# Coarse-Grained Ceramic Heat Shields Made by Pressure Slip Casting

N. Gerlach, Chr. G. Aneziris, F. Lange, H. Grote

Pressure slip casting technology is a common manufacturing process in the silicate industry. The present study introduces a possibility for the production of carbon-free coarse-grained materials using this technology. At the beginning, the filtration behaviour of the slurries is studied in a CPF (compression permeability filtration) test cell. The specimens are ceramic heat shields manufactured by pressure slip casting and had to fulfil several requirements given by Siemens Power Generation. Ceramic heat shields are used as linings in gas turbine combustion chambers.

## 1 Introduction

During the last years pressure slip casting was particularly established for sanitary- and tableware ceramic [1]. The process offers a wide range of advantages. Large numbers of articles but also complex geometry can be produced within a short time. First research activities to use pressure slip casting also in technical ceramics were already accomplished [2]. However in the field of conventional and technical ceramics only fine-grained slurries were casted. First results of research show the possibility of applications in coarse-grained material systems [3].

In the pressure slip casting technology the casting takes place into a porous plastic mould with a pressure up to 20 bar. In opposition to the conventional slip casting the forms consists of polymethyl metacrylate (PMMA) with a median pore diameter of $d_{50}$ = 20 µm and exhibit a higher life cycle. The formation of the body is a filtration process and can be described with the following equation (1):

$$s^2 = \frac{2 \cdot \Delta\rho}{\eta_s \cdot \Phi_c \cdot \rho_p \cdot R_c} \cdot \frac{\phi_s}{(\phi_c \cdot \phi_s} \cdot t \qquad (1)$$

s is the cake wall thickness, Δp the pressure difference, $\eta_s$ the dynamic viscosity of the suspension, $\Phi_c$ the solid content of cake, $\Phi_s$ the solid content of suspension, $\rho_p$ the particle bulk density, Rc the cake filtration resistance and t is the casting or filtration time.

The formation of the ceramic component can be positively affected by the following parameters. Apart from an increase of the casting pressure also a heating up of the slurries can lead to an accelerated formation of the body. The water transportation in the pore volume of the ceramic component is affected to a large extent by its permeability. The body formation rate rises with decrease of the specific surface of the raw materials [4–6].

The porosity of the mould material has a significant influence on the formation of the first solid layer at the surface. The fine-grained particles are about a multiple smaller than the median pore diameter of the mould. A broad particle size distribution and high solid contents in the suspension are advantageous and lead to an arching over the pores of the mould [3–4].

Generally clay minerals support the filter cake formation at the surface of the mould. Due to their absence an adequate binder system is necessary, to prevent the infiltration of the fine particles into the open porosity of the mould material.

The scope of the present work was to establish an environmentally friendly binder system and to manufacture dimensionally stable heat shields after casting. The demoulding behaviour, the component homogeneity, the casting time as well as the thermal shock resistance have been optimised. For the production of the samples a special mould with defined dimensions of a ceramic heat shield was manufactured. The casting attempts took place in a pressure slip casting machine in accordance with the patent

**Tab. 1 The requirements for ceramic heat shields**

| | |
|---|---|
| Cold modulus of rupture [MPa] | 9–12 |
| Hot modulus of rupture$_{1200\,°C}$ [MPa] | 6–8 |
| Dyn. Young's modulus [GPa] | <30 |
| Open porosity [%] | 18–20 |
| Bulk density ρ [g/cm³] | 2,8–3,0 |

*Nora Gerlach, Christos G. Aneziris*
*Institut für Keramik, Glas- und Baustofftechnik*
*TU Bergakademie Freiberg*
09596 Freiberg
Germany

*Friederike Lange, Holger Grote*
*Siemens AG Energy Sector*
45472 Mülheim an der Ruhr
Germany

Corresponding author: *Nora Gerlach*
E-mail: nora.gerlach@ikgb.tu-freiberg.de

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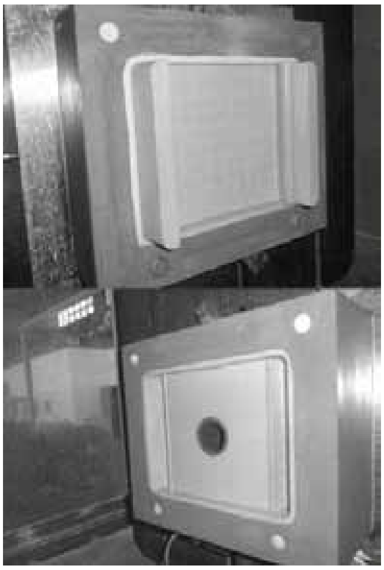

**Fig. 1** Quartered mould for heat shields

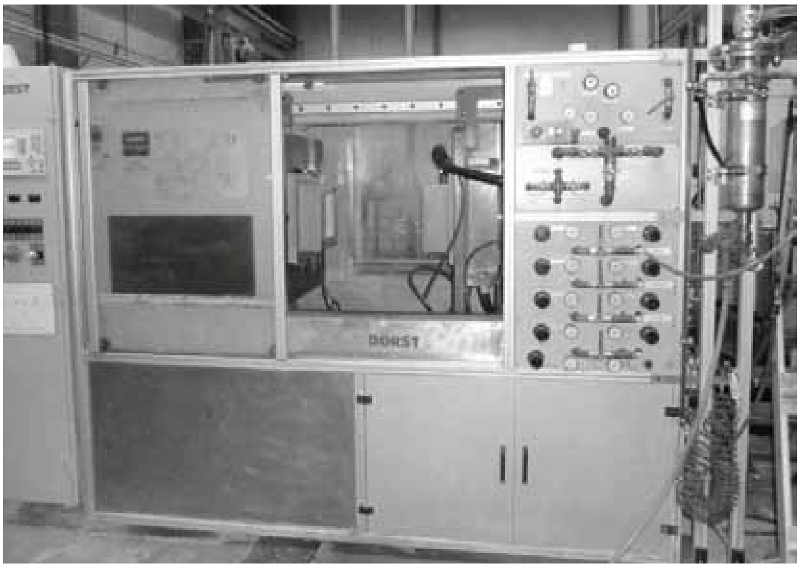

**Fig. 2** Pressure slip casting machine DGM-80D from DORST/DE

of DORST Technologies, Germany [7]. The developed materials had to fulfil several requirements given by Siemens Power Generation, as listed in Tab. 1.
In addition the potential use of pressure slip casting technology to create ceramic heat shields was a main objective in this work.

## 2 Experimental

The slurries, containing reactive alumina, tabular alumina and fused mullite, were prepared in an Eirich RV02 (Eirich/DE) mixer at 3000 rpm. At first the ceramic powders were dry mixed for 5 min. In a next step the water including all additives (mixed by a Heidolph RZR 2102 stirrer, 500 rpm) was added gradually. The slip was homogenized for further 20 min. After a degassing time of 15 min the slip could be used for casting.
At the beginning an optimal system of deflocculants and binders had to be determined. Due to the maximum grain size of several millimetres, it was important to avoid a decomposition of the slurry. Furthermore an optimal grain size distribution had to be found. Therefore an evaluation using Dinger Funk equitation was needed [8].

$$q(x) = \frac{x^k - x_{min}^k}{x_{max}^k - x_{min}^k} \qquad (2)$$

q (x) is the cumulative mesh of fraction x, x the particle size of fraction and $x_{min}$/$x_{max}$ the minimum/ maximum particle size in the batch. The distribution parameter k was determined at 0,25. This is an optimal value for a stable slip [9].

The slurries were stabilized using an additive system consisting of a deflocculant and a temporary binder. The relatively high water content of the heat shields after demoulding made the use of a temporary binder necessary. Due to the temporary binder the green strength of the heat shields could be increased after demoulding. Furthermore, crack formation has been reduced. To avoid sedimentation of coarse particles a gelling agent was tested. Those thickening agents increase the contact area between the particles.
Due to the coarse grain size distribution, the use of a rotation viscometer (maximal grain size ≤1 mm) was not possible. Therefore a falling sphere viscometer was preferred for testing. The falling sphere viscometer was calibrated with a Newton's liquid at the beginning. The temperature of the slurries was kept constant at 21 °C. During the measurement the fall velocity of the sphere was calculated over a defined path per time. The dynamic viscosity can be evaluated with the Stokes' equation (3).

$$\eta_s = \frac{2 \cdot r^2 \cdot g}{9 \cdot \upsilon} (\rho_s \cdot \rho_f) \qquad (3)$$

$\eta_s$ is the dynamic viscosity of the suspension, r the sphere radius, g the gravitational acceleration, $\upsilon$ the the fall velocity, $\rho_f$ the fluid (slurry) density, and ρs is the sphere density.
After the rheological analysis the filtration behavior of the slurries was determined using the CPF (compression permeability filtration) test [10–12]. According to the material of the mould a filter paper with a middle pore diameter of 20 µm was used. The filtration behaviour should be examined at 1,5 bar (filling pressure of the form) and at 4 bar (maximum pressure of the CPF cell). With equation (4) the filtration resistance Rc of the filter cake was determined.

$$\frac{\delta t}{\delta V} = \frac{\eta_w}{A \Delta p}\left(R_c \frac{H_e}{V_e}\frac{V(t)}{H(t)} + R_m\right) \qquad (4)$$

$\eta_w$ is the dynamic viscosity of water, A the filter area, Δp the pressure difference, $R_c$ the filtration resistance of cake, $H_e$ the total cake height, $V_e$ the total filtrate volume, V the filtrate volume, H the cake height, and $R_m$ is the filtration resistance of filter.
After the characterization of the slurries and the selection of an adequate casting slip attempts were accomplished in the pressure slip casting machine DGM-80D by DORST Technologies (Fig. 2). It was converted especially for the casting of coarse-grained slurries, as described in the patent, [7]. Fig. 1 demonstrates the quartered mould for the production of the heat shields. The form consists of two fixedly installed mould halves and two inserts which are demoulded with the body and removed later. The form material possessed a middle pore diameter of 20 µm. Before each casting the form was rinsed alternatingly with air and water for approx 15 min. After closing the form the slip was added into the holding tank. Filling the mould took place with a filling pressure of 1,5 bar and a filling time of

3 min. The pressure was increased in 5 min to 20 bar. The casting time, e. g. closing the form up to opening, amounted to 30 min. For the removal of the heat shields release air was given to the mould.

The drying process took place in three steps in the drying furnace WSU 100 of the company MLW/DE (40 °C for 24 h, 80 °C for 6 h and 120 °C for 24 h). The dried heat shields were sintered in an electric furnace (Nabertherm/DE (276/17 HAVE)) at 1600 °C. Apart from the rheological investigations the sintered heat shields were cut in bars with the dimensions 25 mm × 25 mm × 150 mm and their cold modulus of rupture (TIRA test 2420, Franz well-being & Partner/DE) and hot modulus of rupture were determined (Hot modulus of rupture tester 422, NETZSCH/DE). On cylinders with a height of 50 mm and a diameter of 50 mm the refractoriness under load up to 1600 °C was quantified in an NETZSCH TASC 414/4 hood kiln. The microstructure was examined using scanning electron microscopy at polished samples. Using the water absorption method the total porosity and the bulk density was evaluated.

## 3 Results

With the aid of the CPF test the filtration behaviour at 1,5 bar and 4,0 bar should be regarded. The used filter paper possessed a middle pore diameter of 20 µm according to the material of the form. The filter filtration resistance of the cake and the filtering medium were calculated using equation 2.

As expected the filtration resistance of filter is equal for both pressures (Tab. 2). The differences of the filtration resistance of cake at 1,5 and 4,0 bar suggest solidification. A compression of the bodies due to higher pressures takes place.

A lower filtration resistance of cake causes a better filtration. The results show that the dewatering occurs more slowly with increasing pressures. To result in short casting times the pressure has to be increased. In addition to this behaviour the filter cake resistance rises with increasing pressure. After variation of the particle size distribution and the optimization of the casting parameters stable heat shields could be manufactured, Fig. 3. The casting time was approx 30 min, the maximum pressure 20 bar.

Subsequently the heat shields were sintered as described in the experimental part and prepared for further investigations. In Tab. 3 the mechanical characteristics of the sintered samples are displayed. These results relate to the requirements made by Siemens Energy (Tab. 1).

For an employment in the gas turbine the characterization by the refractoriness under load was mandatory. The test results are summarized in Tab. 4. The temperatures for $T_{0,50\,\%}$ and $T_{1,00\,\%}$ could not be determined, because the maximum temperature of the Nabertherm kiln was achieved. For the application as a heat shield the temperatures are in the rated range.

The microstructure characterization by scanning electron microscope, shows a homogeneously distribution of coarse and fine particles (Fig. 4). No separation is visible. The coarse grains are well sintered with the bond matrix. The structure is porous; capillary tubes of the de-watering process are remaining.

These results are supposed to be a very good basis for ongoing developments for

**Tab. 2** Computed filtering medium and filter cake resistances

| Max. Pressure [bar] | Filtration Resistance of Filter $R_m$ [1/m] | Filtration Resistance of Cake $R_c$ [1/m] |
|---|---|---|
| 4,0 | 6,56E + 15 | 4,50E + 15 |
| 1,5 | 9,95E + 15 | 3,50E + 10 |

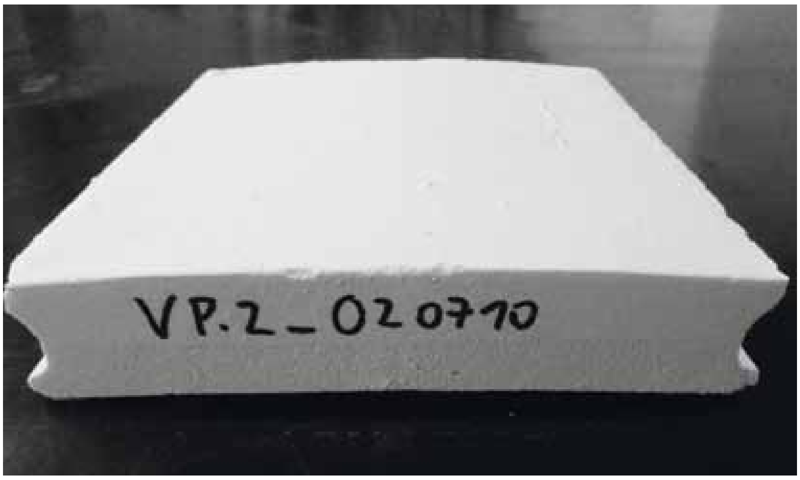


**Fig. 3** Heat shield made by pressure slip casting

**Tab. 3** Mechanical qualities of the slip formulation out of Tab. 2

| Cold Modulus of Rupture [N/mm²] | Dyn. Young's Modulus [GPa] | Bulk Density [g/cm³] | Total Porosity [%] | Hot Modulus of Rupture [N/mm²] |
|---|---|---|---|---|
| 15,9 ± 0,17 | 33 ± 1,08 | 2,87 ± 0,00 | 23 ± 0,05 | 8,7 ± 0,79 |

**Tab. 4** Results of refractoriness under load

| Softening Point | $T_{0,10\,\%}$ | $T_{0,50\,\%}$ | $T_{1,00\,\%}$ |
|---|---|---|---|
| 1495 °C | 1576 °C | >1650 °C (*) | >1650 °C (*) |

(*) Measurement was possible up to 1650 °C

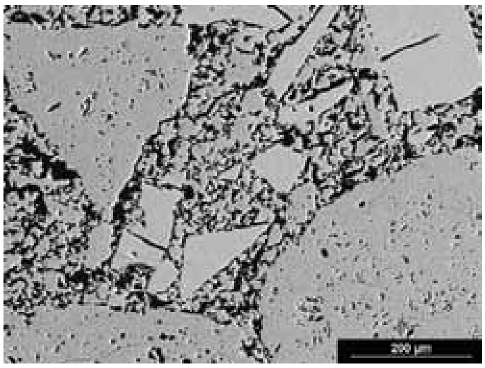


**Fig. 4** Microstructure characterized by scanning electron microscopy, (REM Fa. Philips, 25 kV, 200×, RE)

coarse grained refractory material manufactured by pressure slip casting.

## 4 Conclusions

The presented results demonstrate that ceramic heat shields could be manufactured by pressure slip casting technology. By variation of the grain size distribution and an optimization of the pressure slip casting technology, the production of dimensional stable heat shields was possible. The most optimal results were achieved at a casting time of 30 min with a casting pressure of 20 bar. The used binder system supplies a low viscosity and thus an optimal liquefaction, with a small water content less than 20 mass-%. The mechanical characteristics of the heat shields fulfil the requirements which were given by Siemens Power Generation. Investigations by means of refractoriness under load showed that the high temperature characteristics for the use in the gas turbine are sufficient.

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