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Mullite compacts obtained by colloidal filtration of alumina powders dispersed in colloidal silica suspensions

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Abstract

This work deals with the manufacture of mullite-matrix composites by a colloidal filtration route. A variation of the transient viscous sintering process is studied in which submicrometer sized alumina particles are dispersed in a nanosize colloidal silica suspension. High solids loading suspensions (45 and 50 vol.%) are prepared by ultrasonic mixing and dispersed with citric acid. Rheological optimisation allows to obtain low viscosities and to reduce thixotropy and ageing effects. Specimens are obtained by slip casting and sintered to 1550 °C/2 h by using different heating cycles, the best results being obtained with a plateau after the maximum rate of sintering where reaction occurs.
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1. Introduction

Mullite and mullite-matrix composites are attractive for high temperature structural applications due to their excellent thermo-mechanical properties [1,2]. However, sintering of mullite powders into dense compacts requires high temperatures due to its diffusion coefficient [3]. To enhance densification many studies have been directed toward reduction of particle size and improvement of the green microstructure [4].

Another approach to prepare mullite bodies at lower sintering temperature is by using mullite precursors that react after densification has occurred. The most popular way is by means of sol–gel techniques [5]. However, warpage and cracking is usual. Moreover, densification of mullite-matrix composites is difficult because once mullitization occurs densification is arrested until much higher temperatures [4,6].

Sacks et al. [3,4] proposed a transient viscous sintering (TVS) process in which α-alumina powders instead of alumina precursors were used and coated with amorphous silica source, like TEOS, as in other sol–gel processes. Silica was precipitated onto alumina by adding ammoniated water. These particles were dispersed in water to solids loadings of 30–35 vol.%, slip cast and sintered at 1500–1600 °C to produce mullite.

This work deals with the preparation of mullite bodies through a colloidal filtration route by using concentrated suspensions of submicrometer-sized α-Al_2O_3 powders dispersed in a commercial colloidal silica suspension. Colloidal silica suspensions are readily available and have a very low price, so that the process is cheaper than that reported by Sacks et al. and the use of organic precursors (i.e. TEOS) is replaced by a clean aqueous processing.

By optimising the dispersing conditions through rheological measurements, it is possible to produce uniform composite compacts by colloidal filtration that densify and react to produce mullite by reaction sintering.

2. Experimental

The following starting materials were used: a commercial α-alumina (ALCOA CT3000SG, USA), with average particle size of 0.6 μm and specific surface area of 8 m^2/g, and a colloidal silica suspension (LEVASIL 200A/40%) containing 40 wt.% particles with average size of 15 nm, specific surface area of 200 m^2/g and pH value of 9.

Mixtures were prepared with alumina/silica molar ratio of 13/5 (82/18, wt./wt.), with an excess of Al_2O_3 over the

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stoichiometric ratio of mullite 3:2 (72/28, wt./wt.), which is expected to remain as a secondary phase. Suspensions were prepared in deionised water to total solids loadings of 45 vol.% (70 wt.%) and 50 vol.% (75 wt.%), including the water added with the colloidal silica suspension. Citric acid (PROBUS, Spain) was used as a dispersant in concentrations ranging from 0.1 to 0.5 wt.% on a dry solids basis. Homogenisation was carried out using an ultrasounds probe (IKA 400US, IKA, Germany) for mixing times of 1, 3, 5 and 10 min. The final pH of the mixtures was 6.5.

The rheological behaviour was studied using a rheometer (RS50, Haake,Germany) with a double-cone/plate sensor configuration (DC60/2°, Haake, Germany). Flow curves were obtained with a three-stage measuring program with a linear increase of shear rate from 0 to 1000 s^{-1} in 300 s, a plateau at 1000 s^{-1} for 120 s, and a further decrease to zero shear rate in 300 s. The influence of the volume fraction of solids, the citric acid concentration and the time of ultrasonic mixing on the flow behaviour was studied. Flow curves of every suspension were also determined after times of 30, 60, and 90 min, in order to establish the ageing behaviour. Thixotropy was calculated as the area enclosed between the up-curve and the down-curve in the controlled rate flow curves for the measuring conditions described above.

Ceramic pieces were obtained by slip casting on plaster of Paris moulds. Cast green densities were determined by Archimedes' method in mercury after drying for 48 h at room temperature. Dynamic sintering studies were performed with a differential dilatometer (Setsys 16/18, Setaram, France). For static sintering tests, green specimens were treated according to three different sintering schedules: (a) heating rate of 5 °C/min up to 1550 °C/2 h; (b) heating rate of 5 °C/min up to 1550 °C/2 h with an additional isothermal step at 1250 °C/1 h, considered as the temperature at which flow phenomena occur; and (c) heating rate of 5 °C/min up to 1550 °C/2 h with an isothermal step at 1350 °C/1 h, considered as the nucleation temperature of mullite.

Table 1
Thixotropy (Pa/s) of 50 vol.% Al_2O_3/SiO_2 fresh and aged suspensions prepared with different sonication times

Ageing time (min)	Thixotropy (Pa/s, × 10^4) Sonication time (min)			
	1	3	5	10
Fresh	0.03	0.26	0.20	0.02
30	3300	1.70	0.71	0.52
60	7300	5.10	1.90	1.50
90	12000	7.10	3.10	–

Microstructure of the sintered pieces was observed by scanning electron microscopy (SEM, Carl Zeiss, DSM-950, Germany) on polished and thermally etched surfaces. Sintered densities were measured by immersion in water. X-ray diffraction (D5000, Siemens, Germany) was used to determine the crystalline phases using ground materials.

3. Results and discussion

The effect of mixing and ageing times on the rheological behaviour of the suspensions were first studied. To do this, a constant concentration of dispersing aid (citric acid) of 0.3 wt.% was used. Fig. 1 shows the variation of viscosity (at a shear rate of 100 s^{-1}) with ageing time for 50 vol.% suspensions, prepared with different sonication times. The viscosity increases with ageing time for any ultrasounds treatment and thixotropy strongly increases. Table 1 shows the calculated thixotropy values of the different suspensions. The fresh slips always have the lowest viscosity and thixotropy, but for 1 min sonication time settling occurs rapidly, so that ageing leads to a significant increase of viscosity and very large increase of thixotropy. This means that 1 min US is not enough to homogenise the suspension and the dispersant is not readily adsorbed at the particles surface. The highest stability is reached after 5 min ultrasonic mixing, where both the increase of viscosity with ageing time and the thixotropy

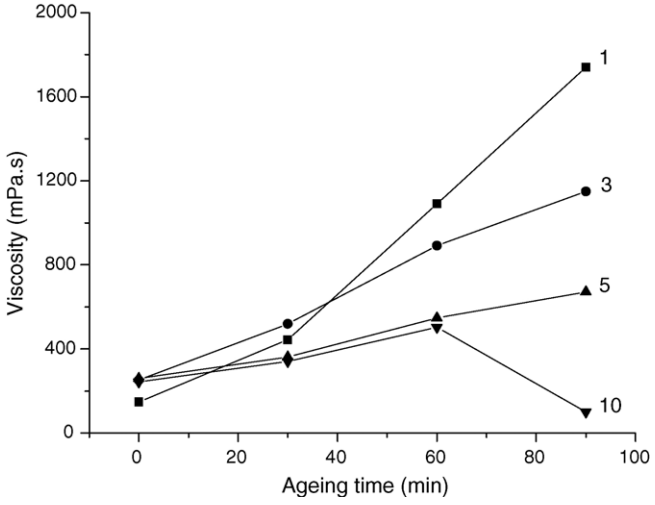

Fig. 1. Viscosity vs. ageing time of 50 vol.% suspensions dispersed with 0.3 wt.% citric acid after different sonication times (min).

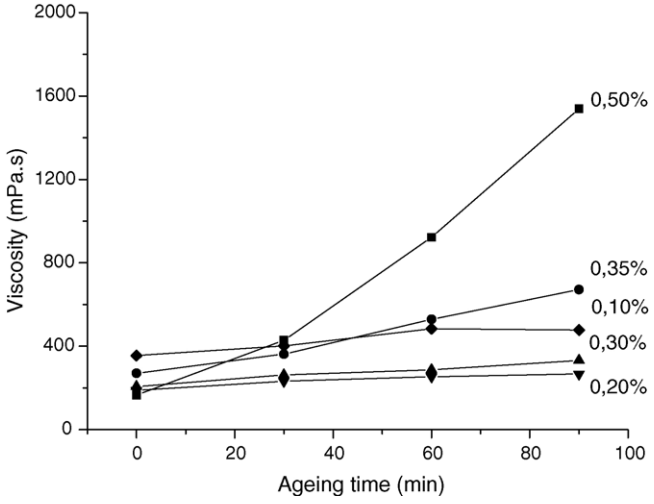

Fig. 2. Viscosity vs. ageing time of 50 vol.% suspensions dispersed with different concentrations of citric acid after sonication time of 5 min.

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