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Pressure slip casting of coarse grain oxide ceramics

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Abstract

This contribution investigates the pressure slip casting of large coarse grain oxide ceramic bodies with a water soluble organic additive system. This organic additive system allows the preparation of a stable and pumpable slip containing alumina rich magnesia aluminate spinel of a size of up to 3 mm and an easy demolding of crack free, dimensionally stable bodies with negligible gradients due to sedimentation. Cut out samples of fired bodies are examined on apparent porosity, dynamic elastic modulus, modulus of rupture, and pore size distribution. Computer tomography showed very homogenous and dense bodies. The effects of different maximum grain sizes as well as possible sedimentation and segregation of the slip on the mechanical properties and microstructure are evaluated by using the Student's t-test. The most promising results of this study indicate that it is possible to reproducible fabricate coarse grain ceramics for refractory and other high temperature applications by pressure slip casting.

Keywords: A. Slip Casting; E. Refractories; Pressure slip casting; Coarse grain ceramics; Magnesia aluminate spinel

1. Introduction

The enhancement of ceramic slip casting by external pressure instead of capillary suction, the so called pressure slip casting, is known for over eighty years [1]. Conventional slip casting usually uses porous plaster of Paris molds, which can be used for about 100 times. Due to capillary forces of the pores in the mold the dispersing medium in the slip is absorbed and the cast layer is formed at the interface of the mold with the slip [2]. Slip casting can be considered as a filtration process [3], which is why the buildup of the filtration cake can be accelerated either by vacuum on the mold side or by additional pump pressure on the slip side. Consequently capillary active plaster of Paris molds with a capillary pressure of less than 200 kPa have been replaced by polymeric molds with larger pores because they allow an effective casting pressure of up to 4 and therefore substantially reduce the casting time. In addition, these plastic molds are more durable and can be used for several thousand times [2,4–6].

In general slip casting is a profitable technology for large, hollow shapes of thin to modest wall thicknesses and for the production of complicated, near-net shape bodies of low texturing [2]. Using pressure slip casting even bodies of larger wall thicknesses can be formed in reasonable process times. Moreover it offers the possibility of automation, which is one reason why it is more profitable than conventional slip casting.

Today, pressure slip casting is state of the art for the production of ceramics containing clay minerals such as sanitary ware, cookware and China. Furthermore there has been considerable effort to produce technical ceramics by pressure slip casting [5–8]. However, until recently there has been only little attention paid to the possibility of forming coarse grained ceramics by pressure slip casting.

The adding of coarse grain into the ceramic matrix has several benefits for high temperature applications, such as improving the thermal shock, corrosion, and creep resistance. Besides, the sintering shrinkage is reduced as well. The improvement of the thermal shock resistance results mainly from a microstructure with a microcrack network. This microcrack network can be induced by thermal expansion mismatch of different phases or by a faster sintering of the finer particle fraction than the coarse grain fraction [9,10].

In a previous work Klippel et al. [11] evaluated significant parameters for the pressure slip casting of suspensions with coarse granular grain. In that study a falling ball viscometer has been applied to investigate the rheological behavior of the slips, i.e. thixotropy and rheopexy. Furthermore, they stated the importance of a plug flow of the slip in the tubes during filling

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of the mold. A plug flow can be achieved through a moderate filling pressure and an adequate large inner tube diameter, depending on the maximum particle size of the slip. In addition, filtration experiments in a compression permeability filtration (CPF) cell as well as scale up experiments in a commercial pressure slip casting machine were conducted. These scale up experiments had the drawback that it was not possible to demold a dimensionally stable body.

Through modifications of the additive system it was possible to overcome these drawbacks. Thus, the purpose of this study is to describe and evaluate the pressure slip casting of large coarse grain alumina rich magnesium aluminum spinel ceramics by determining microstructure features and mechanical properties.

2. Experimental

2.1. Materials and slip preparation

The current study investigates the preparation and testing of fired samples obtained from two different slips. Both slips contained alumina-rich magnesium aluminate spinel (AR 78, Almatis GmbH, Germany) and a fraction of reactive alumina (MR 70, Martinswerk GmbH, Germany). The Magnesium aluminate spinel was chosen due to its wide use as a refractory material for castables [12]. All the samples were prepared and tested in a random order for the purpose of reducing experimental flaws. The particle size distribution was formulated according to the packing model of particles described in Eq. (1) [13–16].

$$P_i = \frac{d_i^q - d_{\min}^q}{d_{\max}^q - d_{\min}^q} \tag{1}$$

d_i is the particle size of fraction i. $d_{\max}$ and $d_{\min}$ are the maximum and minimum particle size of the mixture. P_i is the cumulative percentage finer than. q is the distribution parameter.

As reported by Klippel et al. [11] the particle size distributions were optimized with a distribution parameter of $q = 0.25$, ensuring a low-thixotropy of the slip. In this study, the maximal grain size of the two slips was 1 mm and 3 mm, respectively, with the intention to estimate the influence of the maximal grain size on the stability of the slips and on other properties of the fired bodies. The quantiles of the particle size distribution of the used ceramic raw materials are listed in Table 1. The particle sizes were determined by sieve analysis according to the standard DIN 66165-2 for the two larges particle fractions and by laser granulometry (Beckman Coulter LS230, Beckman Coulter Inc., USA) according to the standard DIN EN 725-5 for the others.

In order to prevent sedimentation of the coarse grain and provide sufficient green strength after casting, organic additives were admixed. The additive system was adapted from [11], but the added amounts were slightly higher. The addive system consisted of a commercial binder (0.9 wt.%, Optapix AC 170, Zschimmer & Schwarz GmbH & Co KG, Germany), a commercial dispersant (0.025 wt.%, Dolapix CE 64, Zschimmer & Schwarz GmbH & Co KG, Germany), and 0.02 wt.% Xanthan as a stabilizer. According to the manufacturer Optapix AC 170 is a polymer dispersion, whereas Dolapix CE 64 is described as a ammonium polyacrylate solution [17]. All the additves were carefully solved without flocculation in 12 wt.% purified water as the dispersing medium using a Heidolph RZR2102 visco-jet stirrer. Then the water with the containing additives and the solid content were well mixed in a laboratory intensive mixer (RV02, Maschinenfabrik Gustav Eirich GmbH & Co KG, Germany) at 3000 rpm for 10 min in order to obtain an according to the standard DIN EN 1402 self-flowing, yet stable slip. During the mixing the slip was examined several times on visible agglomerates. The slip was characterized as stable if there was no water layer on the slip surface visible after five minutes and self flowing if it was running in one stream from a trowel and showed a spreading of at least 80%.

After the slips were examined on stability, they were immediately filled into a receiver tank and pumped into the vertical positioned mold by pressurized air applying a pressure of 0.1 MPa for 3 min. With the intention of performing experiments with coarse grain suspensions a commercial pressure slip casting machine (DGM80D, Dorst Technologies, Germany) was modified according to Aneziris et al. [18]. The mold had a size of 200 mm × 200 mm × 38 mm with the sprue in the middle of the rectangular mold. The median pore size d_{50} of the mold was 20 μm. Once the mold was filled, the pressure was increased slowly to 0.8 MPa and kept at that pressure for 30 min. After pressure release the shaped bodies were gently demolded by counter-current pressurized air and hand thereby preventing cracking. However, the demolding had to be conducted as fast as possible to avoid disturbance of the body surface. Subsequently, the green bodies were dried at 40 °C and 110 °C for 24 h in each case and later fired with a heating rate of 3 K min^{-1} at 1650 °C for 2 h in an oxidizing atmosphere.

Table 1
Particle sizes of the used ceramic raw materials.

Product name	Raw material	Labeling	d_{10}	d_{50}	d_{90}
Almatis AR 78	$MgAl_2O_4$	1–3 mm	0.8 mm	1.3 mm	2.25 mm
		0.5–1 mm	250 μm	400 μm	800 μm
		0–0.5 mm	54.84 μm	314.2 μm	653.7 μm
		0–0.09 mm	0.944 μm	19.98 μm	98.34 μm
		0–0.045 mm	0.761 μm	12.80 μm	36.23 μm
Martinswerk MR 70	Al_2O_3	–	0.412 μm	0.791 μm	1.668 μm