

Short communication

The influence of solid loading in suspensions of a submicrometric alumina powder on green and sintered pressure filtrated samples

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Abstract

The present work deals with preparation of stable suspensions of a submicrometric alumina powder with different contents of solid for pressure filtration. The optimum dispersant content (2.2 wt.% of Darvan C-N) was determined by sedimentation tests and viscosity measurements. By modification of the solid loading and dispersant content two kinds of aggregation were observed. One type of aggregates is related to the use of excessive solid loading in suspension. In samples prepared from these suspensions only minor effect on sintered microstructure was observed, which increased with increasing volume fraction of hard aggregates. In case of excess dispersant addition weak aggregates formed as the result of depletion flocculation. Weak aggregates had stronger negative effect on green microstructure, with consequent negative impact on sinterability. © 2009 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: Aggregate; Alumina; Suspension**1. Introduction**

Colloidal techniques in the manufacturing of ceramics provide considerable benefits for the control of packing uniformity of consolidated powder forms. Compared to powder consolidation in dry or semi-dry state (e.g. pressing in a die), colloidal methods can lead to better packing homogeneity in the green body, which in turn leads to better microstructure control during firing [1–3]. The quality of products formed by wet processing is determined primarily by the state of the dispersion. Dispersion of the particles and the stability of the suspension, therefore, are the key factors for the successful production of a large number of industrial products [4].

Many recently developed ceramic processing techniques including spray drying, slip casting, pressure casting, tape casting, and gel casting employ well-dispersed suspensions with very high levels of solid loading. For these forming techniques, the concentration of suspension influences the green density of the formed ceramic material.

Pressure filtration (PF) is separation of suspension into a green cast and a pure filtrate liquid. The green cast is formed at a

porous filter which is impermeable for the particles but permeable for the liquid. The driving force is a static pressure difference either accomplished by applying vacuum on the backside of the filter (vacuum casting) or by applying a high pressure on the suspension. Using a constant filtration rate instead of constant pressure, a constant growth rate of the cake is induced, which provides homogeneous particle incorporation into the growing cake surface. Therefore, particle packing structure is independent of cake thickness so that gradient-free green compacts are formed. Owing to increasing flow resistance the applied pressure has to be continuously increased with cake thickness [5]. Green compacts of high density are usually produced by PF techniques of highly concentrated (about 49 vol.%) powder suspensions [5], but high relative green density up to 60% can be achieved also from suspensions with only 20 vol.% [6] and 30 vol.% of solid [7].

A number of works deal with the influence of the solid loading and dispersion state of suspension during PF, and their relation to green microstructure [6,8–10], but only a few is focused at the properties of sintered material prepared from these suspension [11,12]. Uchikoshi et al. [12] prepared compacts from two suspensions: well-dispersed and insufficiently dispersed suspensions of fine zirconia powder. Green sample consolidated from the well-dispersed suspension

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showed a good sinterability, while CIP treatment at 400 MPa was needed to increase the sintered density of the compact consolidated from the insufficiently dispersed suspension. Garrido and Aglietti in their work compare [11] the pressure filtration and slip casting of mixed alumina–zircon suspensions. Pressure filtrated bodies were slightly less dense than the compacts prepared by slip casting and relative green density increased with increasing the degree of dispersion of the particles. After sintering 2 h sintering at 1600 °C a clear relation between the green and the sintered density for all compositions was observed, showing that the high green density gave the highest sintered density.

The main objective of this work is the investigation of the influence of different solid loadings on the properties of suspensions prepared from a submicrometric alumina powder, specifically focused on the influence of aggregates at high solid contents. The effect of solid loading on density and porosity of pressure filtrated green samples is also examined. The effect of the excess of a Darvan C-N dispersant on green body is observed and analyzed. The impact of the suspension properties on sintering and final microstructure is discussed.

2. Experimental

High purity 99.99% commercial alumina powder (TAI-MICRON TM-DAR, Taimei Chemicals Co., Ltd., Tokyo, Japan) with primary particle size of 150 nm and specific surface area $13.7 \text{ m}^2 \text{ g}^{-1}$ was used as a starting material. To find out the optimal concentration of dispersant five different concentrations of Darvan C-N (R.T. Vanderbilt Company, Inc., Norwalk, USA) were used: 1.8, 2.0, 2.1, 2.2 and 2.4 wt.% relative to the weight of alumina, respectively, in a 40 vol.% of alumina suspension. The suspensions were prepared in three steps: mixing of distilled water with dispersant, addition of alumina powder, and mixing for 24 h on rollers with alumina milling balls. The rheological properties of the suspensions were examined by viscosity measurements, which have been carried out using a Rheometer AR 2000 (TA Instruments) with Plate/Plate geometry (Peltier plate + 40 mm Steel Plate). The measurement conditions: pre-shear = 100 s^{-1} during 60 s and than 60 s equilibration, duration of measurement = 60 s, shear stress = 0.01–100 Pa, temperature 25 °C.

The sedimentation tests were also used for the optimization of the dispersant content. For the test, 0.75 ml of the 40 vol.% alumina suspension with different contents of dispersant was mixed with 24.25 ml of distilled water. The suspensions were first stirred thoroughly for 10 min in a beaker and then transferred to the measuring cylinder where they were allowed to stand undisturbed for 48 h. The suspension density was measured by pipetting out 5 ml of the cloudy suspension from a predetermined height (corresponding to a volume of 15 ml) from the top of the cylinder. It was then dried and weighed in order to determine the weight of suspended particles in the 5 ml of the suspension. The volume of suspension containing dispersed particles above the settled mass at the bottom was calculated after subtracting the space covered by the sediment. The total amount of solid remaining in suspension was then

estimated from the total volume of suspension over the sediment bed. The remaining solid was assumed to have been reported in the sediment, the volume of which is read directly at the bottom of the graduated cylinder. The relative density of the sediment was then estimated from the ratio of the density of the sediment to the true density of the solid.

To determine the optimal concentration of alumina in suspension in order to optimize the properties of green body six different concentrations were used: 20, 30, 40, 45, 46 and 47 vol.% of alumina (denoted as A20–A47 in the following text), respectively. In addition, a suspension with 40 vol.% of alumina with 5 wt.% of dispersant (A40DC) was also prepared. The suspensions were used for particle size analysis (LS 230 Laser Diffraction Particle Size Analyzer, measured in EMPA, Duebendorf, Switzerland).

Green samples were prepared by vacuum–pressure filtration (constant consolidation rate 0.4 mm/min, maximal consolidation pressure 5.795 MPa) of suspensions with different solid loadings. Vacuum was applied during the whole process of consolidation. Samples were then dried in drying chamber at 120 °C for 2 h. Green samples were used for porosity measurements (PASCAL 140 and 440, measured in EMPA Duebendorf).

The sintering was carried out in an electrical furnace (NETZSCH GmbH, Selb, Germany) with MoSi₂ heating elements in air. During the sintering process the specimens were heated at 20 °C/min up to the maximum temperature 1250 and 1275 °C with 10 min and 1 h of isothermal dwell, respectively, and then cooled down to room temperature. Density was measured by the Archimedeian method in mercury. Sintered microstructures were examined by scanning electron microscopy (Zeiss EVO 40 HV, Germany), at fracture surfaces. For the determination of the average grain size linear intercept method was used.

3. Results and discussion

3.1. Content of dispersant

To obtain a ceramic suspension with high solids content that can be successfully processed, relatively low viscosity must be achieved and stability must be maintained. This requires efficient dispersant added in optimal amount. Lower dispersant concentration results in insufficient degree of dispersion (not all particles are dispersed) whereas too high addition can result in depletion flocculation [13]. In both cases the agglomerates present in suspension increase the suspension viscosity.

Fig. 1 shows the viscosities plotted against the shear rate of 40 vol.% alumina suspensions with five different additions of the dispersant. The lowest addition of dispersant (1.8 wt.%) results in the highest viscosity. With increasing amount of the dispersant the viscosity gradually decreases up to the content 2.2 wt.%. After that, higher dispersant concentration results in higher viscosity again.

The stability of suspensions was evaluated also by sedimentation tests. The effect of different additions of dispersant on relative density of sediment is shown in Fig. 2.

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