



Figure 1. Hydraulic press Alpha 1500 with 1,500-ton pressing force and filling height of 120 mm.

Credit: Alpha Ceramics, Aachen

# Case study— Applying lateral thinking to process development and optimization of specialty kiln furniture

By Roel van Loo

A German company manufactures tall, large-area saggars for a severe firing application by adapting an undersized press and pulling a vacuum.

Situated in the westernmost city of Germany, Alpha Ceramics GmbH in Aachen (ACA) offers a broad range of services for the ceramic industry and related sectors such as glass, refractory, concrete, powder metallurgy, and environmental technology. ACA, a subsidiary of Laeis GmbH (see sidebar), provides testing capacity for new customers as well as in-house process development and optimization. State-of-the-art production scale machinery and equipment for material preparation, shaping, and thermal treatment are available on site.

ACA offers its R&D and testing services to third parties, and the equipment is also available for direct toll productions. This possibility allows international customers to have newly developed products manufactured at ACA for a limited time, for example to test market acceptance or to bridge a time gap until a production plant comes online. Products that are required only in small lots and for which a separate production plant would not be economical can be supplied recurrently on call order. Emphasis of development activities at ACA focuses on applications. Through regular interaction with universities and other R&D institutions, however, new basic research developments also are integrated continuously. In addition, proprietary niche products are developed and directly marketed, especially various types of kiln furniture ranging from cordierite stacking aids to highly sophisticated mullite–corundum pusher plates for rapid-firing purposes.

The following case studies show how ACA developed unique adaptations to produce specialty refractories for extreme environments.

## Resources—Equipment and skills

ACA is equipped with superior technical equipment. The center owns an industrial spray dryer with an evaporation capacity of 180 L/h. It can spray-dry materials such as alumina, zirconia,  $AlTiO_3$ , raw material for sputtering targets, and tile bodies. Crushing and grinding machines include wet ball mills, and a pearl mill. Onsite intensive mixers of various sizes (5-, 40-, and

## Capsule summary

### CHALLENGE

The available press was not suited to form shapes with the high sides and large areas the customer required.

150-L useful volume) mix and prepare materials. As a subsidiary of Laeis, ACA has access to a range of sophisticated uniaxial hydraulic presses featuring modern control techniques and reliable hydraulic components. Three modified production Laeis presses are installed onsite, all with the ability to press under vacuum:

- Alpha 800 (800-ton pressing force with filling height of 80 mm);
- Alpha 1500 (1,500-ton pressing force with filling height of 120 mm) (Figure 1); and
- HPF 630 (630-ton pressing force with filling height of 600 mm).

A large variety of molds for those presses offer the ability to evaluate optimum pressing parameters for all types of products.

A range of equipment for thermal treatment, such as drying and firing of silicate ceramic and oxide ceramic products, whether shaped during customer's trials or customer-supplied green products includes:

- Laboratory dryers and kilns;
- Large-volume chamber dryer with climate control;
- Chamber kilns capable of temperatures to ~1,700°C; and
- Combination roller dryer/roller kiln for drying and firing products at moderately high temperatures (~1,400°C) in a relatively short time.

### About Alpha Ceramics GmbH

Alpha Ceramics in Aachen, Germany (ACA) is a member of TEAM by Sacmi, an alliance of Sacmi (Imola, Italy) companies that supplies cutting-edge technology for advanced ceramics production. TEAM by Sacmi combines the innovative skill and technology of Sacmi, Riedhammer (Nuremberg, Germany), Sama (Weissenstadt, Germany), Laeis (Wecker, Luxembourg), and ACA. ACA was founded in 1999 and serves as the R&D and technology center for Laeis, a manufacturer of hydraulic high-performance presses.

### THE APPROACH

A custom-built, three-step, pneumatically driven mold allowed proper compaction of powders. Careful powder preparation combined with vacuum pressing eliminated structural defects and delamination.

ACA has onsite laboratory equipment for material characterization and for checking basic properties of green and fired products. Detailed investigations, such as scanning electron microscopy and X-ray analysis, are conducted with analytical equipment at the Technical University of Aachen.

Installations are operated by skilled technical staff with scientific know-how, broad industrial expertise, and comprehensive knowledge based on successful developments during the past 15 years. Developments have served worldwide customers solving process- and machinery-related tasks.

### Case study—Saggars for severe firing environment

The following “design-to-production process” example shows how a customer-related development project reaches maturity—starting with design of the shaping mold; followed by selecting a proper body formulation, preparing the

### KEY POINT

Economic production of new products with unusual designs and exacting specifications can be accomplished by working creatively with available resources and paying attention to fundamental principles of ceramic manufacturing.

body, shaping under vacuum conditions, and firing at moderately high temperatures (all under appropriate quality control); and culminating with final product delivery to the customer.

ACA received an inquiry to produce refractory saggars with dimensions of 425 mm × 330 mm and a maximum height of 82 mm for use in a severe firing atmosphere demanding extraordinary resistance to corrosion and thermal shock. Products with a height greater than 50 mm normally must be made on press-type HPF to have enough filling height. However, press HPF 630 was occupied for other production. Also, it does not provide enough pressing force for such a large area. Therefore attention turned to whether the Alpha 1500/120 press could be used for this purpose and how to adapt the production process accordingly. This press is a modified version of an original tile press with an extended filling depth of 120 mm for producing advanced ceramics.



Figure 2. Mold for pressing saggars with more than 120 mm filling height in the Alpha 1500 press.



**Figure 3. Electrically heated top-hat kiln fires at temperatures up to 1,700°C.**

### Mold design

The maximum filling depth of the Alpha 1500/120 press is limited to 120 mm—an important consideration for design and construction of the press mold. A typical compaction ratio of refractory material is approximately 2:1, and one can calculate that this is insufficient for a saggars with maximum height of more than 80 mm. Gaining the needed filling depth required inventive construction of the pressing mold (Figure 2). The mold frame is held in pressing position with pneumatically driven "claws" and an equally driven mandrel, which can be moved manually after pressing. Thus, shaping of saggars with the required dimensions became possible in this press.

This technology also can be adapted and used for other saggars geometries or comparable products in the same way. At present the mold is filled manually. An automatic filling process, which will require a completely different approach, is under investigation.

### Body composition and preparation

The raw material selected for refractory saggars was ACA composition Alphoxit 82 RH (see Table 1).<sup>1</sup> The material is a mullite–corundum composite based on very pure and contamination-free raw materials. Oxides, such as  $K_2O$ ,  $Na_2O$ ,  $CaO$ ,  $MgO$ ,  $Fe_2O_3$ , and  $TiO_2$  negatively influence the refractoriness of mullite–corundum mineral mixtures.<sup>2</sup> These oxides can affect mullite crystal structure, cause release of  $SiO_2$ , and create low-melting eutectics. The material used consists of two fundamental phases: a coarse frame-building tabular alumina phase, and a fine mullite bonding matrix of sintered mullite, kaolin clay (with good conversion to mullite during sintering), and reactive alumina to adjust the stoichiometric ratio between  $Al_2O_3$  and free  $SiO_2$  to maximize mullite ( $3Al_2O_3 \cdot 2SiO_2$ ) content.

Minimizing the coefficient of thermal expansion of the bonding matrix, increasing three-point bend strength, and decreasing Young's modulus of the coarse building phase optimized thermal shock resistance. Optimizing particle size distribution achieved the latter two properties, resulting in a higher density material. These optimizations yielded a refractory material with excellent thermal shock resistance at service temperatures up to 1,450°C and high resistance against chemical, thermal, and oxidizing influences, making the material especially suitable for firing electrical ceramics, corrosive powders, and structural ceramics.

After optimizing formulation, the ceramic body composition must be prepared properly. Spray drying is used widely to convert separate raw material powders or mixtures into a free-flowing granulate of uniform bulk density. A water-based suspension was prepared of the above raw material formulation. ACA developed a proprietary procedure to avoid sedimentation of individual com-

ponents, eliminating heterogeneity in the spray-dried powder caused by segregation.

The slurry was injected under pressure into a spray dryer at 300°C using two-fluid (pneumatic) atomization. This technique uses compressed air to assist atomization, creating many droplets that quickly achieve a spherical shape because of surface tension. The large surface area-to-volume ratio of the droplets allows rapid water evaporation. Finished spray-dried granules have excellent flow properties, which ensures uniform filling of a press mold for shaping. Various spray-drying parameters, such as water content of the slurry, viscosity, pumping pressure, as well as nozzle type and diameter, can decisively influence granule properties.<sup>3,4</sup>

### Shaping

Uniaxial pressing of powder, granulates, and ceramic body formulations is the most common shaping technology for many areas of the ceramic industry.<sup>5,6</sup> To avoid texture heterogeneity or, in the worst case, delamination from entrapped air, standard pressing procedures often include several de-airing steps. Consequently, some air can leak through the edge gap between mold and die. Residual air, however, will accumulate in areas of the part that are compressed last. In the particular case of shaping a saggars, these areas are contact points (edges) between the wall and bottom surface. If compression pressure of the

**Table 1. Datasheet for Alphoxit 82\***

Composition*	
Constituent	Weight %
$Al_2O_3$	83.1
$SiO_2$	15.6
$Fe_2O_3$	0.2
$MgO + CaO$	0.1
$K_2O + Na_2O$	0.6
Mechanical and thermal properties	
Property	Value
Apparent density	2,600 kg/m <sup>3</sup>
Open porosity	27 vol%
Three-point bend strength (room temperature)	30 MPa
Young's modulus	16 GPa
Coefficient of thermal expansion (1,400°C)	$5.8 \times 10^{-6}/K$

\*Average values only. Not for design.

entrapped air exceeds the green strength of the saggars, it will burst or crack after load release.

In principle, there are two approaches to avoid texture damage caused by compacted air inclusion.

- Properly select an organic binder system to increase green strength of the pressed body to a sufficient level beyond compression pressure of the entrapped air; and

- Decrease the internal pressure caused by entrapped air to a level below the green strength of the pressed part, simply by reducing the amount of air inside the mold.

To avoid damage and lamination during production of the saggars described above, ACA chose the second option. This was realized with so-called vacuum pressing technology, where the mold is sealed and evacuated to a certain level (typically less than 100 mbar) before compacting the powder. ACA already optimized this technology in cooperation with Laeis for a variety of applications.<sup>7</sup> The mold cavity was evacuated within seconds to a residual air pressure less than 20 mbar. Doing so required minimizing the volume to be evacuated, which was achieved with a custom sealing technique. Cutting out de-airing strokes keeps cycle times constant or shortens them compared to conventional shaping technology. This new vacuum device thus enhanced product quality while maintaining output performance at a comparable level.

#### *Thermal treatment*

Substantial internal investigations have been conducted during the past several years on rapid-firing of kiln furniture. As a result, firing cycles have been decreased substantially, resulting in reduced energy consumption. This is especially important for products with a wall thickness up to 15 mm. Figure 3 shows the electrically heated top-hat kiln used to fire mullite–corundum saggars.

To optimize this firing process, ACA performed in-house differential thermal analyses. Thermographs showed several endothermic and exothermic reactions between 450°C and 1,200°C during heat-up of kaolinite. The main miner-

alogical transformation—thermal decomposition of kaolinite followed by reaction with alumina to form mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ )—occurs during firing of saggars in an electrically heated kiln.<sup>8</sup> Optimizing the kiln firing cycle made it possible to customize the microstructure of the bonding phase of the refractory material with regard to the special requirements for the application.

#### *Quality control*

Refractories, and especially kiln furniture, continuously experience thermal stress from heating up and cooling down over a long period of time. To determine thermal shock resistance (TSR)—which is the maximum tolerable temperature difference the component can withstand—modulus of rupture, thermal conductivity, and bend strength are measured on a specimen cut from a sample saggars (Figure 4). Fired density, open porosity, and water absorption are determined using Archimedes method to detect heterogeneity in the body or differences in density between the wall and bottom of saggars. Such differences can cause cracks during usage and shorten saggars lifetime.

Quality control tests showed that this new shaping technique does not harm the mechanical and thermal properties. In particular, density differences were reduced to a minimum. Initial runs in the customer's production under very severe kiln conditions look promising—saggars lifetime is already longer than those obtained from other suppliers. Saggars also show improved resistance against chemical, thermal, and oxidizing influences caused by the aggressive kiln atmosphere. This last benefit is a result of body formulation optimization over the past years.

#### **Specialty kiln furniture design**

Detailed investigations for other applications in recent years have led to other new products, many of which have been raised to industrial application. Some of these developments are described below.

#### *MgO substrates*

ACA started a ceramic body development study in response to customers



**Figure 4. Mullite–corundum saggars (425 x 330 mm 330 mm x 82 mm).**

seeking alternatives to available products, with the goal of manufacturing a 100%-fused MgO setter plate or disk for sintering electronic components. When firing electronic components, it is most important to avoid chemical interaction between the component and supporting kiln furniture. This study resulted in a new MgO material with suitable electrical and refractory properties for production to support electronic components during sintering. Production trials have shown that these MgO setter plates and disks perform excellently, particularly for firing electrical insulators at high temperatures (up to 1,600°C). Insulator quality is increased because there is no diffusion into the setter plates and vice versa. Another advantage of MgO setter plates is that the supporting kiln furniture lasts remarkably longer.

#### *Pusher plates*

A new kiln concept developed by TEAM by Sacmi member Riedhammer required a new formulation for pusher plates based on a mullite–corundum refractory material. Production of 390 mm × 460 mm × 41 mm pusher plates with an extremely good TSR will begin in the near future for firing advanced ceramic filter elements for the automotive industry. The firing regime calls for high loading density and exact temperature and atmosphere control in strict compliance with process requirements. The thermal processing plant for this application is extremely complex, requiring sophisticated components and solutions that extend to the kiln furniture used.



**Figure 5. Vacuum pressing eliminates structural defects in cordierite stacking aids with large height variations.**

### Cordierite stacking aids

A special application required complex-shaped stacking aids (Figure 5) with extreme height variations within a single piece. Normally this type of product is slip-cast to avoid density differences within the product. Shaping by hydraulic pressing was not an option because dry pressing does not allow such differences in thickness, and pressing of semiwet plasticized refractory mixtures often results in macroscopic structural defects or layer formation caused by entrapped air (or both). Such structural defects can be eliminated completely with vacuum pressing. The combination of preparation of bodies with exactly defined plasticity and pressing under vacuum conditions allows homogeneous compaction and shaping of complex products with extreme dimensional differences in pressing direction. Additionally, failures, such as cracks and out-of-specification dimensions, which can be caused by relatively high shrinkage in the slip-casting process, are reduced to a minimum when using this semi-wet pressing technology.

### Transparent spinel ceramics

One established process route for producing polycrystalline transparent spinel plates includes material preparation, uniaxial prepressing, cold isostatic pressing (CIP), binder removal, and hot isostatic pressing (HIP). When larger sizes are required, CIP can bottleneck the process chain. ACA and Laeis worked with the Fraunhofer Institute for Ceramic Technologies and Systems (Dresden, Germany) to optimize the uniaxial hydraulic pressing step and to avoid CIP completely. Optimization of the pressing regime in combination with vacuum pressing technology allowed pro-

duction of spinel plates with green densities close to those obtained by CIP. Large plates with dimensions of 300 mm × 400 mm and thickness of 12–15 mm were pressed, even though pressing force was limited to 120 MPa because of the size of the available press. After standard thermal treatment (including HIP), the plates were transparent (Figure 6). Transmittance values were practically the same as samples that were subjected to additional CIP for comparison. This feasibility study proved the possibility of producing large, crack-free, transparent spinel plates without expensive and time-consuming redensification by CIP. Further optimization work is necessary, however, and ACA currently is seeking partners to produce large-sized transparent spinel plates.

### Summary and outlook

Economic and ecological requirements often trigger a reconsideration of established process chains for the ceramic industry. At the same time, development of new products, especially advanced ceramics, needs feasible new process technologies. ACA's case studies show that creative and innovative approaches to adapting equipment, combined with proper body formulation and powder preparation, can lead to successful establishment of new products in production scale. Typically, projects resulted in less costly production techniques or better quality products. The saggars case study in particular proves that lateral thinking can spark new ideas and broaden horizons, thus overcoming alleged restrictions or even "impossibilities" in certain manufacturing technologies. For example, by adapting a mold rig and using vacuum pressing, saggars with a height of up to 80 mm and a densification factor of two were shaped using a press that originally had only 120 mm filling depth, which would not be possible in the conventional way. This opens the opportunity for new manufacturers and those already established in the mar-



**Figure 6. Transparent spinel plate (220 x 300 mm x 4 mm).**

ket to produce such refractory saggars with a relatively inexpensive hydraulic press, compared with alternative refractory press types. Investment costs hereby are remarkably reduced without any particular concessions regarding performance and product quality.

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