig. 5). Some general technical data of this machine are given in Tab. 2. The powder was dry pressed to round discs in a single cavity steel mould. The mould diameter was the same than for the slip pressure casting tests (193 mm). No specific measures were taken with respects to surface treatment (polishing etc.) of the dies. Discs with various thicknesses (nominal green thickness of 10 mm, 5 mm and 2.5 mm) were pressed using the same mould with various filling heights, with specific pressures of 50, 100 and 150 MPa. Such pressures are typical for uniaxial hydraulic pressing of technical ceramics, but for special applications specific pressures up to approx. 400 MPa have also been applied. The mould was volumetrically filled by hand. The press was equipped with a vacuum system, which allows the mould cavity to be evacuated before the densification starts. Pressing under vacuum provides many advantages, e.g. better densification, no formation of layers caused by entrapped air, and shorter pressing cycles, since multiple de-aeration strokes can be avoided [13]. Vacuum pressing technology is especially recommended for press bodies with a high densification factor, for extremely fine press bodies (not granulated, highly dispersed powders) or for product geometries with large volume or with high aspect ratios. In the tests reported here, the mould was evacuated to a residual atmospheric pressure < 50 mbar (min. 90 % vacuum).

After pressing, the discs were ejected and manually removed from the mould. Even very thin plates (green thickness down to about 1 mm) showed a good strength and could be handled without damage (Fig. 6). The total pressing cycle time including evacuation was about 20 s.

Tape casting

Compared to other shaping methods applied in ceramic production, tape casting is a relatively young shaping method, first introduced successfully for the production of dielectric ceramic tapes. Today tape casting is very common for the preparation of thin ceramic tapes, e.g. for ceramic substrates in electronic applications, piezo actuators, micro filters, components for fuel cell stacks and many others. The technology is based on a very simple principle: a plane surface (polished steel belt or polymer carrier tape) is driven with constant speed under the doctor blade, which is exactly adjustable with regard to height. Thus the ceramic slip is distributed in a homogeneous coat layer.

Precondition is a homogeneously prepared slip, without bubbles and free-flowing, which will dry to a useable, faultless tape. Two kinds of slip can be used:

- Slips with organic solvent offer a numerous variety of combinations of additives (solvent, binder, plasticizer, liquefier, wetting agent, etc.) to reach a maximum density and a sufficient stability of the slip. However, due to regulations regarding toxicity, explosion risk and environmental pollution, additional measures have to be taken which can be quite cost-intensive.

<table>
<thead>
<tr>
<th>Tab. 4</th>
<th>SAMA FGT 250 tape casting machine; technical data</th>
</tr>
</thead>
<tbody>
<tr>
<td>useful casting width</td>
<td>250 mm</td>
</tr>
<tr>
<td>casting track length</td>
<td>3 200 mm</td>
</tr>
<tr>
<td>casting speed</td>
<td>20 - 200 mm/min</td>
</tr>
<tr>
<td>casting height</td>
<td>0.05 - 4 mm</td>
</tr>
<tr>
<td>drying system</td>
<td>radiation (IR panel)</td>
</tr>
</tbody>
</table>
• Aqueous systems provide a considerably lower range of possibilities to select and combine the different additives and therefore cause a much higher effort in development. Also the drying period is longer and requires a bigger variety in adjusting the drying conditions, but the effort regarding environmental and safety measures is considerably less. Very thin ceramic tapes (green thicknesses as low as 0.05 mm, up to about 4 – 5 mm as a maximum) with a very large surface area can be produced. The higher the thickness, the more difficult is the drying procedure, and the casting speed has to be adjusted accordingly. Tape casters allow to adjust the width of the tape very easily. According to the working principle, the side areas of the tape do not show the same properties as the main tape area and need to be cut off. A high organic content is necessary, which allows a very flexible tape to be produced, but also requires a separate debinding process before firing of the tapes. The final shaping of the product is done after drying by cutting, milling, boring, punching and/or lamination, allowing a broad range of different products to be manufactured. A big advantage is that such different geometries are achieved by almost standardized machines. For the tape casting tests the SAMA tape casting unit FGT 250 (Fig. 7) was utilised. The technical data of this laboratory equipment are summarized in Tab. 4. The base of the casting station is a precisely machined and polished hard-stone plate, on which the casting unit including doctor blade is fixed. The homogeneously prepared and de-aired slip was added through a slot gate onto the casting surface (polyester foil). The requested amount was adjusted by the gate opening. Subsequently the slip was spread uniformly onto the carrier foil by means of the exact height adjustment of the doctor blade. During the trials the absolute wet tape thickness was varied from 0.08 to 3.0 mm. After casting, the ceramic tape was passed through the drying zone, which was equipped with an infrared panel in order to accelerate the drying process. In case the casting speed was too high or the tape too thick, the casting process was stopped. After completion of the drying process the ceramic tape was separated from its carrier foil. The dried green tape showed a good flexibility (Fig. 8) and could be processed easily (cutting and firing) for further characterization.

2.3 Thermal Treatment
After shaping, the firing process is another key technology which determines the quality of the final product. For industrial high temperature applications in the ceramic industry continuous kilns are used in the design as tunnel kilns with cars conveyance, as roller hearth type or as pusher type. Intermittent high temperature kilns are used in the ceramic industry whenever high flexibility is required.

Depending on the necessary temperature profile, the required throughput capacity and other factors, Riedhammer offers various solutions of both continuous and intermittent high temperature kilns for heat treatment of advanced ceramics, e.g.:
• Tunnel kiln: gas heated kilns with car conveyance with continuous operation for high throughput capacities, with different cross section dimensions depending on the capacity requirement. Kiln length between 24 and 60 m, maximum temperature up to 1800 °C.
• Roller kiln: gas and/or electrically heated kilns, designed for continuous operation for low, medium and high throughput capacities. 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. Electrically heated with gas-tight design for technical ceramic applications. Useful volumes from 0.13 m³ up to...
Selective debinding measures as thermal post combustion for all flue gases extraction via the kiln car. Swinging, sliding or lifting doors as general options are available like:

- Shuttle kiln: simpler design as top-hat kiln, but with the possibility of gas and electric heating. Useful volumes from 0.5 m³ up to 30 m³.
- Maximum temperature up to 1800 °C. Fig. 9 shows a typical intermittent high temperature shuttle kiln used in the production of advanced alumina products. According to customer’s request several options are available like:
  - Swinging, sliding or lifting doors as closure of the chamber and shuttle kilns
  - Flue gases extraction via the kiln car soles and the kiln rear wall and conduction from there via heat exchanger into the open air. Depending on kiln size, flue gases are exhausted by means of a flue gas channel underneath the kiln
  - Thermal post combustion for all above mentioned kiln types
  - Selective debinding measures as low-O₂-technology, depending on binder content and binder composition [14, 15]

For firing of the tests samples in this investigation, a high temperature chamber kiln RIEDHAMMER HTK 150 (Fig. 10) was used, one of the smallest standardized kilns of the delivery program with automatic temperature and atmosphere control. This type of kiln is normally used for high temperature sintering processes, for testing the shrinkage of refractory materials and for testing kiln furniture. A summary of technical data of this kiln is given in Tab. 5. The basic temperature and diffusion air curve used for the tests is shown in fig. [11]. The samples were pre-dried at 120 °C to constant weight. Then a heating-up gradient of 1 K/min has been applied up to the maximum temperature of 1600 °C. Between 200 and 400 °C the heating-up gradient was reduced to 0.65 K/min for an optimal debinding. Diffusion air was used to control the debinding process by cooling down the flame temperature. While heating up after debinding the diffusion air was reduced to a minimum to save fuel and to control the oxygen content in the atmosphere. The soaking time at 1600 °C was 4 h. During the cooling phase again diffusion air was inserted to control the temperature. The resulting total firing cycle time was 66 h. It has to be mentioned that this cycle time was not optimized, but was kept constant in order to get comparable results for the different test samples.

Some samples have been fired also in an electrically heated chamber kiln, using the same firing curve as in the gas fired kiln. The results were quite similar, but the temperature distribution seemed to be less homogeneous because of the lack of convection.

### Tab. 6

<table>
<thead>
<tr>
<th>shaping technique</th>
<th>specific pressure</th>
<th>green plate thickness</th>
<th>green density</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MPa</td>
<td>average mm</td>
<td>min mm</td>
</tr>
<tr>
<td>pressing</td>
<td>50</td>
<td>8.55</td>
<td>8.33</td>
</tr>
<tr>
<td>pressing</td>
<td>100</td>
<td>8.40</td>
<td>8.20</td>
</tr>
<tr>
<td>pressing</td>
<td>150</td>
<td>8.30</td>
<td>8.16</td>
</tr>
<tr>
<td>casting</td>
<td>1.5</td>
<td>8.08</td>
<td>7.99</td>
</tr>
<tr>
<td>casting</td>
<td>2</td>
<td>8.06</td>
<td>7.80</td>
</tr>
<tr>
<td>casting</td>
<td>2.5</td>
<td>8.08</td>
<td>8.30</td>
</tr>
<tr>
<td>casting</td>
<td>3</td>
<td>8.11</td>
<td>8.06</td>
</tr>
</tbody>
</table>

3 Results and Discussion

In the following chapter, some properties of green and fired samples, prepared with the different shaping technologies as described above, will be compared and discussed. The aim of this work was not to optimize special properties of the products for a specific application, but to show general advantages and/or restrictions of the shaping processes.

2.4 Characterization of the Samples

For comparison of green sample properties, all samples were dried at 120 °C to constant weight. They were visually inspected and the thickness was measured at different spots at the outer, middle and central area of the plates (Fig. 12). After firing, the thickness of the samples was measured accordingly. Then the samples were divided in smaller parts and the density of the fragments was determined according to the Archimedes’ principle. Typically, about 2 fragments from the center area, 4 fragments from the middle area and 6 fragments from the outer area were measured for each sample plate. For some samples also the green density and the density distribution was controlled. The green tape cast samples were only visually inspected, thickness and density were measured only at fragments of the fired tape. Only fragments from the inner strip of the tape were investigated, side strips were neglected.

Fig. 12 Sketch of characterization areas of cast and pressed plates—blue = outer area—purple = middle area yellow = central area

| Tab. 6 | Green plate thickness and density | 1.74 m³. Maximum temperature 1600 °C. Shutter kiln: simpler design as top-hat kiln, but with the possibility of gas and electric heating. Useful volumes from 0.5 m³ up to 30 m³. Maximum temperature up to 1800 °C. Fig. 9 shows a typical intermittent high temperature shuttle kiln used in the production of advanced alumina products. According to customer’s request several options are available like:

- Swinging, sliding or lifting doors as closure of the chamber and shuttle kilns
- Flue gases extraction via the kiln car soles and the kiln rear wall and conduction from there via heat exchanger into the open air. Depending on kiln size, flue gases are exhausted by means of a flue gas channel underneath the kiln
- Thermal post combustion for all above mentioned kiln types
- Selective debinding measures as low-O₂-technology, depending on binder content and binder composition [14, 15]

For firing of the tests samples in this investigation, a high temperature chamber kiln RIEDHAMMER HTK 150 (Fig. 10) was used, one of the smallest standardized kilns of the delivery program with automatic temperature and atmosphere control. This type of kiln is normally used for high temperature sintering processes, for testing the shrinkage of refractory materials and for testing kiln furniture. A summary of technical data of this kiln is given in Tab. 5. The basic temperature and diffusion air curve used for the tests is shown in fig. [11]. The samples were pre-dried at 120 °C to constant weight. Then a heating-up gradient of 1 K/min has been applied up to the maximum temperature of 1600 °C. Between 200 and 400 °C the heating-up gradient was reduced to 0.65 K/min for an optimal debinding. Diffusion air was used to control the debinding process by cooling down the flame temperature. While heating up after debinding the diffusion air was reduced to a minimum to save fuel and to control the oxygen content in the atmosphere. The soaking time at 1600 °C was 4 h. During the cooling phase again diffusion air was inserted to control the temperature. The resulting total firing cycle time was 66 h. It has to be mentioned that this cycle time was not optimized, but was kept constant in order to get comparable results for the different test samples.

Some samples have been fired also in an electrically heated chamber kiln, using the same firing curve as in the gas fired kiln. The results were quite similar, but the temperature distribution seemed to be less homogeneous because of the lack of convection.

2.4 Characterization of the Samples

For comparison of green sample properties, all samples were dried at 120 °C to constant weight. They were visually inspected and the thickness was measured at different spots at the outer, middle and central area of the plates (Fig. 12). After firing, the thickness of the samples was measured accordingly. Then the samples were divided in smaller parts and the density of the fragments was determined according to the Archimedes’ principle. Typically, about 2 fragments from the center area, 4 fragments from the middle area and 6 fragments from the outer area were measured for each sample plate. For some samples also the green density and the density distribution was controlled. The green tape cast samples were only visually inspected, thickness and density were measured only at fragments of the fired tape. Only fragments from the inner strip of the tape were investigated, side strips were neglected.

3 Results and Discussion

In the following chapter, some properties of green and fired samples, prepared with the different shaping technologies as described above, will be compared and discussed. The aim of this work was not to optimize special properties of the products for a specific application, but to show general advantages and/or restrictions of the shaping processes.

2.4 Characterization of the Samples

For comparison of green sample properties, all samples were dried at 120 °C to constant weight. They were visually inspected and the thickness was measured at different spots at the outer, middle and central area of the plates (Fig. 12). After firing, the thickness of the samples was measured accordingly. Then the samples were divided in smaller parts and the density of the fragments was determined according to the Archimedes’ principle. Typically, about 2 fragments from the center area, 4 fragments from the middle area and 6 fragments from the outer area were measured for each sample plate. For some samples also the green density and the density distribution was controlled. The green tape cast samples were only visually inspected, thickness and density were measured only at fragments of the fired tape. Only fragments from the inner strip of the tape were investigated, side strips were neglected.

3 Results and Discussion

In the following chapter, some properties of green and fired samples, prepared with the different shaping technologies as described above, will be compared and discussed. The aim of this work was not to optimize special properties of the products for a specific application, but to show general advantages and/or restrictions of the shaping processes.

2.4 Characterization of the Samples

For comparison of green sample properties, all samples were dried at 120 °C to constant weight. They were visually inspected and the thickness was measured at different spots at the outer, middle and central area of the plates (Fig. 12). After firing, the
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.

Thickness and density variations after firing
For the investigation of fired sample properties, slip pressure cast plates have been used which were prepared with slip pressures of 2.0, 3.0, 3.5 and 4.0 MPa and plates pressed to nominal green thicknesses of 2.5, 5.0 and 10.0 mm, all with a specific pressure of 90 MPa. For samples from the tape casting experiments, random fragments of the tape has been used with various thickness. The thickness of the plates has been measured at different spots from the outer, middle and centre area, represented by different colours according to Fig. 12. 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 were prepared (Fig. 6), but in this comparison only plates of about 8 mm green thickness have been included, prepared in both machines with different pressures.

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
drop in thickness from 3.5 to 4.0 MPa can not be explained at the moment, but shows that an increase in pressure does not automatically give better results.

The thickness variations of the pressed plates are in the same range as the pressure cast plates. The absolute values are in the range of 2 mm, 4 mm and 8 mm, respectively, but the right picture in Fig. 13 has been recalculated to the same scale as in the left part for the pressure cast samples. The pressed plates, however, do not show a systematic effect in any direction, the variations are due to fluctuations caused by the manual filling of the mould. The linear firing shrinkage, both in diameter and in thickness, was about 17 % for all plates.

Fig. 14 shows the density variations of the same set of plates. It can be seen, that all pressed plates and the pressure cast plates prepared with 2,0 and 3,0 MPa slip pressure have very similar and consistent densities in the range of 3.92 – 3.93 g/cm³, which is very close to the maximum fired density of 3.96 g/cm³ as specified by the supplier. The density of the slip pressure cast plates made with a pressure of 3.5 and 4.0 MPa have significantly lower densities and especially the plates made with 3.5 MPa also have bigger variations from the outer to the center area. This result confirms again, that at least the conditions which have been applied in these tests do not lead to better results with increasing pressure. Also the pressed plates show no advantage of higher pressure after firing. However, the green strength is higher with increasing pressure (for both technologies) and allows an easier handling of the products before firing.

During the tape casting tests, tapes with various casting thickness from about 0.05 mm up to about 3 mm have been produced. From the center area of the thinner tapes smaller segments have been taken and thickness and density of such segments after drying and firing have been determined. In fig. 15 are the densities sorted with increasing thickness between 0.05 and 0.4 mm, each bar representing an average of several samples of the same thickness. Despite the fact, that density measurement of such thin samples is more difficult and the error of the measured values might be larger than for thicker plates, it is very clear that the density variations are much higher than for the plates and also the absolute value of the density is much lower, in average well below 3.85 g/cm³. There is also no systematic change of the density with increasing thickness.

**Influence of firing technology**

The firing parameters have also been modified during these tests, but their influence is not to be discussed here in detail. Of course heating rates and maximum firing temperature are very important. Plates with the same dimensions as described above could be fired to a density of 3.85 – 3.88 g/cm³ only, when heated to a maximum temperature of 1550 °C. Also attention needs to be paid to the type of kiln and the heating system. In an electrically heated kiln e.g. less homogeneous density distributions were achieved due to a less homogeneous temperature in the kiln volume (not enough convection).

**General aspects of product properties**

Both slip pressure casting and hydraulic pressing can be operated at very low binder contents with surprisingly high green strength, even of rather thin plates with a high aspect ratio. The moisture content of the pressure cast plates causes a linear shrinkage in the diameter of the plates during the drying process, decreasing with increasing slip pressure (about 1,3 % at 1,5 MPa and about 0,9 % at 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. Tape casting requires a much higher organic content in the slip, leading to much higher shrinkage and to lower final densities. The smoothness of the surface as well as the sharpness of the edges are clearly better at the pressed plates, due to the porosity of the pressure casting mold. The pressed plates showed a surface roughness Rₐ of < 0,6 µm after firing without any machining which can still be

---

**Tab. 7**

Pre-selection table for shaping technologies

<table>
<thead>
<tr>
<th>factor</th>
<th>slip pressure casting</th>
<th>hydraulic pressing</th>
<th>tape casting</th>
</tr>
</thead>
<tbody>
<tr>
<td>green density</td>
<td>+</td>
<td>++</td>
<td>--</td>
</tr>
<tr>
<td>fired density</td>
<td>++</td>
<td>++</td>
<td>+</td>
</tr>
<tr>
<td>density distribution</td>
<td>++</td>
<td>++</td>
<td>+</td>
</tr>
<tr>
<td>dimensional accuracy</td>
<td>++</td>
<td>++</td>
<td>-</td>
</tr>
<tr>
<td>contour sharpness</td>
<td>+/+</td>
<td>++</td>
<td>-</td>
</tr>
<tr>
<td>green strength</td>
<td>++</td>
<td>++ (rigid)</td>
<td>++ (flexible)</td>
</tr>
<tr>
<td>surface quality</td>
<td>+/+</td>
<td>++</td>
<td>+</td>
</tr>
<tr>
<td>reproducibility</td>
<td>++</td>
<td>++</td>
<td>+</td>
</tr>
<tr>
<td>material requirements</td>
<td>slip</td>
<td>powder</td>
<td>slip (water / solvent)</td>
</tr>
<tr>
<td>binder content</td>
<td>low</td>
<td>low</td>
<td>high</td>
</tr>
<tr>
<td>drying necessary</td>
<td>yes</td>
<td>no</td>
<td>yes (+debinding)</td>
</tr>
<tr>
<td>large specimen</td>
<td>limited</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>product / wall thickness</td>
<td>limited</td>
<td>flexible</td>
<td>flexible</td>
</tr>
<tr>
<td>geometry complexity</td>
<td>++</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>process scrap</td>
<td>negligible</td>
<td>negligible</td>
<td>yes</td>
</tr>
<tr>
<td>process flexibility</td>
<td>high</td>
<td>high</td>
<td>lower</td>
</tr>
<tr>
<td>mould costs</td>
<td>medium</td>
<td>high</td>
<td>none</td>
</tr>
<tr>
<td>production capacity</td>
<td>medium</td>
<td>high</td>
<td>medium</td>
</tr>
<tr>
<td>range of available machines</td>
<td>lower</td>
<td>higher</td>
<td>lower</td>
</tr>
<tr>
<td>useful for advanced ceramics production</td>
<td>+++</td>
<td>+++</td>
<td>+++</td>
</tr>
</tbody>
</table>

---

**process characteristics**

- **General**
  - slip powder slip (water / solvent)
  - yes  no  yes (+debinding)
  - medium  high  low
  - medium  high  none
  - medium  high  medium
  - lower  higher  lower

**Tab. 7**

- **Product properties**
  - green density
  - fired density
  - density distribution
  - dimensional accuracy
  - contour sharpness
  - green strength
  - surface quality
  - reproducibility
  - material requirements
  - binder content
  - drying necessary
  - large specimen
  - product / wall thickness
  - geometry complexity
  - process scrap
  - process flexibility
  - mould costs
  - production capacity
  - range of available machines
  - useful for advanced ceramics production
Hydraulic pressing and slip pressure casting both 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).

Tape casting is a flexible technology for production of thin tapes, but is limited simple shapes which have to be tailored in additional steps.

Pressed products can be fired directly, while slip pressure cast articles need previous drying and cast tapes mostly require an additional debinding step.

Firing to full density is easier for pressed and pressure cast products than for tapes.

Depending on local conditions and which product requirements are paramount, the right technology can be chosen. The synopsis given in table 7 can help to identify the optimum technology for a given task. Additionally, tests which original material can be done in our technology centers, where different technologies are available and can be investigated even up to a certain production scale.

5 Summary

The results of the reported investigations confirm, that slip pressure casting, uniaxial hydraulic pressing and tape casting are generally very suitable technologies for shaping of advanced ceramics. All of them are available and well proven for industrial scale production. Each of these technologies provides special advantages and restrictions, e.g. regarding possible product geometries, throughput capacities, material preparation etc. When such limitations are taken into consideration, they allow in combination with adequate firing technologies the manufacturing of advanced ceramic products fulfilling utmost quality requirements.

TEAM by Sacmi offers a unique opportunity to evaluate alternative solutions for any given task in technical centers equipped with production scale machinery. Thus, optimal solutions for an economical production are produced in direct cooperation with the customers, taking various technologies into consideration.

Acknowledgements

The authors want to thank Almatis GmbH for supply of the alumina raw material, and the technical staff in the TEAM labs for the experimental and analytical work.

Literature

[12] Almatis GmbH, Frankfurt, Germany: Reactive and calcined aluminas for the ceramic industry; MSDS 387