Hydraulic Pressing of Advanced Ceramics

Abstract

Uniaxial hydraulic pressing is the most common shaping technology for ceramic products. However, when production technologies for new advanced ceramic products with high quality requirements are discussed, in most cases this technology is considered to be only second choice. Other technologies like isostatic pressing, injection moulding, pressure casting, tape casting etc. seem to provide better quality products, even if they are more expensive, have much lower production capacities or are limited in other factors.

The paper describes new possibilities for shaping of advanced ceramic products with a wide variety of dimensions and aspect ratios and high quality requirements. Recent developments in pressing technologies are shown and results of pressing tests for advanced ceramics are presented which demonstrate that highest quality can be achieved using uniaxial hydraulic presses. Finally, a selection of application examples is given, indicating advanced ceramic products already hydraulically pressed in industrial scale.

Introduction

Hydraulic pressing is a well known and widespread technology for shaping of ceramics [1 - 4]. Especially for the production of "classic" ceramics like silicate ceramics (floor and wall tiles, facing elements etc.), for all types of refractories (even if many of them nowadays have to be regarded as "advanced ceramics") and for many types of kiln furniture the uniaxial hydraulic pressing is the mostly used shaping technology. Also technical ceramic products (in this paper "technical ceramic" will be referred to as ceramic produced mainly from synthetic powders, either oxide or non-oxide) are made already using hydraulic pressing. But up to now this has been limited to smaller sizes with additional restrictions regarding the complexity of the specimen and their aspect ratio.

Main advantages of uniaxial hydraulic pressing technology are:

- possibility of „dry“ pressing
- low binder content
- high throughput capacities
- moderate investment costs.

On the other hand, there have been also some limitation factors for uniaxial hydraulic pressing of advanced ceramics reported in the past, namely:

- only simple geometries possible
- limited dimensions of the specimens to be pressed
- comparably high variations in the green density; especially from top to bottom (3...10 % variation are reported)
- limited dimensional accuracy, e.g. thickness variations in the range of ±10 %.

When it comes to set up a production technology for high quality technical ceramic products, thus very often uniaxial hydraulic pressing is not at all discussed as a real alternative for shaping in competition to isostatic pressing (cold and hot), injection moulding, slip casting, pressure casting, extrusion, injection moulding, tape casting etc. [5 - 10].

However, in the last decade many advances have been made in hydraulic pressing technology which allow to overcome the above mentioned limitations and open up completely new fields for advanced ceramics production [11 - 14]. In the following some aspects of improvements made in hydraulic pressing technology are reported, some characteristic data of samples obtained using this advanced technology are given and examples of new applications resulting thereof are shown.

Recent Improvements in Hydraulic Pressing Technology

Quality requirements are increasing constantly for practically all types of shaped ceramic products. Thus it has become necessary to improve also the performance of presses permanently. Læis GmbH, being one of the leading suppliers of high duty hydraulic presses especially for refractories, but also for many other industries, made numerous contributions to define a new level of state-of-the-art pressing technology.
• simple operation, no need for manual setting of slow speed and deceleration distance
• very accurate repetitive movement, maintaining an optimum product quality over the complete production time
• reduction of cycle time.

Internal connections are realized with Profibus as a worldwide standardized system. The bus system allows to replace extensive parallel cabling by one single bus cable and reduces the switch cabinet size by about 30%. Comprehensive diagnosis options of the bus system allow for short commissioning times and improved plant availability. In case of service needs, complete bus modules can be replaced without disconnecting a single line or screw.

Hardware and software, both in modular design, provide for a totally integrated automation with intrinsic communication features. Connections to process host control systems can be provided easily as well as production data storage for quality control and documentation.

Even very special features like an e-mail function from the press to a preselected address or to a mobile phone are possible. The control system can be scaled depending on the requirements of the individual process, according to customers request.

A specially developed, window based application software ("ProVi-Control") allows a product oriented entry of data. Thus, setting of production parameters is possible without knowledge of the internal machine functions, just by entry of product specific parameters. An intelligent control minimizes the number of input parameters. Thanks to windows technology (Fig. 2) the operator interface has a clear process oriented structure, easy to understand. The design of the windows is highly graphic oriented with very little wording and standardized symbols. New pressing programs are established quickly using the integrated copy function, the storage capacity is limited only by the capacity of the hard drive.

The pressing regime controls the end of the pressing stroke either by reaching a predefined "nominal" product thickness or by reaching a nominal final pressure or by a combination of both (whatever comes first). Always superimposed is a "tolerance window" for accepted upper and lower limits for pressure and thickness (Fig. 3). Depending on the defined pressing regime the press control tries to compensate for changes in the material bulk density or compactibility, e.g. by modifying the pressure or adjusting the filling height, as long as this is possible within the given tolerances.

In order to achieve an optimum product quality, the setup allows to adjust many parameters in a wide range, e.g.:
• mould filling box movement
• punch entry speed
• speed ratio upper punch / mould frame
• pressure increase curve
• pressure holding time
• de-aeration strokes
• ejection speed
• and many others.

Vacuum Technology

Pressing of powders or granulated material in dry, semi-wet or wet condition often results in microstructure inhomogeneities, uneven density distribution and/or formation of layers, even when de-aeration strokes are applied. These effects are mainly caused by entrapped air, due to insufficient release of the air before and during compaction.

A well proven solution for this problem is the vacuum pressing technology, where the air inside the material is removed to a certain (selectable) level before the compaction starts. With this technology, additional de-airing cycles and slow pressing speeds can be reduced. A recently designed, new economic vacuum pressing system reduces the volume to be evacuated to a minimum, thus enabling a very short evacuation time of about 2...4 s and reducing the energy consumption of the vacuum pump. A pre-evacuation container additionally shortens the pressing cycle. The evacuation can take place via upper and lower die or...
via upper die only, depending on material characteristics and product dimensions. Resulting advantages of vacuum pressing technology are the avoidance of lamination, a higher green density and an even density distribution of the product (Fig. 4) as well as an improved productivity thanks to the reduced cycle time. Several materials and product dimensions can be realized only with vacuum technology, whereas conventional pressing technology, even with multiple de-aeration strokes, leads to the destruction of the pressed body after release of the pressure (Fig. 5). In special cases it can be advantageous to combine vacuum technology with backstrokes of the die (which in this case are no longer de-aeration strokes).

Vacuum pressing technology is especially recommended for:
• granulates with a high densification factor (i.e. mixtures having a low bulk density before pressing and/or reaching a high green density (low residual porosity) after pressing
• extremely fine granulates, e.g. very fine spray dried material for technical ceramics
• not granulated highly dispersed powders which for conventional pressing technology would have to be granulated
• product geometries with large volume or with high aspect ratios (thin plates, rods etc.), where the air needs to move a long distance to the gap between die and mold frame
• mixtures with higher plasticity, which are pressed with higher content of water or other liquid binders.

It has to be mentioned, that the application of vacuum technology requires special care in design and fabrication of the moulds to be used.

Moulds and Mould Filling Systems

When the quality of pressed ceramic products is discussed, especially with respect to thickness variation or density distribution, it is essential to consider also the mould filling process. The best pressing technology can not overcome major inhomogeneities in the material distribution after mould filling (except when the press body has a sufficient plastic behavior, which is not the case for most technical ceramic products). In order to achieve a good homogeneity of the material distribution in the mould, an adequate material preparation process must be ensured. This process has to provide for minimal variations of the bulk density of the body (both within one mould filling or from filling to filling) and for a good (and constant) flowing behavior.

This pre-requisite being fulfilled, the mould filling step itself has to be adapted according to the individual requirements of each process. Therefore, various modifications of mould filling systems have been developed. Basically, it can be distinguished between volumetric and gravimetric filling systems. Gravimetric systems normally have a high weighing accuracy and result in small variations of the weight of the pressed specimen, but are difficult for automation. Volumetric filling systems on the other hand can easily be automated, but the weight accuracy depends on material characteristics. For most of the applications the accuracy of volumetric filling systems is sufficient, but sometimes gravimetric systems or even special filling systems have to be used. Such special systems can be external filling templates, which are filled gravimetrically or volumetrically and are then transferred to the mould (e.g. vacuum assisted). Other possibilities are filling systems with double layer options. It has to be mentioned, that the higher the required accuracy and complexity of the filling system, the more the necessary cycle time will be extended.

The further downstream production steps, especially the drying and firing process, will of course also contribute to the quality of the final product. However, this is valid for all different types of shaping and therefore these process steps are not looked at here. The following description of sample properties will concentrate on properties of green samples.

Properties of Technical Ceramic Samples Obtained by Uniaxial Hydraulic Pressing Technology

Experimental Procedure

In order to demonstrate the possibilities of state-of-the-art hydraulic pressing technology, a series of specimens have been pressed on a LAEIS press type ALPHA 800 equipped with vacuum technology (Fig. 6). As material for these tests a reactive alumina powder CT3000 with 99.7 % Al₂O₃ (Almatis, D) was used, which was spray dried at the LAEIS test center Alpha Ceramics in Aachen. The binder system with the main components acrylate and wax had been developed for other applications and was not optimized for this special task. The binder content was in the range of 2 mass %. The powder was dry pressed to round discs in a single cavity steel mould, diameter 193 mm. No specific measures were taken with respect to surface treatment (polishing etc.) of the dies. Discs with various thicknesses were pressed under various pressures, using the same mould with various filling heights (Fig. 7).

The mould was filled volumetrically. Densification took place after evacuation of the mould to a residual atmospheric pressure of < 50 mbar. After pressing, the disc was ejected.
applied, however, the plates were destroyed immediately after releasing the pressure (Fig. 9).

Table 1: Linear springback effect, depending on filling height and applied pressure

<table>
<thead>
<tr>
<th>Filling Height (mm)</th>
<th>Specific Pressure (kN/cm²)</th>
<th>Linear Diameter Springback [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>10</td>
<td>0.15</td>
</tr>
<tr>
<td>8</td>
<td>5</td>
<td>0.04</td>
</tr>
<tr>
<td>8</td>
<td>10</td>
<td>0.13</td>
</tr>
<tr>
<td>16</td>
<td>5</td>
<td>0.09</td>
</tr>
<tr>
<td>16</td>
<td>10</td>
<td>0.17</td>
</tr>
<tr>
<td>16</td>
<td>15</td>
<td>0.29</td>
</tr>
<tr>
<td>20</td>
<td>10</td>
<td>0.34</td>
</tr>
<tr>
<td>40</td>
<td>10</td>
<td>0.48</td>
</tr>
</tbody>
</table>

Dimensional Accuracy, Density and Density Distribution

After pressing and ejection from the mould cavity, the plates underwent a springback in diameter (re-expansion of the material), depending on the plate thickness and the applied pressure (Tab. 1). A springback in vertical direction was prevented by ejection under load. The green plates showed a very high dimensional accuracy. Variations in green plate thicknesses are shown in Tab. 2.

The average values and standard deviations given for each set of filling height and densification pressure represent a collective of 4-10 plates pressed under the same conditions and 4 thickness measurements of each plate at different spots. Though the thickness variations are already quite low, they can be reduced further in an optimized automatic process, because the data given in Tab. 2 are obtained with manual filling of the mould with small quantities of powder and without a special adjustment of the pressing die parallelism. Both factors contribute to deviations in thickness, the first one mainly between the plates, the second one within a single plate. Naturally, the percentage of deviation is increasing with decreasing thickness. The densification factors, also given in Tab. 2, can not be discussed in detail, because the different test pressings were performed with different pressing parameter combinations. However, the data of the 3 series with 16 mm filling height (with specific pressures of 5, 10 and 15 kN/cm²) were obtained under constant conditions. They show a good consistency of densification factor and accordant average thickness, but also the standard deviation decreases with increasing pressure. The green density values are also shown in Tab. 2, calculated from the weight and dimensions of each individual plate and given as average value with the corresponding standard deviation for each test pressing series. The absolute density values should not be discussed here, because the aim of these tests was not to obtain maximum density, but to demonstrate reliability and reproducibility of the hydraulic pressing process. Again, the 3 test series with 16 mm filling height can be compared to each other, showing a steady increase in density with increasing pressure. More important, however, is the reproducibility of the green density within each test series, having standard deviations of typically <0.4 %.

In an earlier test series, also thickness and density variations were investigated on similar samples after firing. The results are shown in Tab. 3. In this table, only one plate of each type was investigated. Each line of the table represents values of one plate, where the thickness and density were measured at up to 30 spots each, distributed over the whole area of the plate. Again the results are very good, with fluctuations only in the range of typically <2 % (it has to be noted that the values in Tab. 3 are absolute values, not standard deviation values!). Naturally, the thinnest plate with a fired thickness of <1 mm again has a bigger percentage of thickness fluctuation, but the absolute value is in the same range as the other plates.

Fig. 9: Destroyed plate (pressed without vacuum)

Surface Quality

The plates show a very smooth surface already in the green state. After firing a similar surface quality is obtained as with tape casted sheets.
without any machining of the pressed products. A surface roughness $R_s$ of $<0.6 \mu m$ was measured at one plate (Fig. 10).

Application Examples

A big variety of applications was investigated in detail during the last years and many of them have been brought to industrial application. The following section describes some of these applications.

Sputtering Targets for PVD Technology

For functional coating of TV screens, displays, touchscreens etc. several target materials are used which have to be applied using the sputtering technology (PVD = physical vapour deposition process). Since the substrates are constantly increasing in size, also the sources of the sputtering material (so called “sputtering targets”) have to become bigger and bigger. In addition to the large dimensions, high dimensional accuracy and near net shaping are required to reduce necessary machining and to avoid excessive loss of the sometimes very expensive material (e.g. ITO = indium tin oxide). Therefore, the moulds must be manufactured with very high precision. Vacuum pressing technology is favorable for this process in order to avoid lamination and to achieve high green strength with low binder content. The uniaxial hydraulic pressing can be used as a precompaction process for subsequent hot isostatic pressing, offering the advantage of getting much closer to the required dimensions and to make better use of the capacity of the hot isostatic press. In case an optimized separate firing process is used, this process can completely dispense with the cost-intensive hot isostatic pressing. Several presses are used in industrial production, producing ITO sputtering targets up to a size of 420 mm x 1100 mm x 420 mm with a thickness of about 2 mm extremely low dimensional tolerances (thickness variation of fired plates). The green strength of such plates is high enough to remove them from the mould by means of a vacuum gripper or even manually. This development opens up a possibility of competing with tape casting technology, but working at a much lower binder and solvent content and with a higher throughput capacity. This technology could be used also for production of fuel cell components, e.g. for Ni based anodes of anode-supported SOFC (solid oxide fuel cells) or for zirconia based membranes of electrolyte-supported SOFC. Uniaxial hydraulic pressing technology has also been successfully introduced for the production of bipolar plates for PEM (proton exchange membrane) fuel cells, based on resin bonded carbon material, which is pressed at elevated temperatures where the resin shows plastic deformation. Such bipolar plates have a highly structured surface (ducts for gas transport), e.g. with a depth of the structure patterns of about 0.6 mm on both sides and an overall thickness of the plate of about 1.8 mm (Fig. 11). For plates with dimensions of up to 300 x 400 mm$^2$ and a thickness of about 2 mm extremely low dimensional tolerances (thickness variations $\pm 20 \mu m$) could be achieved.

Substrates for Electronic and Other Applications

Most recent developments in dry powder pressing technology with uniaxial hydraulic presses as well as development of improved binder systems allow for pressing ceramic plates with extremely high aspect ratios, especially very thin plates of large sizes. It is possible e.g. to press thin sheets of 250 x 250 mm$^2$ with a green thickness of $<1 mm$ (thickness after firing down to the range of 0.5 mm). As ceramic materials various alumina qualities, zirconia (Y-stabilized) and silicon carbide have been investigated, but others are also possible. With a suitable binding system the green strength of such plates is high enough to remove them from the mould by means of a vacuum gripper or even manually. This development opens up a possibility of competing with tape casting technology, but working at a much lower binder and solvent content and with a higher throughput capacity. This technology could be used also for production of fuel cell components, e.g. for Ni based anodes of anode-supported SOFC (solid oxide fuel cells) or for zirconia based membranes of electrolyte-supported SOFC. Uniaxial hydraulic pressing technology has also been successfully introduced for the production of bipolar plates for PEM (proton exchange membrane) fuel cells, based on resin bonded carbon material, which is pressed at elevated temperatures where the resin shows plastic deformation. Such bipolar plates have a highly structured surface (ducts for gas transport), e.g. with a depth of the structure patterns of about 0.6 mm on both sides and an overall thickness of the plate of about 1.8 mm (Fig. 11). For plates with dimensions of up to 300 x 400 mm$^2$ and a thickness of about 2 mm extremely low dimensional tolerances (thickness variations $\pm 20 \mu m$) could be achieved.

Other Carbon Products

Another interesting example showing the ability to realize very complex shapes by uniaxial hydraulic pressing with sophisticated moulds are carbon filter elements for molten metal...
filtering (Fig. 12). These elements were made from carbon (graphite + carbon black) with binder by pressing under vacuum, without subsequent machining. Also big carbon blocks as intermediates for machining of small parts are pressed, where it is mandatory to achieve a very homogeneous density distribution in all parts of the volume.

Special Kiln Furniture and Refractories

Also kiln furniture, e.g. for firing of electronic components like varistors etc. has to be considered as technical ceramics with appropriate requirements. Special systems based on high purity ZnO and corundum have been developed and are produced on hydraulic presses, where also aspect ratios of cassettes are realized which could not be pressed uniaxially in the past (Fig. 13). Most recently, also a technology has been developed for the pressing of 2-component sliding gates for the steel industry (Fig. 14).

Summary

Uniaxial hydraulic pressing is a well known and proven technology in the ceramic industry. It was used for "traditional" ceramic products for many years. New developments make this technology available also for the production of a wide variety of advanced ceramics, offering many advantages to the producer like high production capacity and flexibility, near net shape forming with high dimension-al accuracy, good densification with even density distribution, shaping of dry powders with low binder content, production of large dimension specimens with high aspect ratios and others. Though uniaxial hydraulic pressing is not an universal solution for each shaping task, it is a very flexible and versatile technology which can be considered in many cases as a reliable and economic alternative to isostatic pressing, injection moulding, pressure casting, tape casting and other shaping processes. Precondition for premium quality products is an optimal knowledge of the requirements in the individual processes and an appropriate adaptation of the pressing system and the pressing parameters using the best available state-of-the-art technology.

Acknowledgements

The author wants to thank R. Kremer (Alpha Ceramics GmbH, D) and K. Müller (LABS GmbH, L), who did most of the experimental and analytical work and made many valuable contributions to the experimental setup.

References